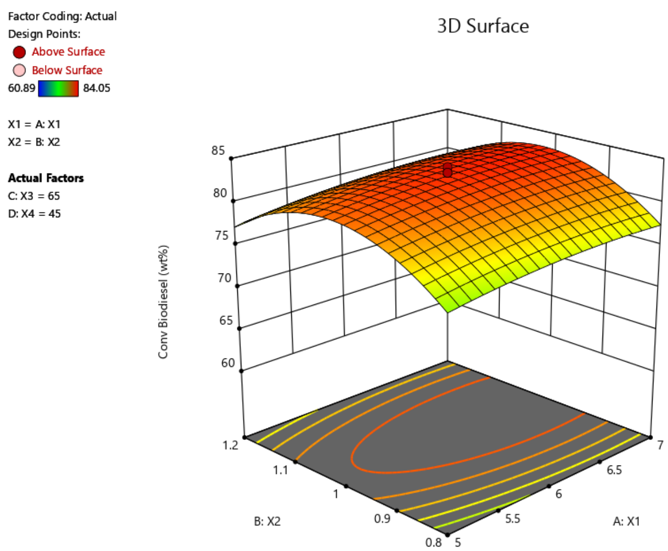
Applications of DOE in Engineering and Science: A Collection of 2^6 Case Studies



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1st Edition (Revised)

Forward to *Use of DOE in Engineering and Science*

Dr. Lye's association with Stat-Ease and me goes back to 2005 when he started using our software for teaching DOE. He came to our headquarters in Minneapolis for a workshop and gave us a book on Newfoundland that inspired me to visit him in St. John's in 2018. Dr. Lye told me then that upon his retirement from Memorial University he would devote time to a comprehensive collection of DOE case studies. I am very pleased to see this come to fruition.

I did my first DOE in 1974 and took to this multifactor testing approach immediately as a catalyst for my work as a chemical engineer working on process development. Why anyone would continue to study only one factor at a time (OFAT) remains a mystery to me. However, OFAT being established throughout the educational process as the scientific method cannot be easily undone in the minds of highly-trained experimenters. The only way that I've found to do so is by presenting relevant examples. This book by Dr. Lye provides a treasure trove for anyone who wants to persuade others to do DOE or those that seek compelling evidence of its advantages for their research work.

Now that Dr. Lye has done such a great service to the field of DOE by presenting these 64 examples, I hope that he will turn his attention to making his DOE-Golfer™ training device legal for use on the course. That will solve all my problems for putting. However, I will settle for Dr. Lye sparing time from happy retirement to continue his good work spreading the good word about DOE. Well done!

- Mark J. Anderson, PE, CQE
Principal, Stat-Ease, Inc.
Minneapolis, MN, USA
July 9, 2019

PREFACE

“The only way to know how a complex system will behave — after you modify it — is to modify it and see how it behaves.” George Box

Since 1995, I have been teaching a graduate course strangely titled “Similitude, Modelling, and Data Analysis” ENGI 9516 at the Faculty of Engineering and Applied Science, Memorial University of Newfoundland. The original instructor of this course, Dr. James Sharp, was an expert in the field of hydraulics with particular expertise in hydraulic models and dimensional analysis. He taught this course for many years and also wrote several books on these subjects. A few years before he retired, he asked me to co-teach the course. Since my expertise is in statistical hydrology, I added a few topics on data analysis, particularly regression analysis. When Dr. Sharp retired, I became the sole instructor of the course, and as I am not an expert in similitude or dimensional analysis, the current course content is mostly on the design and analysis of multifactor experiments. Dimensional analysis and how it can be combined with modern design of experiment methodologies is now only a small part of my course. However, to prevent unnecessary university calendar changes, the name of the course has remained the same. The course is now better known as the Design of Experiment or the DOE course and is one of the core courses for graduate students in engineering. Students taking the course come from all disciplines of engineering and from the Faculty of Science. Over the years, the class size has grown from about 20 students to 40-50, in recent years. The course covers the following topics:

1. Design of Experiments, definition of and strategies for experimentation
2. Factorial vs. one-factor-at-a-time (OFAT) experiments
3. Review of one-factor experiments, regression, and ANOVA
4. General factorial experiments
5. Design and analysis of 2-level factorial experiments
6. Concepts of blocking and confounding
7. Fractional factorial design and analysis, fold-over designs
8. Response Surface Methodology (RSM): Central Composite Design (CCD), and Box-Behnken Design (BBD)
9. Design for computer experiments, uniform designs, and other designs
10. Restricted randomization and hard-to-change factors
11. Optimal designs, multiple linear constraints, and definitive screening designs.
12. Methods of dimensional analysis
13. The combined use of DOE and dimensional analysis

Mixture designs, combined mixture-process designs, Taguchi methods, and other advanced topics are not covered.

The use of design of experiments (DOE) methodologies such as the topics covered in the course has increased exponentially over the years in almost all areas of science and engineering. There are also many textbooks by both statisticians and others that cover topics related to the statistical design of experiments.

Over the last 20 or more years, I have learned that, according to my students, one of the most useful parts of the course is reviewing journal papers that use DOE methods in experiments. Students are asked to search for journal papers in their discipline or field of interest and do a thorough review. This review requires that students identify the objectives of the paper, the factors used in the experiment, the responses measured, and the choice of experimental design. Then students must evaluate the correctness of the statistical analyses, and the results. Invariably, on reanalysis of the data given in the paper, the students often find that the data reported may be wrong, the statistical model choice is wrong, or the ANOVA assumptions may not have been checked. Or, the students may find the results reported are not reproducible. Having students review published journal papers provides an opportunity to develop confidence in their knowledge of DOE and helps students realize that not all published papers can be taken at face value. Important errors in analysis or data can be missed either by the authors or reviewers despite the fact that many of the reviewed papers are published in reputable journals.

Over the years, I have amassed a large collection of journal and some conference papers that use DOE methodologies in engineering and science. This book provides a selection of 2^6 case studies from this collection. The case studies cover a wide range of applications in engineering and science. I chose only papers where there is a complete set of data available for reanalysis. The selection is not exhaustive and does not cover every discipline of engineering or science. However, I hope that readers of the book get a good sense of the wide application of DOE methods, and will try their hand at analyzing the published data. The methods most commonly used in the papers deal mainly with factorial designs, fractional factorial designs, and response surface methodologies, particularly the use of the central composite and Box-Behnken designs.

The book is ideal for students who have taken or is taking a course in DOE. It is also useful for those who want to learn more about the power of DOE methods or who are looking for research ideas. Each dataset is available in print form in the book and available as an Excel file (.xls) and as a Design-Expert® file (.dxpx). Hence this collection of case studies is also be a good resource for instructors of DOE. Please contact me at llye@mun.ca for the files.

I want to thank the hundreds of students who have taken my course over the years and the feedback they have offered. I learned so much from interacting with them and helping them design unique experiments for their thesis and other research work. I would particularly like to acknowledge the unwavering support of Mark Anderson of Stat-Ease Inc. the publisher of Design-Expert® software, by providing a free 6-month version of their software to my students each year. My class has been using Design-Expert® as the software of choice since version 5. There are other statistical packages available for DOE such as Excel Add-ins, Minitab, and JMP, but I have found Design-Expert® to be comprehensive and easy to use. Furthermore, the company Stat-Ease Inc. provides excellent support through their website, YouTube channel, and regular webinars.

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TABLE OF CONTENTS

1. INTRODUCTION

This book provides a collection of 2^6 case studies in the field of engineering and science based on articles published in a wide variety of journals from 2000 to 2018. The methodology used in each study falls into one of eight types which form the eight main chapters of this book. These methods are:

- General factorial designs
- 2-level factorial designs or 2^k designs
- 2-level fractional factorial designs or 2^{k-p} designs
- 3-level factorial designs or 3^k designs
- Response surface methodology or RSM: Box-Behnken designs or BBD
- Response surface methodology or RSM: Rotatable Central composite design or CCD
- Response surface methodology or RSM: Face Centered Central composite design or FCD
- Combination designs

The chapter on general factorial designs has four case studies on the use of multi-factored general factorial design with a different number of levels for each factor. There are 10 case studies on the use of 2-level factorial designs, nine on 2-level fractional factorial designs, and six on 3-level factorial designs. There are three chapters on the use of response surface methodology or RSM sub-divided as follows: Box-Behnken design or BBD (eight case studies), rotatable Central Composite Designs or CCD (10 case studies), and face-centered designs or FCD (10 case studies). Rotatable and face-centered designs are in two separate chapters. Combination designs are those that use more than one type of design. They could be a 2-level factorial or a 2-level fractional factorial design, followed by a RSM design. There are seven case studies of this type. The number of case studies in each chapter roughly represents the popularity of each design.

The field of experimental design is very wide and this book only covers the most common DOE methodologies found in journals of science and engineering. Other DOE methodologies such as Latin square designs, repeated measures design, nested designs, optimal designs, space-filling designs, definitive screening designs, split-plot designs, mixture designs, and other less commonly used or more advanced methodologies are not covered.

The case studies in this book are based on articles published in over 55 different journals and shows the wide application of DOE methodology in science and engineering. As mentioned in the Preface, only papers that contain the complete design and responses are included so that readers can analyse the data for themselves and compare their results with those presented in the paper. Naturally the list of journals is not exhaustive. In alphabetical order, the list of journals is as follows. Case study and page numbers are shown for ease of reference.

Advances in Environmental Research – CS #2.1, p 8.

Applied Mathematical Modelling – CS #3.7, p 37.

Journal of Materials Processing Technology – CS #8.6, p 153.

Journal of Materials Research and Technology – CS #7.4, p 120.

Journal of Membrane Science – CS #4.1, p 45.

Journal of Safety Research = CS #2.2, p 10.

Journal of the Taiwan Institute of Chemical Engineers – CS #2.4, p 15.

Journal of Water Reuse and Desalination – CS #3.6, p 35.

Korean Journal of Chemical Engineering – CS #3.3, p 26.

Materials and Design – CS #3.10, p 43; CS #4.4, p 55.

Material Science and Engineering A – CS #2.3, p 13; CS #9.5, p 184.

Materials and Manufacturing Processes – CS #6.4, p 98.

Microbial Pathogenesis – CS #6.5, p 100.

Numerical Heat Transfer – CS #4.5, p 60.

Petroleum Science and Technology – CS #3.8, p 39.

Pigment & Resin Technology – CS #8.3, p 146.

Practice Periodical of Hazardous, Toxic, and Radioactive Waste Management - CS #4.8, p 67.

Proceedings Institution of Mechanical Engineers – CS #5.3, p 78.

Promet – Traffic and Transportation – CS #3.5, p 31.

Renewable Energy – CS #7.10, p 136.

Separation and Purification Technology – CS #8.10, p 165.

Surface and Coatings Technology – CS #4.6, p 63.

The Scientific World Journal – CS #5.1, p 73.

Total Quality Management – CS #9.4, p 181.

Ultrasonic Sonochemistry – CS #8.9, p 162.

Water Science and Technology – CS #3.1, p 19.

In addition to the case studies presented in this book, there are also numerous other case studies published or available in standard text books on DOE such as Box et al (2005), Hicks et al (1999), Kuehl (2000), Montgomery (2017), Myers et al (2016), and Ryan (2007). There are also a few standard text books on science and engineering that contain case studies. These include among others Anthony (2003), Berthouex and Brown (2002), and Mason et al (2003). Most of these case studies are in the assignment sections of these books. “Simplified” books on DOE and RSM that focus on practical applications using the Design-Expert® software have also been published by

Anderson and Whitcomb (2015, 2016) who are principals at Stat-Ease Inc., publisher of the software. Other software for DOE and RSM besides Design-Expert include JMP by SAS, Minitab® by Minitab Inc., Fusion-Pro by S-Matrix, among many others.

The case studies in each chapter of this book are arranged by alphabetical order of the first author's last name. Each case study describes the objective of the experiment, the number of factors and responses in the experiment, the type of design used, the full data set resulting from the experiment, the software used, and a summary of the results obtained by the authors. I encourage the reader to read the original papers and re-analyze the data to compare with the results obtained by the authors. If you find that you obtain the same results, then congratulate yourself and the authors for correctly doing the analysis. If you did not get the same results, here is your opportunity to figure out why. Were the assumptions of regression checked? Were the correct model terms included in the final model? Were you able to obtain a better model? Should there be a transformation for the response? Sometimes there are differences because of the software used, and sometimes there could be typographical errors. In any case, reanalyzing published data is a great learning experience.

Disclaimer

While every effort has been made to reproduce the data and tables in the published papers as accurately as possible, it is advisable that the reader read the original papers for the details of the experiments carried out and to ensure that the data and tables presented here are indeed correct.

2. GENERAL FACTORIAL DESIGNS

Four case studies using general factorial designs are presented in this Chapter. The number of factors ranges from three to four. For each case study, the factors have a differing number of levels ranging from two to five levels.

Case Study #2.1

Annadurai, G., Ruey-Shin Juang, and Duu-Jong Lee (2002): Factorial design analysis for adsorption of dye on activated carbon beads incorporated with calcium alginate. Advances in Environmental Research, 6, pp. 191-198.

This study used a factorial design with three factors to investigate the batch adsorption equilibrium of the dye Rhodomine 6G, using activated carbon beads incorporated with calcium alginate (ACCA beads). The effect of three factors that govern the adsorption process, dye concentration in mg/l, pH, and temperature °C were considered. The factors and levels are shown in Table 2.1.

Table 2.1: Factors and levels for adsorption of dye study.

Factor	Description	Unit	Level 1	Level 2	Level 3
A	Init. Dye Concentration	Mg/l	100	200	300
B	pH		7	8	9
C	Temperature	°C	30		60

The response was the percentage of adsorption of Rhodamine 6G using a fixed dosage of ACCA beads (1 g/l).

Three levels of dye concentrations (100, 200, 300) and pH (7, 8, 9) were used and the temperature was at two levels (30 °C and 60 °C). In total there were (3 x 3 x 2 = 18) runs. The run combinations and responses are shown in Table 2.2. These were taken from Tables 2 and 3 of Annadurai et al (2002).

According the authors, the use of the factorial design and subsequent ANOVA allowed a polynomial model to be fitted, shown in Equation (1) of the paper. The method used to calculate the effects and their sum of squares are given in the paper. There is no mention if any software was used for the ANOVA calculations.

Table 2.2: Operating conditions and responses (after Annadurai et al, 2002).

Trial No.	Dye Concentration (mg/l)	pH	Temperature (°C)	% dye adsorption	Predicted value (%)
1	100	7	30	98.50	98.47
2	200	7	30	96.70	96.77
3	300	7	30	94.80	95.07
4	100	8	30	98.70	98.71
5	200	8	30	97.00	97.01
6	300	8	30	95.30	95.31
7	100	9	30	99.20	98.95
8	200	9	30	97.30	97.25
9	300	9	30	95.60	95.55
10	100	7	60	99.90	99.80
11	200	7	60	98.20	98.10
12	300	7	60	96.40	96.40
13	100	8	60	100.00	100.04
14	200	8	60	98.60	98.34
15	300	8	60	96.70	96.64
16	100	9	60	100.00	100.02
17	200	9	60	98.20	98.59
18	300	9	60	97.10	96.89

The ANOVA results were shown in Table 5 of the paper and are reproduced here as Table 2.3. The R^2 was given as 0.9884 based on fitting a linear model. The model was not shown in the paper but the authors gave the predicted values together with the actual values of the responses in Table 4 of their paper. These predicted values are shown here in Table 2.2.

Table 2.3: Regression analysis for adsorption of dye by linear model fitting (ANOVA). Table 5 of Annadurai et al (2002).

Source	Sum of Squares	df	Mean Square	F-value	P-value
Model	43.38	3	14.46	396.73	<0.0001
Residual	0.51	14	0.036		
Corr. Total	43.89	17			

$R^2 = 0.9884$

From Table 2.3, no interaction or quadratic terms were included in the model. It is not mentioned whether the assumptions of regression were checked. The authors concluded that the initial dye concentration has the most significant effect while the temperature has the least effect on the percentage of dye adsorption.

Case Study #2.2

Al-Darrab, I. A., Zahid A. Khan, and Shiekh I. Ishrat (2009): An experimental study on the effect of mobile phone conversation on drivers' reaction time in braking response. Journal of Safety Research, 40, pp. 185-189.

This study considered the effect of three factors on drivers' reaction time in braking. The aim of the study was primarily to investigate the effect of mobile phone use on driving performance. The factors and levels are shown in Table 2.4:

Table 2.4: Factors and levels for mobile phone study.

Factor	Description	Unit	Level 1	Level 2	Level 3
A	Distance between cars	m	10	15	20
B	Call Duration	s	30	60	90
C	Time of driving		Day		Night

Response = RT, Drivers' reaction time (s)

This experiment had three levels for Factor A and B, and two levels for Factor C. The number of replications was three. The total number of runs was 3 x (3 x 3 x 2) = 54. Two cars travelling at a given distance apart (Factor A) were used. Factor B, the call duration, refers to the time spent talking on a mobile phone. For Factor C, day time is from 1600 to 1800 hours, and night time is from 2030 to 2230 hours. Twenty seven (27) volunteer drivers were used in the experiment where each driver was randomly assigned to two run combinations. The location of the study was in Jeddah, Saudi Arabia. The detailed experimental procedure was explained in the paper.

Design Expert by Statease Inc. was used for the design and analysis. The version of the software was not mentioned.

The results of the experiment were given in Table 2 of the paper, and are reproduced here in Table 2.5.

For the initial analysis, the authors treated each factor as categorical. The ANOVA results are shown in Table 2.6.

Table 2.5: Driver's reaction time for different treatment combinations (data from Al-Darrab et al (2009).

Treatment Combination	Factors			Reaction time (s)		
				Replicate		
	A (m)	B(s)	C	I	II	III
1	10	30	Day	0.09	0.08	0.10
2	10	60	Day	0.25	0.09	0.18
3	10	90	Day	0.23	0.20	0.18
4	15	30	Day	0.20	0.12	0.09
5	15	60	Day	0.15	0.06	0.21
6	15	90	Day	0.22	0.15	0.17
7	20	30	Day	0.07	0.12	0.11
8	20	60	Day	0.09	0.07	0.11
9	20	90	Day	0.18	0.13	0.12
10	10	30	Night	0.12	0.35	0.20
11	10	60	Night	0.18	0.30	0.09
12	10	90	Night	0.46	0.19	0.29
13	15	30	Night	0.15	0.12	0.27
14	15	60	Night	0.18	0.15	0.12
15	15	90	Night	0.63	0.71	0.59
16	20	30	Night	0.17	0.14	0.12
17	20	60	Night	0.21	0.12	0.15
18	20	90	Night	0.90	0.76	0.71

Table 2.6: Results of ANOVA for categorical variables. Results from Al-Darrab et al (2009).

Source	Sum of Squares	df	Mean Square	F-value	P-value	
Model	1.802	17	0.106	22.12	<0.0001	Significant
A	0.018	2	0.009	1.92	0.1612	
B	0.640	2	0.320	66.73	<0.0001	
C	0.394	1	0.394	82.12	<0.0001	
AB	0.137	4	0.034	7.12	0.0002	
AC	0.063	2	0.031	6.52	0.0038	
BC	0.380	2	0.190	39.59	<0.0001	
ABC	0.172	4	0.043	8.98	<0.0001	
Pure Error	0.173	36	0.005			
Corr Total	1.975	53				

The assumptions of ANOVA have been checked by the authors and found to be satisfied. Since Factor A and B were quantitative, the authors also considered developing prediction equations for the response by re-running the ANOVA, treating Factors A and B as quantitative. The results of the ANOVA is shown in Table 2.7.

Table 2.7: Results of ANOVA for quantitative Factors. Results from Al-Darrab et al (2009).

Source	Sum of Squares	df	Mean Square	F-value	P-value	
Model	1.725	9	0.192	33.75	<0.0001	Significant
A	0.014	1	0.014	2.40	0.1287	
B	0.490	1	0.490	86.29	<0.0001	
C	0.005	1	0.005	0.82	0.3693	
B^2	0.150	1	0.150	26.35	<0.0001	
AB	0.089	1	0.089	15.64	0.0003	
AC	0.063	1	0.063	11.01	0.0018	
BC	0.250	1	0.250	44.03	<0.0001	
B^2C	0.130	1	0.130	22.81	<0.0001	
ABC	0.147	1	0.147	25.93	<0.0001	
Residual	0.250	44	0.006			
Lack of Fit	0.077	8	0.010	2.02	0.0723	not significant
Pure Error	0.173	36	0.005			
Corr Total	1.975	53				

The most important of the three factors affecting braking reaction time is the mobile call duration (Factor B), followed by the time of driving (Factor C), with the distance between cars (Factor A) being least important. The prediction equations of the reaction time in terms of actual factors were given as:

Time: Day

$$RT = 0.060556 + 0.00255556\ A + 0.00182407\ B + 0.00000864198\ B^2 - 0.0001167\ A.B$$

Time: Night

$$RT = 1.28056 - 0.0434444\ A - 0.035990\ B + 0.000239506\ B^2 + 0.000927778\ A.B$$

No goodness-of-fit statistics were given in the paper. The authors also left out the statistically significant three-factor interaction terms to simplify the models. The results cannot be generalized to older drivers as the volunteer drivers were all between the ages of 22 and 24 years.

Case Study #2.3

Grosselle, F., Giulio Timelli, and Franco Bonollo (2010): DOE applied to microstructural properties of Al-Si-Cu-Mg casting alloys for automotive applications. Material Science and Engineering A, 527, pp. 3536-3545.

The basic aim of the study was to investigate the solidification rate and the effect of T7 heat treatment on microstructural and mechanical properties of cast AlSi7CuMg based alloy for engine block application. That is, by studying the effect of each factor and their interactions on the production of the alloy, the authors were trying to determine whether there is potential to improve performance of the alloy. The four factors were the cooling rate measured by the secondary dendrite arm spacing (SDAS), Titanium content (Ti) Copper content (Cu), and T7 heat treatment. The process factors with their different levels are shown in Table 2.8.

Table 2.8: Process factors with their levels of observation. From Grosselle et al (2010).

Factor designation	Factor name	Lower level	Central level	Higher level
A	SDAS (μm)	17	-	34
B	Titanium content (wt.%)	0	-	0.2
C	Copper content (wt.%)	2	3	4
D	T7 heat treatment	0 (no)		1 (yes)

Five different responses were measured. These were: equivalent diameter (d) and roundness (r) of the eutectic Si particles, yield strength (YS), ultimate tensile strength (UTS), and elongation to fracture (s_f).

The first two responses were obtained from microstructural analysis using an optical microscope and quantitatively analyzed with an image analyzer. The next three responses concerned the effects of the factors on strength related properties. These were obtained from a computer controlled tensile testing machine. The details on how the images were analyzed and how the tensile tests were carried out were described in the paper.

This experiment had 2 levels for Factor A, B, and D, and 3 levels for Factor C. The total number of run combinations was (2 x 2 x 3 x 2) = 24. These run combinations were labelled P1 to P24 by the authors. Hence, a general factorial design was used.

Table 2.9 shows the run combinations together with the five responses obtained from the experiments. In the paper, for each response, the corresponding standard deviation was also given. However, these standard deviations are not shown in Table 2.9 because they were not analyzed by the authors. The run combinations were given in Table 2, and the responses were given in Tables 4 and 5 of the paper.

The software used for the design and analysis of the experiment was not mentioned. However, from the printout of the results, my guess is that the authors used Minitab.

Table 2.9: Run combinations and responses. Tables 2, 4 and 5 of Grosselle et al (2010).

Run	SDAS (μm)	Ti (%)	Cu (%)	Heat treat	d (μm)	r	YS (MPa)	UTS (MPa)	S_f (%)
P01	17	0	2	0	2.3	4.1	159	210	1.3
P02	34	0	2	0	4.6	5.4	161	204	1.1
P03	17	0.2	2	0	2.9	4.3	137	209	1.7
P04	34	0.2	2	0	4.8	4.7	161	214	1.3
P05	17	0	3	0	3.3	5.1	160	212	1.1
P06	34	0	3	0	4.5	5.2	172	210	0.0
P07	17	0.2	3	0	4.4	6.6	171	213	0.5
P08	34	0.2	3	0	6.3	6.3	182	225	1.1
P09	17	0	4	0	2.7	3.9	169	242	1.4
P10	34	0	4	0	4.5	5.4	182	218	0.9
P11	17	0.2	4	0	4.3	3.9	187	227	0.9
P12	34	0.2	4	0	7.5	7.1	187	207	0.7
P13	17	0	2	1	2.9	1.7	255	292	1.4
P14	34	0	2	1	4.2	2.9	244	272	1.1
P15	17	0.2	2	1	3.5	2.3	261	308	1.7
P16	34	0.2	2	1	5.3	3.5	258	278	0.9
P17	17	0	3	1	2.8	2.0	268	305	1.2
P18	34	0	3	1	4.1	2.5	266	289	1.0
P19	17	0.2	3	1	4.2	2.5	260	309	1.6
P20	34	0.2	3	1	5.4	2.5	263	296	1.1
P21	17	0	4	1	2.6	1.7	256	316	1.7
P22	34	0	4	1	3.7	2.5	256	284	1.0
P23	17	0.2	4	1	4.0	2.5	279	321	1.2
P24	34	0.2	4	1	5.7	2.6	267	289	0.9

No ANOVA tables were reported in the paper. However, the main effects and some interaction plots and Pareto charts were given for each response. The prediction equations using only statistically significant terms at α=0.05, for each response, and the corresponding R^2 are given below.

(i) Equivalent diameter of eutectic Si particles, d.

$$d = 0.93 + 0.12\ \text{SDAS} - 8.28\ \text{Ti} - 0.073\ \text{Cu} + 4.62\ \text{Ti.Cu} \qquad R^2 = 0.82$$

(ii) Roundness of eutectic Si particles, r.

$$r = 1.64 + 0.07\ \text{SDAS} + 3.99\ \text{Ti} - 1.17\ \text{T7} \qquad R^2 = 0.87$$

The variable T7 is either 0 for as-cast, or 1 for T7 heat treated conditions.

(iii) Yield strength, YS.

$YS = 186.2 + 9.2\ Cu + 45.8\ T7)$ $R^2 = 0.95$

(iv) Ultimate tensile strength, UTS.

$UTS = 263 – 1.2\ SDAS + 7.3\ Cu + 39.3\ T7)$ $R^2 = 0.95$

(v) Elongation to fracture, s_f.

$s_f = 1.83 – 0.0242\ SDAS + 0.0013\ Cu – 1.19\ Cu.Ti + 0.0404\ T7$ $R^2 = 0.67$

The authors did not mention whether the assumptions of ANOVA were checked. Standard deviations for each of the responses were reported in the paper but were not analyzed. The prediction equations were also not validated using additional experimental runs.

Case Study #2.4

Kordkandi, S. A. and Mojtaba Forouzesh (2014): Application of full factorial design for methylene blue dye removal using heat-activated persulfate oxidation. Journal of the Taiwan Institute of Chemical Engineers, 45, pp. 2597-2604.

This study considered the use of a thermally activated persulfate oxidation process to treat aqueous methylene blue (MB) dye. Four factors which include reaction time, persulfate concentration, initial MB concentration, and process temperature were investigated. The factors and levels used are shown in Table 2.10.

Table 2.10: Levels and factors used in the experimental design (adapted from Kordkandi and Farouzesh, 2014)

Factors	Operating variables	Units	Levels				
			1	**2**	**3**	**4**	**5**
t	Reaction time	min	5	10	15	20	25
C_{OX}	Persulfate concentration	mg/L	355	710	1065	-	-
C_{MB}	Initial MB concentration	mg/L	10	15	20	-	-
T	Process temperature	(°C)	60	70	-	-	-

The response variable was the percentage color removal efficiency (CR%). From the experimental data, activation energy and kinetic parameters were also calculated and a model was proposed for predicting the performance of the color removal percentage. The details of the experiments and how they were carried out were well explained in the paper.

From Table 2.10, reaction time (t, min) has 5 levels (5, 10, 15, 20, and 25), persulfate concentration (C_{OX}, mg/l) has 3 levels (355, 710, and 1065), initial MB concentration (C_{MB}, mg/l) has 3 levels (10, 15, and 20) and the process temperature (T, °C) was at 2 levels (60 and 70). The total number of runs is hence (5 x 3 x 3 x 2) = 90. The data were not given in the paper but have been generously provided herein by the authors. The full dataset is given in Table 2.11.

Minitab 16 was used by the authors to analyze the data and a first order of the form shown below (Equation 9 of Kordkani and Forouzesh, 2014), was fitted to the data.

$$Y_{predicted} = b_0 + \sum_{i=1}^{n} b_i x_i + \sum_{i=1}^{n} \sum_{j=1}^{n} b_{ij} x_i x_j + \cdots,$$

where Y is the CR% and x_i, x_j, … are coded variables, b_0 is the global mean, and b_i, b_{ij}, are estimated regression coefficients for the main and interaction effects. The adjusted R^2 was used as a model selection criterion.

Based on a significance level of 5%, the authors suggested the following prediction equation (Equation 10 in the paper) for CR%:

$$CR\% = 14.393 + 1.233\ t - 11.806\ C_{OX} + 1.064\ C_{MB} + 5.525\ T + 2.18\ t.C_{OX} - 1.365\ t.C_{MB}$$
$$+ 3.64\ t.T + 9.62\ T.C_{OX} - 4.2\ T.\ C_{MB},$$

where all the terms are defined in Table 2.10. The corresponding ANOVA results of the proposed model are shown in Table 2.12.

Table 2.12: ANOVA for proposed model (Table 5 of Kordkandi and Forouzesh, 2014)

Source	Degree of freedom	Sum of Squares	Mean Square	F_o	P-value
Regression	9	42,644.10	4738.23	214.875	0.000
Residual Error	80	1,764.10	22.05	-	-
Total	89	44,408.20	-	-	-

The model has a standard deviation of 4.7, Durbin-Watson statistic = 1.61, R^2 = 96%, and R^2 (adjusted) = 95.6%. The authors did not consider any three-factor interaction or quadratic terms in the proposed model.

It is important to note that the prediction equation given above (Equation 10 in the paper), was based on the levels used rather than the actual values in the experiment. For example, to predict CR% say at the low level for all factors, then the value of 1 must be used for t, C_{OX}, C_{MB}, and T, and not 5 min, 355 mg/L, 10 mg/L, and 60 °C, respectively. It is suggested that the reader redo the regression using the actual values rather than using the levels so that the prediction equation can be used directly in terms of actual values without the need to convert levels into actual values. The prediction equations were also not validated using additional experimental runs.

Table 2.11: Run combinations and responses (data provided by S. A. Kordkandi via personal communications).

Run	Reaction Time (min)	Initial Oxidant Conc. (mg/l)	Initial Dye Conc. (mg/l)	Temp. (C)	CR%
1	1	1	1	1	14.6
2	1	1	1	2	33.5
3	1	1	2	1	12.4
4	1	1	2	2	20.4
5	1	1	3	1	10.6
6	1	1	3	2	19.5
7	1	2	1	1	17.8
8	1	2	1	2	40.6
9	1	2	2	1	14.1
10	1	2	2	2	29.3
11	1	2	3	1	12.0
12	1	2	3	2	27.1
13	1	3	1	1	20.5
14	1	3	1	2	48.6
15	1	3	2	1	16.0
16	1	3	2	2	33.5
17	1	3	3	1	10.5
18	1	3	3	2	30.6
19	2	1	1	1	26.1
20	2	1	1	2	48.5
21	2	1	2	1	18.8
22	2	1	2	2	33.3
23	2	1	3	1	17.3
24	2	1	3	2	29.2
25	2	2	1	1	28.7
26	2	2	1	2	66.2
27	2	2	2	1	22.4
28	2	2	2	2	43.3
29	2	2	3	1	16.6
30	2	2	3	2	39.0
31	2	3	1	1	33.0
32	2	3	1	2	74.4
33	2	3	2	1	27.2
34	2	3	2	2	56.4
35	2	3	3	1	20.0
36	2	3	3	2	51.6
37	3	1	1	1	34.3
38	3	1	1	2	59.4
39	3	1	2	1	25.9
40	3	1	2	2	38.8
41	3	1	3	1	21.4
42	3	1	3	2	33.7
43	3	2	1	1	37.1
44	3	2	1	2	81.1
45	3	2	2	1	29.3

Run	Reaction Time (min)	Initial Oxidant Conc. (mg/l)	Initial Dye Conc. (mg/l)	Temp. (C)	CR%
46	3	2	2	2	52.2
47	3	2	3	1	22.1
48	3	2	3	2	51.8
49	3	3	1	1	43.1
50	3	3	1	2	89.7
51	3	3	2	1	35.1
52	3	3	2	2	71.8
53	3	3	3	1	25.7
54	3	3	3	2	65.2
55	4	1	1	1	39.6
56	4	1	1	2	65.4
57	4	1	2	1	27.6
58	4	1	2	2	43.2
59	4	1	3	1	23.8
60	4	1	3	2	38.5
61	4	2	1	1	45.3
62	4	2	1	2	88.5
63	4	2	2	1	35.0
64	4	2	2	2	60.1
65	4	2	3	1	25.7
66	4	2	3	2	59.3
67	4	3	1	1	51.7
68	4	3	1	2	95.9
69	4	3	2	1	42.8
70	4	3	2	2	82.4
71	4	3	3	1	31.6
72	4	3	3	2	74.6
73	5	1	1	1	44.0
74	5	1	1	2	70.6
75	5	1	2	1	30.4
76	5	1	2	2	46.8
77	5	1	3	1	26.1
78	5	1	3	2	44.1
79	5	2	1	1	50.0
80	5	2	1	2	92.6
81	5	2	2	1	38.3
82	5	2	2	2	67.1
83	5	2	3	1	29.5
84	5	2	3	2	66.1
85	5	3	1	1	60.0
86	5	3	1	2	99.1
87	5	3	2	1	49.9
88	5	3	2	2	88.6
89	5	3	3	1	36.3
90	5	3	3	2	81.5

Note that all factors are in terms of the levels used and not the actual units.

3. 2-LEVEL FACTORIAL DESIGNS

10 case studies using 2-level or 2^k full factorial designs are presented in this Chapter, where k is the number of factors. The number of factors ranges from three to five. 2^k designs are one of the most popular experimental designs because they use only two levels (a low and a high value). Center points may also be included to test for curvature and to give a measure of pure error. For five or more factors it is more economical to use fractional factorial designs (Chapter 4). The 2^k designs allow the fitting of only linear and interaction terms.

Case Study #3.1

Alimi, F., Ali Boubakri, Mohamed M. Tlili, and Mohamed Ben Amor (2014): A comprehensive factorial design study of variables affecting $CaCO_3$, scaling under magnetic water treatment. Water Science and Technology, 70.8, pp 1355-1361.

This study used a three-factor two-level (2^3) full factorial design to investigate the effects of magnetic water treatment (MWT) to prevent calcium carbonate ($CaCO_3$) scaling for domestic and industrial equipment. The three factors studied were pH, flow rate, and application of magnetic field. The factors and levels used were shown in Table 1 of the paper and are reproduced here as Table 3.1.

Table 3.1: Coded levels and actual values of factors used in the design. Table 1 of Alimi et al (2014).

		Real values of coded levels		
Variable	Symbol	Low (-1)	Central point (0)	High (1)
pH (numeric)	X_1	6	6.75	7.5
Flow rate, Q L/min) (numeric)	X_2	0.54	0.74	0.94
Magnetic field (text)	X_3	With		Without

Three responses were of interest – induction time (IT), total precipitation (TP) rate, and homogenous precipitation (HP) rate of $CaCO_3$ scale from hard water. The materials and methods used in the experiment were described in the paper. Minitab 15 statistical software was used for the design and analysis of the experiment. The 2^3 full factorial design and results of the three responses were shown in Table 2 of the paper are reproduced here as Table 3.2. The pH and flowrate were numeric factors while the application of magnetic field was a categorical factor. Two center points per category were added giving a total of 12 (eight factorial plus 4 center points) combinations.

Table 3.2: Experimental matrix design and results obtained for each of the studied response variables. Table 2 of Alimi et al (2014).

	Input variables						Responses		
Run	pH		Q (L/min)		Magnetic field				
number	Actual	Coded	Actual	Coded	Actual	Coded	IT (min)	TP (%)	HP (%)
1	6	-1	0.54	-1	With	-1	14	79.2	21.5
2	7.5	1	0.54	-1	With	-1	5	89.4	42.6
3	6	-1	0.94	1	With	-1	7	83.4	45.0
4	7.5	1	0.94	1	With	-1	5	91.9	52.4
5	6	-1	0.54	-1	Without	1	18	72.5	13.4
6	7.5	1	0.54	-1	Without	1	4	72.5	40.0
7	6	-1	0.94	1	Without	1	12	72.5	39.0
8	7.5	1	0.94	1	Without	1	2	72.5	49.5
9	6.75	0	0.74	0	With	-1	9	82.8	35.0
10	6.75	0	0.74	0	Without	1	13	72.5	29.4
11	6.75	0	0.74	0	With	-1	11	84	34.0
12	6.75	0	0.74	0	Without	1	14	72.5	28.5

From Table 3.2, IT ranged from 2 to 18 minutes, TP ranged from 72.5% to 91.9%, and HP ranged from 13.4 to 52.4%. A first order polynomial regression model was fitted to each response. The effects, regression coefficient estimates, associated p-values, and goodness of fit statistics for all three responses were shown in Table 3 of the paper and are reproduced here as Table 3.3. Selecting regression coefficients that were statistically significant at the 5% level, the following empirical models in terms of coded values were obtained:

Model for IT:

$$IT = 8.8375 - 4.375\ X_1 - 1.875\ X_2 - 1.625\ X_1X_3$$

Model for TP rate:

$$TP = 79.237 + 2.337\ X_1 - 6.308\ X_3 - 2.337\ X_1X_3$$

Model for HP rate:

$$HP = 37.925 + 8.200\ X_1 + 8.550\ X_2 - 2.558\ X_3 - 3.725\ X_1X_2 + 1.075\ X_1X_3$$

However, no goodness of fit statistics were given for these reduced models. Also note that except for TP, the models for IT and HP were not hierarchical. Not maintaining hierarchy is not wrong per se, but justification for it should perhaps be given.

Table 3.3: Estimated effect and coefficients for (a) IT, (b) TP, and (c) HP. Table 3 of Alimi et al (2014).

Model term	Effect	Coefficient	S.E.	p-value
(a) For IT (Y_1)				
Constant		8.375	0.4948	<0.000
X_1	-8.750	-4.375	0.4948	<0.003
X_2	-3.750	-1.875	0.4948	<0.032
X_3	2.000	1.000	0.404	0.090
X_1X_2	2.750	1.375	0.4948	0.069
X_1X_3	-3.250	-1.625	0.4948	<0.046
X_2X_3	-0.250	-0.125	0.4948	0.817
$X_1X_2X_3$	-0.750	-0.375	0.4948	0.504
S=1.39940	PRESS = 728.277			
R^2 = 97.80%	R^2 (pred) = 0.00%		R^2 (adj) = 91.93%	
(b) For TP rate (Y_2)				
Constant		79.237	0.4628	<0.000
X_1	4.675	2.337	0.4628	<0.015
X_2	1.675	0.838	0.4628	0.168
X_3	-12.617	-6.308	0.3779	<0.000
X_1X_2	-0.425	-0.213	0.4628	0.677
X_1X_3	-4.675	-2.337	0.4628	<0.015
X_2X_3	-1.675	-0.838	0.4628	0.168
$X_1X_2X_3$	0.425	0.213	0.4628	0.677
S=1.30900	PRESS = 865.150			
R^2 = 99.12%	R^2 (pred) = 0.00%		R^2 (adj) = 96.79%	
(c) For HP rate (Y_3)				
Constant		37.925	0.2224	<0.000
X_1	16.400	8.200	0.2224	<0.000
X_2	17.100	8.550	0.2224	<0.000
X_3	-5.117	-2.558	0.1816	<0.001
X_1X_2	-7.450	-3.725	0.2224	<0.000
X_1X_3	2.150	1.075	0.2224	<0.017
X_2X_3	0.450	0.225	0.2224	0.3860
$X_1X_2X_3$	-0.600	-0.300	0.2224	0.2700
S=0.628932	PRESS = 301.158			
R^2 = 99.92%	R^2 (pred) = 78.89%		R^2 (adj) = 99.69%	

Coefficients are given in coded units. S.E.: standard error coefficient;
S: standard deviation; PRESS: Prediction Error Sum of Squares.

The addition of center points allow for estimating pure errors and test for curvature. The test for curvature was not mentioned in the paper.

Reanalysis of the data using Design-Expert 12 showed that the curvature was highly statistically significant for IT (p-value = 0.0072), and HP (p-value = 0.0004). The regression equation suggested for IT is missing the X_1X_2 term. Furthermore, the standard errors, p-values, and goodness of fit statistics for all responses were quite different compared to those shown in Table 3.3. The predicted R^2 values for IT and TP were very likely to be in error as they should not be 0.00%.

Case Study #3.2

Berrama, T., N. Benaouag, F. Kaouah, and Z. Bendjama (2013): Application of full factorial design to study the simultaneous removal of copper and zinc from aqueous solution by liquid-liquid extraction. Desalination and Water Treatment, 51, pp. 2135-2145.

This study used a five-factor two level (2^5) full factorial design to investigate the removal of zinc and copper by liquid-liquid extraction. Liquid-liquid extraction is a widely used method for recovering heavy metals. The details of the method was described in the paper. Five factors that affect the extraction process were the pH of the initial solution, the initial concentration of the metal (Zn or Cu), the concentration of the extractant, the medium type of initial aqueous solution, and the stirring rate. The factors and levels used in the experiment were shown in Table 1 of the paper and are reproduced here as Table 3.4. The experimental procedure and materials used were described in the paper.

Table 3.4: Design factors and their levels. Table 1 of Berrama et al (2013).

Control factors	Code	Unit	Factor levels	
			Low (-1)	High (+1)
pH of initial solution	X_1		4.5	6.5
Initial concentration of metal [$(Zn)_0$ or $(Cu)_0$]	X_2	mg/L	25	75
Concentration of extractant (D_2EHPA)	X_3	(% vol.)	5	10
Medium type of initial aqueous solution	X_4		Sulphate	Chloride
Stirring rate	X_5	rpm	400	500

Two responses were of interest – percentage removal of zinc (II) (Y_{Zn}), and the percentage removal of Copper (II) (Y_{Cu}).

The full-factorial design with 5 factors required 32 runs. No center points were added. There was no mention of any statistical software used for the design or analysis of the experiment. The experimental design in terms of coded factors and results were shown in Table 2 of the paper and are reproduced here as Table 3.5.

Table 3.5: Experimental design matrix and results. Table 2 of Berrama et al (2013).

Run	Factor					Response	
	X_1	X_2	X_3	X_4	X_5	Y_{Zn}	Y_{Cu}
1	-1	-1	-1	-1	-1	90.1	96.5
2	1	-1	-1	-1	-1	93.4	96.7
3	-1	1	-1	-1	-1	83.7	96.1
4	1	1	-1	-1	-1	85.9	96.8
5	-1	-1	1	-1	-1	96.7	98.4
6	1	-1	1	-1	-1	97.0	98.8
7	-1	1	1	-1	-1	93.7	98.5
8	1	1	1	-1	-1	98.3	98.8
9	-1	-1	-1	1	-1	95.9	95.8
10	1	-1	-1	1	-1	97.2	95.9
11	-1	1	-1	1	-1	95.0	95.3
12	1	1	-1	1	-1	96.6	99.5
13	-1	-1	1	1	-1	99.3	98.4
14	1	-1	1	1	-1	99.0	98.6
15	-1	1	1	1	-1	91.9	98.1
16	1	1	1	1	-1	97.3	98.2
17	-1	-1	-1	-1	1	98.0	96.1
18	1	-1	-1	-1	1	94.8	96.7
19	-1	1	-1	-1	1	88.0	96.0
20	1	1	-1	-1	1	94.5	97.4
21	-1	-1	1	-1	1	97.0	94.3
22	1	-1	1	-1	1	97.0	98.2
23	-1	1	1	-1	1	89.5	98.5
24	1	1	1	-1	1	93.4	98.9
25	-1	-1	-1	1	1	94.4	96.2
26	1	-1	-1	1	1	95.4	96.7
27	-1	1	-1	1	1	89.3	95.7
28	1	1	-1	1	1	88.1	98.4
29	-1	-1	1	1	1	98.0	98.5
30	1	-1	1	1	1	96.8	98.5
31	-1	1	1	1	1	85.8	98.0
32	1	1	1	1	1	90.1	98.2

A standard first order polynomial regression model was fitted to each of the responses and all 31 effects and the overall mean were estimated. The estimated effects, regression coefficients and associated t-values and p-values were shown in Table 3 of the paper and are reproduced here as Table 3.6.

Table 3.6: Estimated effects and student's t-test for the yield of Zn(II) and Cu(II) using 2^5 full factorial design. Table 3 of Berrama et al (2013).

	Removal of Zn			Removal of Cu		
Variable	Effect	t-value	p-value	Effect	t-value	p-value
Mean	93.78	320.20	3.20E-37	97.39	1015.00	8.90E-36
X_1	0.89	3.04	0.00336	0.50	5.252	6.10E-05
X_2	-2.46	-8.42	3.90E-08	0.25	2.626	0.00997
X_3	1.26	4.32	0.00018	0.78	8.138	5.60E-07
X_4	0.60	2.04	0.02787	0.10	1.075	0.15025
X_5	-0.65	-2.23	0.01901	-0.13	-1.320	0.10356
X_1X_2	0.81	2.79	0.00591	0.13	1.355	0.09839
X_1X_3	0.17	0.59	0.28212	-0.15	-1.550	0.07163
X_1X_4	-0.21	-0.71	0.2417	0.01	0.117	0.45415
X_1X_5	-0.26	-0.89	0.19346	0.11	1.160	0.13276
X_2X_3	-0.08	-0.29	0.3882	-0.03	-0.370	0.35795
X_2X_4	-0.15	-0.52	0.30358	-0.08	-0.870	0.20038
X_2X_5	-0.83	-2.83	0.00538	0.11	1.166	0.13148
X_3X_4	-0.87	-2.98	0.00388	0.02	0.228	0.41145
X_3X_5	-0.95	-3.23	0.00219	-0.16	-1.650	0.06075
X_4X_5	-1.49	-5.09	3.30E-05	0.15	1.544	0.07241
$X_1X_2X_3$	0.40	1.36	0.09565	-0.34	-3.544	0.00162
$X_1X_2X_4$	-0.23	-0.80	0.21674	0.28	2.893	0.0059
$X_1X_2X_5$	0.24	0.82	0.21076	-0.14	-1.510	0.07643
$X_1X_3X_4$	0.17	0.59	0.28212	-0.28	-2.920	0.00561
$X_1X_3X_5$	0.07	0.25	0.40439	0.11	1.173	0.13021
$X_1X_4X_5$	-0.06	-0.20	0.42076	-0.19	-1.940	0.03628
$X_2X_3X_4$	-0.80	-2.72	0.00678	-0.33	-3.410	0.00212
$X_2X_3X_5$	-0.37	-1.27	0.10977	0.17	1.779	0.04849
$X_2X_4X_5$	-0.46	-1.59	0.0642	-0.24	-2.510	0.01252
$X_3X_4X_5$	0.99	3.38	0.00156	0.14	1.414	0.08961
$X_1X_2X_3X_4$	0.42	1.44	0.08302	-0.04	-0.460	0.32765
$X_1X_2X_3X_5$	-0.28	-0.95	0.17713	-0.05	-0.480	0.31857
$X_1X_2X_4X_5$	-0.41	-1.40	0.08916	0.04	0.469	0.32309
$X_1X_3X_4X_5$	0.00	-0.01	0.4958	-0.04	-0.430	0.33685
$X_2X_3X_4X_5$	0.44	1.50	0.07446	-0.06	-0.680	0.25252
$X_1X_2X_3X_4X_5$	0.42	1.44	0.08302	0.19	1.981	0.0338

For Zn(II) removal, the following prediction model was suggested (Equation 4 of the paper):

$$Y_{Zn} = 93.78 + 0.89X_1 - 2.64X_2 + 1.26X_3 + 0.60X_4 - 0.65X_5 + 0.81X_1X_2 - 0.83X_2X_5 - 0.87\,X_3X_4 - 0.95X_3X_5 - 1.49X_4X_5 - 0.80X_2X_3X_4 + 0.99X_3X_4X_5$$

For Cu(II) removal, the following prediction model was suggested (Equation 5 of the paper):

$$Y_{Cu} = 97.39 + 0.50X_1 + 0.25X_2 + 0.78X_3 - 0.34X_1X_2X_3 + 0.28X_1X_2X_4 - 0.28X_1X_3X_4 - 0.33X_2X_3X_4 - 0.24X_2X_4X_5$$

The R^2 values for Y_{Zn} and Y_{Cu} were 0.908 and 0.814, respectively. No other goodness of fit statistics were given. Apparently, only coefficients that were statistically significant at the 5% were selected to be in the model. For Y_{Cu}, $X_1X_4X_5$, $X_2X_3X_5$, and $X_1X_2X_3X_4X_5$ should also be in the model as their p-values were less than 5%. Also note that both models were not hierarchical. The summarized ANOVA results for the two responses were shown in Table 4 of the paper and are reproduced here as Table 3.7.

Table 3.7: ANOVA models. Table 4 of Berrama et al (2013).

Source	Zn(II)			Cu(II)		
	Degree of freedom	Sum of square	Mean of square	Degree of freedom	Sum of square	Mean of square
Model	12	515.304	42.942	8	43.5947	5.4493375
Residual	19	52.158	2.745	23	9.9341875	0.4319212
Total	31	567.462	18.305	31	53.5288875	1.72673831
F-ratio	15.64			12.61		
p-value	2.26619E-07			9.01885E-07		
R^2	0.908			0.814		

The interpretation of the results were given in the paper using a variety of interaction plots. As shown by the prediction equations, the importance of the main and interaction effects were quite different for zinc and for copper removal.

To test the accuracy of the models, two additional experiments were conducted to compare predictions obtained by the models and with those obtained experimentally. However, levels used for the factors were not mentioned. The results were shown in Table 5 of the paper and are reproduced here as Table 3.8.

Table 3.8: Comparison between the experimental and simulated values. Table 5 of Berrama et al (2013).

	Experimental value	Model response	Error (%)
Cu(II)	99.22	98.28	0.95
	99.07	98.08	1.07
Zn(II)	95.56	95.52	0.99
	96.98	95.34	1.69

The results showed that the error of prediction were less than 2% and the removal percentages were over 95%.

Case Study #3.3

Boubakri, A., Nawel Helali, Mohamed Tlili, and Mohamed Ben Amor (2014): Fluoride removal from diluted solutions by Donnan analysis using full factorial design. Korean Journal of Chemical Engineering, 31(3), pp. 461-466.

This study used a four-factor two-level (2^4) full factorial design to investigate the influence of different physico-chemical factors on fluoride removal efficiency and fluoride flux from diluted solutions using Donnan dialysis. Fluoride occurs naturally in the public water supply system and is one of the chemical compounds that has significant health effects through drinking water at excessive concentrations. The four factors and their levels investigated and were shown in Table 1 of the paper and are reproduced here as Table 3.9.

Table 3.9: Experimental range and levels of independent variables. Table 1 of Boubakri et al (2014).

Variable	Real values of coded levels		
	-1	0	1
C (mg/L)	5	10	15
Q (L/h)	0.4	0.7	1
A (rpm)	167	500	833
T (°C)	25	30	35

The two responses of interest were the fluoride removal efficiency (Y_F in %) and the fluoride flux (J_F in mg/cm^2.h). The materials and methods used in the experiment were described in the paper.

The full-factorial design with 4 factors required 16 runs. Four center points were added for estimating pure error and testing for curvature. The total number of run combinations was 20. Minitab 15 statistical software was used for the design and analysis of the experiment. The experimental design in terms of actual factors and results were shown in Table 2 of the paper and are reproduced here as Table 3.10.

From Table 3.10, the fluoride efficiency ranged from 34.14% to 75.52% and fluoride flux ranged from 0.196 and 2.4 mg/cm^2.h. All 14 estimated effects, regression coefficients, and their associated p-values for both fluoride removal efficiency and fluoride flux were shown in Table 2 and 3 of the paper, respectively. These tables are reproduced here as Tables 3.11 and 3.12, respectively. The R^2 values for both responses were for the full factorial model.

The authors used the p-values from Tables 3.11 and 3.12 and Pareto charts to select the final prediction models. For Y_F, the final model in terms of coded factors was given as (Equation 4 in the paper):

$$Y_F = 58.112 - 6.472C + 3.391A + 6.18T - 2.5C.Q + 1.863Q.T + 2.808\ C.Q.T + 2.999\ C.A.T$$

For J_F, the final model in terms of coded factors was given as (Equation 5 in the paper):

$$J_F = 0.846 + 0.340C + 0.367Q + 0.060A + 0.142T + 0.119C.Q + 0.046C.A + 0.124\,C.T + 0.114Q.T + 0.122C.Q.T + 0.044\,C.A.T$$

No goodness of fit statistics were reported for these reduced equations. Also note that these equations were not hierarchical. There was no mention of whether curvature was tested as part of the analysis or if any regression assumptions checks were made.

Table 3.10: Full factorial design matrix for fluoride removal efficiency. Table 2 of Boukakri et al (2014).

Run	C (mg/L)	Q (L/h)	A (rpm)	T (°C)	Y_F (%)	J_F (mg/cm^2.h)
1	5	0.4	167	25	45.40	0.196
2	15	0.4	167	25	52.27	0.680
3	5	1	167	25	58.26	0.690
4	15	1	167	25	34.14	1.060
5	5	0.4	833	25	64.38	0.270
6	15	0.4	833	25	50.64	0.660
7	5	1	833	25	67.79	0.800
8	15	1	833	25	42.58	1.280
9	5	0.4	167	35	66.74	0.284
10	15	0.4	167	35	47.92	0.630
11	5	1	167	35	75.52	0.800
12	15	1	167	35	57.96	1.950
13	5	0.4	833	35	69.33	0.280
14	15	0.4	833	35	63.24	0.830
15	5	1	833	35	69.26	0.730
16	15	1	833	35	64.81	2.400
17	10	0.7	500	30	57.98	0.840
18	10	0.7	500	30	60.49	0.920
19	10	0.7	500	30	61.05	0.920
20	10	0.7	500	30	56.48	0.850

Reanalysis of the data using Design-Expert 12 gave similar but not identical results. The standard errors of the estimated coefficients and associated p-values were quite different. However, in general the estimated regression coefficients were similar in magnitude and the models chosen by the authors gave good results.

Table 3.11: Estimated effects and coefficients for fluoride removal efficiency (coded units). Table 3 of Boubakri et al (2014).

Term	Effect	Coefficient	S.E. coef	P-value
Constant		58.112	0.5365	0.000
C	-12.945	-6.473	0.5365	0.001
Q	1.245	0.623	0.5365	0.330
A	6.783	3.392	0.5365	0.008
T	12.360	6.180	0.5365	0.001
C*Q	-5.000	-2.500	0.5365	0.019
C*A	0.572	0.286	0.5365	0.631
C*T	1.105	0.553	0.5365	0.379
Q*A	-2.032	-1.016	0.5365	0.154
Q*T	3.725	1.863	0.5365	0.040
A*T	-2.542	-1.271	0.5365	0.152
C*Q*A	2.615	1.308	0.5365	0.099
C*Q*T	5.615	2.808	0.5365	0.014
C*A*T	5.998	2.999	0.5365	0.011
Q*A*T	-2.188	-1.094	0.5365	0.134
C*Q*A*T	-2.337	-1.169	0.5365	0.117
R-sq = 0.9932				

Table 3.12: Estimated effects and coefficients for fluoride flux (coded units). Table 4 of Boubakri et al (2014).

Term	Effect	Coefficient	S.E. coef	P-value
Constant		0.846	0.01087	0.000
C	0.680	0.340	0.01087	0.000
Q	0.735	0.368	0.01087	0.000
A	0.120	0.060	0.01087	0.012
T	0.283	0.142	0.01087	0.001
C*Q	0.237	0.119	0.01087	0.002
C*A	0.092	0.046	0.01087	0.024
C*T	0.249	0.125	0.01087	0.001
Q*A	0.057	0.029	0.01087	0.077
Q*T	0.229	0.115	0.01087	0.002
A*T	0.024	0.012	0.01087	0.350
C*Q*A	0.065	0.033	0.01087	0.058
C*Q*T	0.243	0.122	0.01087	0.002
C*A*T	0.088	0.044	0.01087	0.027
Q*A*T	-0.011	-0.006	0.01087	0.634
C*Q*A*T	0.014	0.007	0.01087	0.566
R-sq = 0.9990				

Case Study #3.4

Golshani, T., Jorjani, E., Chelgani S. Chehreh, Shafaei, S. Z., and Nafechi Y. Heidari (2013): Modeling and process optimization for microbial desulfurization of coal by using a two-level full factorial design. International Journal of Mining Science and Technology, 23, pp. 261-265.

This study used a five-factor two-level (2^5) full factorial design to model and optimize coal microbial desulfurization conditions from the Tabas coal preparation plant in Iran. The goals of the study were to determine the effects and interactions on the total sulfur reduction and to maximize the reduction of sulfur from high sulfur content coal samples. The five factors and their levels used in the experiment were shown in Table 2 of the paper and are reproduced here as Table 3.13.

Table 3.13: Variables, symbols, and levels used for full factorial design. Table 2 of Golshani et al (2013).

Variable	Symbol	Lowe level	Center level	High level
		-1	0	1
pH	A	1.5	2.0	2.5
Particle size (μm)	B	180	340	500
Fe^{2+} (mmol)	C	0	30	60
Pulp density (%)	D	2	6	10
Leaching time (d)	E	6	10	14

The response of interest was the sulfur reduction (%). The methods and materials used in the experiment were described in the paper. Design-Expert 7.0 statistical software was used for the design and analysis of the experiment. The full-factorial design with 5 factors required 32 runs. The experimental design in terms of actual factors and results were shown in Table 3 of the paper and are reproduced here as Table 3.14.

The response ranged from 22.47% to 53.12%. The authors used a half-normal plot of effects to select the significant effects for the prediction model. The final suggested model (Equation 3 in the paper) in actual factors was given as:

$$\begin{aligned}\log_{10}(\text{sulfur reduction}) = {} & 1.4379 - 0.06128\ \text{pH} - 1.18057 \times 10^{-4}\ \text{particle size} \\ & + 8.44246 \times 10^{-3}\ \text{pulp density} + 0.21607\ \text{time}\end{aligned}$$

The ANOVA results for the above reduced first order model were shown in Table 4 of the paper and are reproduced here as Table 3.15. The results were in log base 10 units. As can be seen from the ANOVA results, all terms were statistically significant at the 5% level. Factor C (Fe^{2+}) was not statistically significant at the 5% level and no two-factor interaction terms were included. The goodness of fit statistics showed that this model had a R^2 of 91.19% and a adjusted R^2 of 89.89%.

Table 3.14: Results of experiments for sulfur removal in different operating levels regard to full factorial design. Table 3 of Golshani et al (2013).

Test No.	Run	pH	Particle size (μm)	Fe^{2+} (mmol)	Pulp density (%)	Time (d)	Sulfur reduction (%)
1	1	1.5	180	0	2	6	29.76
2	11	2.5	180	0	2	6	25.56
3	25	1.5	500	0	2	6	28.87
4	2	2.5	500	0	2	6	24.65
5	30	1.5	180	60	2	6	29.75
6	18	2.5	180	60	2	6	24.35
7	10	1.5	500	60	2	6	26.24
8	5	2.5	500	60	2	6	22.47
9	32	1.5	180	0	10	6	34.52
10	24	2.5	180	0	10	6	29.85
11	29	1.5	500	0	10	6	35.88
12	21	2.5	500	0	10	6	25.36
13	8	1.5	180	60	10	6	34.26
14	3	2.5	180	60	10	6	34.62
15	27	1.5	500	60	10	6	31.35
16	12	2.5	500	60	10	6	24.08
17	26	1.5	180	0	2	14	42.31
18	7	2.5	180	0	2	14	38.03
19	6	1.5	500	0	2	14	40.76
20	31	2.5	500	0	2	14	35.31
21	19	1.5	180	60	2	14	42.88
22	20	2.5	180	60	2	14	39.27
23	9	1.5	500	60	2	14	39.92
24	16	2.5	500	60	2	14	37.53
25	15	1.5	180	0	10	14	48.47
26	28	2.5	180	0	10	14	38.50
27	17	1.5	500	0	10	14	43.78
28	13	2.5	500	0	10	14	37.58
29	22	1.5	180	60	10	14	53.12
30	14	2.5	180	60	10	14	51.97
31	23	1.5	500	60	10	14	48.82
32	4	2.5	500	60	10	14	46.82

Reanalysis of the data using Design-Expert 12 obtained identical results to those reported. However, a better model can be obtained without transforming the response and including the C, CD, and CE which were all statistically significant at the 5% level. Furthermore, the predicted R^2 would increase from 0.876 to 0.913. Hence all five main effects were statistically significant at the 5% level and there were two two-factor interaction terms. Concluding that Factor C (Fe^{2+}) was not statistically significant may be in error.

Table 3.15: Analysis of variance for sulfur reduction. Table 4 of Golshani et al (23013).

Source	Sum of squares	df	Mean square	F value	p-value
Model	0.320	4	0.079	69.89	<0.0001 S
A-pH	0.030	1	0.030	26.50	<0.0001
B-Particle size	0.011	1	0.011	10.07	0.0037
D-Pulp density	0.036	1	0.036	32.18	<0.0001
E-Time	0.240	1	0.240	210.82	<0.0001
Residual	0.031	27	1.13E-03		
Cor total	0.350	31			

S: significant, CV=2.18%, R^2=91.19%, Adj. R^2=89.89%

The authors then used the optimization routine in Design-Expert 7 together with the developed model to determine the conditions that will maximize sulfur reduction. The optimum conditions obtained were pH of 1.5, particle size of 180 μm, iron sulfate concentration of 2.67 (mmol/L), pulp density of 10%, and bioleaching time of 14 days. The predicted sulfur reduction was 51.47%. The experimental result at these optimal conditions was 52.89%.

Since the iron sulfate concentration factor was not statistically significant according to the author's model, it could have been set to 0 instead of 2.67.

Case Study #3.5

Khademi, A., Nafiseh G. Renani, Maryam Mofarrahi, Alireza Rangraz Jeddi, and Noordin M. Yusof (2013): The best location for speed bump installation using experimental design methodology. Promet – Traffic and Transportation, Vol. 25, No. 6, pp. 565-574.

This study used a four-factor two-level (2^4) full factorial design to determine the optimum location to install speed bumps before stopping points along a road to control traffic speeds through critical areas. Speed bumps are well-known traffic calming techniques used around the world. The factors and levels considered in this study are shown in Table 3.16.

Table 3.16: Factors and levels used in the speed bump installation experiment.

Factor	Symbol	Low Level (-1)	Mid Level (0)	High Level (+1)
Car weight (No. of passengers)	A	1	3	5
Car speed (km/h)	B	10	20	30
Distance (m)	C	10	15	20
Surface inclination (%)	D	0	3.5	7

The response was supposed to be the speed at the stop point. However, due to the lack of a proper speed measurement instrument, the time in seconds taken between the bump and the stop point was taken as the response. The experimental procedure used was described in the paper. A two-level full factorial design with four factors would require 16 run combinations. The authors considered three replications for a total of 3 x 2^4 = 48 runs. Due to the large number of runs, the experiments were conducted over two days. Each day was considered as a block using I=ABCD as the block generator. Two center points were also added to each block to check for curvature. Hence, the total number of runs was 52.

Design-Expert 8 was used for the experimental design and analysis. The design and responses were shown in Table 2 of the paper and are reproduced here as Table 3.17.

Table 3.17: Response factors, which were measured in the performed actual experimental design. Table 2 of Khademi et al (2013).

Std	Treatment Combination	ABCD Factorial Effect	Factors				No. of replicates (Time (s))			Total	Average	Standard Deviation
			A: Car weight (No. of passengers)	B: Car Speed (km/h)	C: Distance (m)	D: Surface Inclination (%)	R1	R2	R3			
1	(1)	+	1	10	10	0	2.18	4.90	3.26	10.34	3.45	1.37
2	a	-	5	10	10	0	3.05	3.69	3.35	10.09	3.36	0.32
3	b	-	1	30	10	0	1.20	1.35	1.68	4.23	1.41	0.25
4	ab	+	5	30	10	0	1.36	1.26	1.71	4.33	1.44	0.24
5	c	-	1	10	20	0	4.31	7.76	6.56	18.63	6.21	1.75
6	ac	+	5	10	20	0	4.41	7.95	7.72	20.08	6.69	1.98
7	bc	+	1	30	20	0	3.08	3.05	3.29	9.42	3.14	0.13
8	abc	-	5	30	20	0	3.13	3.16	3.37	9.66	3.22	0.13
9	d	-	1	10	10	7	4.34	2.79	3.91	11.04	3.68	0.80
10	ad	+	5	10	10	7	3.53	4.27	3.87	11.67	3.89	0.37
11	bd	+	1	30	10	7	1.65	1.66	1.96	5.27	1.76	0.18
12	abd	-	5	30	10	7	1.70	1.58	2.14	5.42	1.81	0.29
13	cd	+	1	10	20	7	8.12	8.42	8.64	25.18	8.39	0.26
14	acd	-	5	10	20	7	5.71	8.93	10.28	24.92	8.31	2.35
15	bcd	-	1	30	20	7	3.45	3.18	3.05	9.68	3.23	0.20
16	abcd	+	5	30	20	7	3.20	3.24	3.46	9.90	3.30	0.14
17	CP	0	3	20	15	3.5	2.79	3.41	3.44	9.64	3.21	0.37
18	CP	0	3	20	15	3.5	3.46	5.13	4.29	12.88	4.29	0.84
19	CP	0	3	20	15	3.5	3.05	3.41	3.14	9.60	3.20	0.19
20	CP	0	3	20	15	3.5	4.53	3.41	2.96	10.90	3.63	0.81

From Table 3.17, there were 60 data points in total because three replications were also used for the center points. No explanation was given on how the center point results were used in the analysis as there should be only 52 points used in the analysis.

In the first round of analysis, the authors found that an inverse square-root transformation of the response was required to meet the assumptions of regression. The ANOVA results with only the statistically significant effects at the 5% level and the goodness of fit statistics were shown in Table 6 of the paper and are reproduced here as Table 3.18. The regression coefficient estimates and associated standard errors were shown in Table 7 of the paper and are reproduced here as Table 3.19.

Table 3.18: New ANOVA Table (Partial sum of squares - Type III). Table 6 of Khademi et al (2013).

Source	Sum of Squares	d.f.	Mean Square	F Value	p-value (Prob > F)	
Block	5.37E-05	1	5.368E-05			
Model	1.11	4	0.28	118.03	<0.0001	significant
B- Car Speed	0.59	1	0.59	249.45	<0.0001	significant
C - Distance	0.48	1	0.48	203.16	<0.0001	significant
D - Surface Inclination	0.026	1	0.026	11.07	0.0018	significant
BC	0.020	1	0.020	8.44	0.0057	significant
Curvature	4.768E-03	1	4.768E-03	2.02	0.1619	not significant
Residual	0.11	45	2.360E-03			
Lack of fit	0.013	11	1.200E-03	0.44	0.9260	not significant
Pure Error	0.093	34	2.73E-03			
Cor. Total	1.22	51				
Stad Dev.	0.049	R^2		0.9094		
Mean	0.56	Adj R^2		0.9015		
C.V. %	8.70	Pred. R^2		0.8840		
PRESS	0.14	Adeq. Precision		28.1740		

Note: After Inverve square-root transform of Time.

Table 3.19: New Post ANOVA Table. Table 7 of Khademi et al (2013).

Factor	Coefficient estimate	d.f.	Standard error	95% CI (Low)	95% CI (High)	VIF
Intercept	0.56	1	6.81E-03	0.55	0.58	
Day 1	1.02E-03	1				
Day 2	-1.02E-03					
B - Car Speed	0.110	1	7.09E-03	0.10	0.12	1
C - Distance	-0.100	1	7.09E-03	-0.11	-0.086	1
D - Surface Inclination	-0.023	1	7.09E-03	-0.04	-9.05E-03	1
BC	-0.020	1	7.09E-03	-0.03	-6.10E-03	1

Note: After inverse square-root transform of Time.

From Table 3.19, the final empirical models in terms of coded and actual factors were given as (Equations 3 and 4 in the paper, respectively):

Coded:

1/Sqrt(Time) = 0.56 + 0.11 B – 0.10 C – 0.02 C – 0.023 D – 0.020 BC

Actual:

1/Sqrt(Time) = 0.54379 + 0.017176 x Car speed – 0.011835 x Distance
– 3.10876E-0.003 x Surface inclination – 4.07165E-004 x Car Speed x Distance

Eight confirmation experiments were then performed to confirm the accuracy of the model developed. The run combinations and results were shown in Tables 8 of the paper. The comparison of the average experimental results and model results were shown in Table 9 of the paper and are reproduced here as Table 3.21.

Table 3.20: Treatment combinations used for confirmation experiments. Table 8 of Khademi et al (2013).

Run	Factors				No. of replicates (Time (sec))			Total	Average
	A: Car Weight (No. of passengers)	B: Car Speed (km/h)	C: Distance (m)	D: Surface Inclination (%)	R_1	R_2	R_3		
1	5	10	20	7	5.71	8.68	9.98	24.38	8.13
2	1	10	20	7	8.42	8.54	8.15	25.11	8.37
3	5	10	15	0	4.73	4.71	4.78	14.23	4.74
4	1	10	15	0	5.00	4.76	5.14	14.9	4.97
5	5	20	10	0	1.76	2.27	2.14	6.17	2.06
6	1	20	10	0	1.95	2.00	2.19	6.14	2.05
7	1	20	20	0	4.65	3.83	4.14	12.62	4.21
8	1	20	15	0	2.13	2.29	2.20	6.62	2.21

Table 3.21: Confirmation experiments. Table 9 of Khademi et al (2013).

Run	Factors				Average (Actual Time (Sec)	Prediction (Time (sec))	Residual	Error (%)	95% PI low	95% PI high
	A: Car Weight (No. of passengers)	B: Car Speed (km/h)	C: Distance (m)	D: Surface Inclination (%)						
1	5	10	20	7	8.13	8.12	0.00	0.06	6.26	10.96
2	1	10	20	7	8.37	8.12	0.25	2.98	5.78	12.24
3	5	10	15	0	4.74	4.39	0.35	7.35	3.68	5.34
4	1	10	15	0	4.97	4.39	0.58	11.53	3.64	5.41
5	5	20	10	0	2.06	2.11	0.06	2.91	1.85	2.44
6	1	20	10	0	2.05	2.11	0.07	3.28	1.84	2.46
7	1	20	20	0	4.21	4.20	0.01	0.12	3.56	5.04
8	1	20	15	0	2.21	2.05	0.16	7.05	1.80	2.36

The two runs were the same combinations as those performed previously while the next six run combinations were within the range of the levels used. The maximum time between the starting point after the speed bump and the stop point was reported by the authors as 8.13 sec although Table 3.21 showed the maximum time was 8.37 sec. The optimum conditions found were a low car speed, 20 m distance, and high inclination.

Reanalysis of the provided data using Design-Expert 12 produced similar but not identical results. This is likely due to the uncertainty of which center point data were used in the analysis.

Case Study #3.6

Mtaallah, S., Ikhlass Marzouk, and Bechir Hamrouni (2018): Factorial experimental design applied to adsorption of cadmium on activated alumina. Journal of Water Reuse and Desalination, 08.1, pp. 76-85.

In this study a four-factor two-level (2^4) full factorial design was used to investigate the influence of four factors on the removal efficiency of cadmium from aqueous solutions and industrial effluents by adsorption on activated alumina. Cadmium, a toxic heavy metal, adversely affects humans, animals, and plants. The materials and methods used in the experiment were described in the paper. The factors and levels used in the experiment were shown in Table 3 of the paper and are reproduced here as Table 3.22.

Table 3.22: Experimental ranges and levels of the factors studied in the factorial design. Table 3 of Mtaallah et al (2018).

Variables	Factors	Low level	High level
X_1	Dose AA (g) (A)	0.5	1.5
X_2	Initial Cd(II) concentration ([Cd], mg/L) (B)	10	100
X_3	pH (pH) (C)	5	8
X_4	Temperature (T, °C) (D)	10	40

The response of interest was the percentage removal of cadmium II (%Cd) as defined in the paper. Minitab 16 statistical software was used for the design and analysis of the experiment. The full-factorial design with 4 factors required 16 runs. Two center points were added to test for curvature and provide a measure of pure error. The experimental design in terms of actual factors and results were shown in Table 4 of the paper and are reproduced here as Table 3.23. The response ranged from 16.24% to 99.09%.

The reduced ANOVA results were shown in Table 5 of the paper and are reproduced here as Table 3.24. No goodness of fit statistics were given. The model representing Cd(II) removal was expressed as (Equation 3 of the paper):

$$\%Cd = 63.25 + 14.67X_1 - 20.23X_2 - 5.17X_3 + 13.54X4 + 2.3X_1X_2 + 3.45X_1X_3 + 0.89X_1X_4 + 0.83X_2X_3 + 0.64X_3X_4 + 3.29X_2X_4.$$

As can be seen from Table 3.23, only factors A, B, and D were statistically significant at the 5% level, all other terms could have been left out of the model.

Reanalysis of the data using Design-Expert 12 showed that the model suggested by the authors had a predicted R^2 of -0.1036 indicating that the model was no better than using the overall mean response. Furthermore, the lack of fit was also statistically significant and a highly statistically significant term (p-value <0.0001), ABD, was not included in the model.

Table 3.23: Studied parameters in their reduced and normal forms. Table 4 of Mtaallah et al (2018).

Experiment	A	X_1	B	X_2	C	X_3	D	X_4	%Cd
1	0.5	-1	10	-1	5	-1	10	-1	68.38
2	1.5	1	10	-1	5	-1	10	-1	94.62
3	0.5	-1	100	1	5	-1	10	-1	23.34
4	1.5	1	100	1	5	-1	10	-1	35.78
5	0.5	-1	10	-1	8	1	10	-1	35.78
6	1.5	1	10	-1	8	1	10	-1	94.13
7	0.5	-1	100	1	8	1	10	-1	16.24
8	1.5	1	100	1	8	1	10	-1	29.83
9	0.5	-1	10	-1	5	-1	40	1	95.88
10	1.5	1	10	-1	5	-1	40	1	99.09
11	0.5	-1	100	1	5	-1	40	1	41.22
12	1.5	1	100	1	5	-1	40	1	89.08
13	0.5	-1	10	-1	8	1	40	1	84.43
14	1.5	1	10	-1	8	1	40	1	95.5
15	0.5	-1	100	1	8	1	40	1	23.34
16	1.5	1	100	1	8	1	40	1	85.75
17	1	0	55	0	6.5	0	25	0	73.54
18	1	0	55	0	6.5	0	25	0	73.21

Table 3.24: ANOVA of the 2^4 design. Table 5 of Mtaallah et al (2018).

Term	Sum of Squares	Degrees of freedom	Mean square	F-value	p-value
A	3443.34	1	3443.340	10.25589	0.023929
B	6548.05	1	6548.050	19.50316	0.006916
C	428.9	1	428.900	1.27748	0.309653
D	2933.31	1	2933.310	8.73676	0.031672
A X B	85.47	1	85.470	0.25457	0.635319
A X C	190.58	1	190.580	0.56763	0.485145
A X D	12.92	1	12.920	0.03849	0.852180
B X C	11.26	1	11.260	0.03353	0.861912
B X D	173.32	1	173.320	0.51622	0.504623
C X D	6.68	1	6.680	0.01990	0.893317
Error	1678.71	5	335.743		
Total sum of squares	15512.54	15			

The authors concluded that the highest percentage removal of cadmium was obtained at a temperature of 40C, adsorbent dose of 1.5 g, and initial cadmium concentration of 10mg/L.

Case Study #3.7

Nasirabadi, P. S., M. Jabbari, and J. H. Hattel (2017): CFD simulation and statistical analysis of moisture transfer into an electronic enclosure. Applied Mathematical Modelling, 44, pp. 246-260.

This study used a four-factor two-level (2^4) full factorial design and computational fluid dynamics (CFD) to investigate the moisture transfer into a typical electronic enclosure. The factors and levels investigated were shown in Table 2 of the paper and are reproduced here as Table 3.25. The CFD simulations were conducted using the COMSOL Multiphysics version 5.1 software package. Both isothermal and non-isothermal studies were carried out using the package. The geometry of the electronic enclosure and the equations used in the modelling were described in the paper.

Table 3.25: The studied ranges of the parameters in the factorial design. Table 2 of Nasirabadi et al (2017).

Factor	Notification	Coded symbol	Low level	High level	Unit
Length of the opening (or tube)	L	A	2.00	50.00	[mm]
Radius of the opening	R	B	0.50	5.00	[mm]
Temperature	T	C	273.15	333.15	[K]
Initial RH	RH	D	40.00	80.00	[%]

The main response of interest was the diffusion time of moisture into the enclosure at constant ambient temperature and relative humidity. The four-factor full factorial design with two levels required 16 run combinations. A center point was added to check for curvature. Note that only one center point was added because this was a computer based experiment with no random error. The software used for experimental design and statistical analysis was not reported in the paper. The experimental design and results were shown in Table 4 of the paper and are reproduced here as Table 3.26.

Preliminary analysis showed that the response needed a logarithmic transformation to meet the assumptions of regression. The ANOVA results after a log-transform (base 10) of the response were shown in Table 5 of the paper and reproduced here as Table 3.27. Table 5 in the paper was wrongly captioned. The caption should read "ANOVA results" and should not be identical to Table 4 of the paper.

The ANOVA results showed that only factors A, B and D were statistically significant at the 5% level and curvature was also statistically significant. The proposed regression model in terms of actual factors was (Equation 17 of the paper):

$$\text{Log(Response [s])} = 7.52153 + 0.022926 \times \text{L [mm]} - 0.39190 \times \text{R [mm]} - 8.03937 \times 10^{-3} \times \text{RH [\%]}$$

The goodness of fit statistics were given as $R^2 = 0.9363$ and adjusted $R^2 = 0.9216$.

Table 3.26: The factorial design table for the factors and the responses. Table 4 of Nasirabadi et al (2017).

Case #	A [mm]	B [mm]	C [K]	D [%]	Response (diffusion time) [s]
1	2	0.5	273.15	40	8,500,000.00
2	50	0.5	273.15	40	208,000,000.00
3	2	5	273.15	40	143,000.00
4	50	5	273.15	40	1,474,000.00
5	2	0.5	333.15	40	6,210,000.00
6	50	0.5	333.15	40	149,450,000.00
7	2	5	333.15	40	464,000.00
8	50	5	333.15	40	1,544,000.00
9	2	0.5	273.15	80	4,892,000.00
10	50	0.5	273.15	80	166,460,000.00
11	2	5	273.15	80	108,000.00
12	50	5	273.15	80	1,095,000.00
13	2	0.5	333.15	80	3,815,000.00
14	50	0.5	333.15	80	28,500,000.00
15	2	5	333.15	80	72,000.00
16	50	5	333.15	80	880,000.00
17	26	2.75	303.15	60	18,318,000.00

Table 3.27: The ANOVA table for the response. Table 5 of Nasirabadi et al (2017).

Source	Sum of squares	Degree of freedom	Mean square	F-value	p-value (Prob>F)	
Model	17.7	3	5.90	104.85	<0.0001	Significant
A	4.84	1	4.84	86.09	<0.0001	Significant
B	12.44	1	12.44	221.11	<0.0001	Significant
D	0.41	1	0.41	7.35	0.0189	Significant
Curvature	0.53	1	0.53	9.39	0.0098	Significant
Residual	0.68	12	0.056			
Total	18.90	16				

Reanalysis of the data using Design-Expert 12 gave practically identical ANOVA results to those reported but the intercept term in the regression equation was 7.47745 instead of the reported 7.52153. The R^2 and adjusted R^2 values were 0.9633 and 0.9541, respectively which were a little larger than those reported.

Since the curvature was statistically significant at the 5% level, a better fit to the data would likely be obtained using a response surface model.

Case Study #3.8

Ridzuan, N., F. Adam, and Z. Yaacob (2016): Screening of factor influencing wax deposition using full factorial experimental design. Petroleum Science and Technology, Vol. 34, No. 1, pp. 84-90.

This study used a four-factor two-level (2^4) full factorial design to investigate the rate of wax deposition of Malaysia crude oil under the influence of four parameters or factors. The factors were the speed of rotation of the impeller, the cold finger temperature, experimental duration, and inhibitor concentration. The factors and levels used for the experiment are summarized in Table 3.28. The experimental setup and materials used were described in the paper.

Table 3.28: Factors and levels used in the wax deposition experiment.

Factor	Symbol	Levels		
		Low level (-1)	Mid level (0)	High level (+1)
Speed of rotation, rpm	A	0	300	600
Cold finger temperature, °C	B	5	10	15
Experimental duration, h	C	2	13	24
Inhibitor concentration, ppm	D	200	2600	5000

The response of interest was the wax deposition (g). The 2^4 experiment required 16 run combinations and three center points were added to check for curvature and as a measure of pure error. Hence a total of 19 runs was used. Design-Expert 7.1.6 software was used for the experimental design and statistical analysis.

The experimental design in coded and actual factors, and results were shown in Table 1 of the paper and are reproduced here as Table 3.29. Wax deposition ranged from 0.75 g to 3.0 g. The partial ANOVA results were shown in Table 2 of the paper and are reproduced here as Table 3.30.

The authors proposed the following regression model for wax deposition (in coded factors) (Equation 2 of the paper):

$$\text{Wax deposit} = 1.68 + 0.12 - 0.60 + 0.34\,C - 0.059\,D + 0.059\,AD - 0.12\,BC - 0.053\,BD$$

The R^2 value was reported as 0.9795. No other goodness of fit statistics were reported. Note that from the ANOVA table, effect D, AD, and BD were not statistically significant at the 5% level. Furthermore, the curvature was statistically significant at the 5% level. The authors did not discuss the statistically significant curvature term and did not provide reasons for including the three insignificant terms in the model. This experiment should be followed up with a response surface experiment to account for the curvature effect.

Table 3.29: Results for the screening design according to standard order. Table 1 of Ridzuan et al (2016).

	Factors				
	A	B	C	D	
Standard order	Uncoded/ coded	Uncoded/ coded	Uncoded/ coded	Uncoded/ coded	Wax deposit, g
1	0 (-1)	5	2	200	1.90
2	600 (+1)	5	2	200	1.80
3	0 (-1)	15	2	200	0.80
4	600 (+1)	15	2	200	1.00
5	0 (-1)	5	24	200	2.65
6	600 (+1)	5	24	200	2.80
7	0 (-1)	15	24	200	1.40
8	600 (+1)	15	24	200	1.60
9	0 (-1)	5	2	5000	1.50
10	600 (+1)	5	2	5000	2.10
11	0 (-1)	15	2	5000	0.75
12	600 (+1)	15	2	5000	0.90
13	0 (-1)	5	24	5000	2.50
14	600 (+1)	5	24	5000	3.00
15	0 (-1)	15	24	5000	1.05
16	600 (+1)	15	24	5000	1.20
17	300 (0)	10	13	2600	1.40
18	300 (0)	10	13	2600	1.50
19	300 (0)	10	13	2600	1.45

Table 3.30: Analysis of variance. Table 2 of Ridzuan et al (2016).

Source	Sum of squares	DF	Mean square	F value	p-value Prob>F	% Contribution
Model	8.14	7	1.16	68.220	<0.0001	
A - Speed of rotation	0.21	1	0.21	12.540	0.0053	2.54
B - Cold finger temperature	5.70	1	5.70	334.290	<0.0001	67.65
C - Experimental duration	1.86	1	1.86	108.870	<0.0001	22.03
D - Inhibitor concentration	0.06	1	0.06	3.310	0.099	0.67
AD	0.06	1	0.06	3.310	0.099	0.67
BC	0.21	1	0.21	12.540	0.0053	2.54
BD	0.05	1	0.05	2.650	0.1347	0.54
Curvature	0.11	1	0.11	6.600	0.0279	
Residual	0.17	10	0.017			
Lack of fit	0.16	8	0.02	4.350	0.2003	
Pur error	9.27E-03	2	4.63E-03			
Cor total	8.43	18				

Case Study #3.9

Shah, Mumtaj and S. K. Garg (2014): Application of 2^k full factorial design in optimization f solvent-free microwave extraction of ginger essential oil. Journal of Engineering, Article ID 828606, 5 pages.

This study used three-factor two-level full factorial design to determine the optimum conditions for a solvent-free microwave extraction of essential oil from ginger. Solvent-free microwave extraction (SFME) combines microwave heating with distillation to extract essential oils from plant materials such as aromatic and medicinal plants. The factors and levels used in the experiment were shown in Table 1 of the paper and are reproduced here as Table 3.31. The materials and methods used in the experiment were described in the paper.

Table 3.31: Coded and natural variables in 2^3 factorial design. Table 1 of Shah et al (2014).

Levels	X_1 (min)	X_2 (watt)	X_3
Basic level (0)	20	464	-
High level (+1)	30	640	Sliced
Low level (-1)	10	288	Crushed
Interval	10	176	-

X_1:extraction time (min); $X_{2:}$microwave power (watt); $X_{3:}$ sample type.

The response of interest is the oil yield (%) from fresh ginger. Design-Expert 8.0.7 software was used for the design and analysis of the experiment. The authors used two replications per run combination hence 16 runs were conducted. The experimental design and results were shown in Table 2 of the paper and are reproduced here as Table 3.32.

Table 3.32: The 2^3 factorial design including corresponding responses. Table 2 of Shah et al (2016).

	Coded variables			Oil yield
Run	x_1 (extraction time)	x_2 (microwave power)	x_3 (sample type)	y (%)
1	-1	-1	-1	0.10
2	1	-1	-1	0.26
3	-1	1	-1	0.14
4	1	1	-1	0.35
5	-1	-1	1	0.22
6	1	-1	1	0.20
7	-1	1	1	0.28
8	1	1	1	0.44
9	-1	-1	-1	0.12
10	1	-1	-1	0.25
11	-1	1	-1	0.14
12	1	1	-1	0.32
13	-1	-1	1	0.24
14	1	-1	1	0.17
15	-1	1	1	0.31
16	1	1	1	0.46

The ANOVA results were shown in Table 3 of the paper and are reproduced here as Table 3.33. All seven effects were statistically significant at the 5% level.

Table 3.33: Analysis of variance. Table 3 of Shah et al (2016).

Source	Sum of squares	Degree of freedom (df)	Mean square	F value	P-value prob > F	Remark
Model	0.17	7	0.024	97.83	<0.0001	Significant
A-extraction time	0.051	1	0.051	202.50	<0.0001	
B - power	0.048	1	0.048	193.60	<0.0001	
C - sample	0.026	1	0.026	102.40	<0.0001	
AB	0.016	1	0.016	62.50	<0.0001	
AC	0.013	1	0.013	52.90	<0.0001	
BC	0.012	1	0.012	48.40	0.0001	
ABC	5.625E-03	1	5.63E-03	22.50	0.0015	
Pure error	2.000E-03	8	2.500E-04			
Cor total	0.17	15				

The regression model for oil yield was given as (Equation 4 in the paper):

$$Y \text{ (oil yield)} = 0.25 + 0.056X_1 + 0.055X_2 + 0.040X_3 + 0.031X_1X_2 - + 0.028X_2X_3 - 0.029X_1X_3 + 0.019\,X_1X_2X_3$$

The R^2 and adjusted R^2 were 0.9885 and 0.9783, respectively. The predicted R^2 was also close to the adjusted R^2. Regression assumptions were checked and were found to be fulfilled.

Optimization of the oil yield was then carried out using the desirability function approach available in Design-Expert. The settings for the various factors and results were shown in Table 4 of the paper and are reproduced here as Table 3.34.

Table 3.34: Setting goal for each factor and response for formulation of optimization and selected optimized conditions. Table 4 of Shah et al (2016).

Factors/response	Goal	Selected optimum conditions: Coded	Actual
Extraction time (min)	In range	1	30 min
Microwave power (watt)	In range	1	640 watts
Sample type	In range	1	Crushed sample
Oil yield	Maximum	0.45%	0.45%

The maximum oil yield achieved was 0.45% at the high settings of all three factors. However, no confirmation experiments were carried out to validate the predicted results.

Reanalysis of the data using Design-Expert 12 obtained identical results as reported by the authors.

Case Study #3.10

Yusoff, N. H. N., M. J. Ghazali, M. C. Isa, A. R. Daud, A. Muchtar, and S.M. Forghani (2012): Optimization of plasma spray parameters on the mechanical properties of agglomerated Al_2O_3-13%TiO_2 coated mild steel. Materials and Design, 39, pp. 504-508.

This study used a three-factor two level (2^3) full factorial design to investigate the effect of plasma spray parameters of deposited agglomerated nano Al_2O_3-13%TiO_2 powders on commercial marine-grade mild steels. The parameters or factors and levels used in the experiment were shown in Table 1 of the paper and are reproduced here as Table 3.35. The experimental procedures and materials used in the experiment were described in the paper.

Table 3.35: Experimental range and levels of independent parameters for plasma spray coating. Table 1 of Yusoff et al (2012).

Independent parameters	Range (coded level)		
	Low (-1)	Central point (0)	High (+1)
Primary gas pressure (X_1) psi	40	80	120
Carrier gas pressure (X_2) psi	20	32.5	45
Powder feed rate (X_3) rpm	1	2	3

Three responses of interest were – Y_1, microhardness; Y_2, specific wear rate; and Y_3, surface roughness. Design-Expert 6.0.10 software was used for the design and analysis of the experiment. The authors added one center point to the 2^3 design resulting in 9 runs. The experimental design and results were shown in Table 2 of the paper and are reproduced here as Table 3.36.

Table 3.36: Full-factorial design for plasma spray experiment. Table 2 of Yusoff et al (2012).

Run	X_1	X_2	X_3	Y_1	Y_2	Y_3
1	-1	1	1	350	0.0240	9.88
2	1	1	1	555	0.0068	9.52
3	-1	1	-1	375	0.0176	9.19
4	-1	-1	1	413	0.0219	7.49
5	1	-1	1	1057	0.0023	8.36
6	1	1	-1	958	0.0012	11.29
7	1	-1	-1	714	0.0050	10.00
8	-1	-1	-1	965	0.0004	7.43
9	0	0	0	473	0.0499	9.63

The partial ANOVA results and estimated regressions coefficients in coded units were shown in Table 3 of the paper and are reproduced here as Table 3.37.

Table 3.37: Full 2^3 factorials design (Coefficient are given in coded unit). Table 3 of Yusoff et al (2012).

Term	SS	Coefficient	P value
(a) for Y_1			
X_1	5.60E+05	-264.88	0.0004
(b) For Y_2			
X_1	1.37E-03	0.013	0.0122
(c) For Y_3			
X_1	0.097	-0.11	0.42
X_2	1.59	0.45	0.06
X_2	0.41	0.23	0.18
X_1X_2	6.93	-0.93	0.01
X_2X_3	3.18	0.63	0.03

The final regression models in coded factors with statistically significant terms at the 5% level were given as (Equations 2 to 4 of the paper):

Y_1 = microhardness = 663.13 – 260.38 X_1

Y_2 = specific wear rate = 0.015 + 0.013 X_1

Y_3 = surface roughness = 9.16 + 0.45 X_2 – 0.93 X_1X_3 + 0.63 X_2X_3

Goodness of fit statistics for these models were not reported. Also note from Table 3.37, Y_3, X_2 was not statistically significant at the 5% level.

The authors found that the optimum properties of the coatings with the best wear resistance, surface roughness, and microhardness occurred at the lowest primary pressure of 40 psi, carrier gas pressure of 20 psi, and the highest powder feed rate of 3 rpm.

Reanalysis of the given data using Design-Expert 12 did not reproduce the same ANOVA results or estimated regression coefficients. Hence, the results reported may not be valid.

4. FRACTIONAL FACTORIAL DESIGNS

Nine case studies using two-level fractional factorial or 2^{k-p} designs are presented in this Chapter. The number of factors ranges from five to seven and fractions range from half to one-sixteenth.

Case Study #4.1

Chen, J. P., S. L. Kim, and Y. P. Ting (2003): Optimization of membrane physical and chemical cleaning by a statistical designed approach. Journal of Membrane Science, 219, pp. 27-45.

In this study, the authors used several 2-level fractional factorial designs (2^{k-p}) to investigate the efficiency of physical and chemical cleaning of ultrafiltration (UF) and reverse osmosis (RO) membranes in municipal wastewater reclamation. The responses measured were the clean water flux recovery in percentage and the wash water usage. The definitions of these responses and the technical specifications of the two types of membranes were given in the paper.

For the physical cleaning experiment of the UF and RO membranes, the authors first used a resolution III 2^{6-3} design with eight runs followed up with a complete foldover to give a resolution IV design with 16 runs. The same strategy was used for the chemical cleaning of RO membranes. For the chemical cleaning of UF membranes, only five factors were involved. Hence a 2^{5-2} design was used followed by a complete foldover giving 16 runs. The authors did not mention the defining relationships used for the fractional factorial design nor the statistical software used for design and analysis of the experiments.

For this case study, only the physical and chemical cleaning experiments for the UF membranes are presented. The other experiments are similar and hence will not be described herein. The designs and results can be found in the paper.

The first experiment investigated six factors that affect the physical cleaning of the UF membrane. The levels and factors considered were listed in Table 1 of the paper and are reproduced here as Table 4.1.

Table 4.1: Factors and levels for the physical cleaning of UF membranes (PC-UF).

Factor	Details	Low Level (-)	High level (+1)
A	Production interval between physical cleaning (h)	0.5	3
B	Duration of forward flush (min)	1	5
C	Duration of backwash (min)	1	5
D	Pressure during forward flush (kPa)	172	345
E	Type of water used	RO permeate	Tap water
F	Sequence of forward flush and backwash	Backwash followed by forward flush	Forward flush followed by backwash

The experimental design, a 2^{6-3} design with a complete foldover, and results were shown in Table 2 of the paper. It is adapted here in Table 4.2. The two responses CWF recovery (%) and wash water usage are labelled here as y_1 and y_2, respectively.

Table 4.3: Experimental design and results in physical cleaning of UF (PC-UF). Adapted from Table 2 of Chen et al (2003).

Run	Factors						CWF %	WWU
	A	B	C	D	E	F	y_1	y_2
1	-1	-1	-1	1	1	1	82.54	0.15
2	1	-1	-1	-1	-1	1	80.56	0.03
3	-1	1	-1	-1	1	-1	92.89	0.32
4	1	1	-1	1	-1	-1	75.80	0.08
5	-1	-1	1	1	-1	-1	88.73	0.34
6	1	-1	1	-1	1	-1	86.34	0.08
7	-1	1	1	-1	-1	1	94.89	0.48
8	1	1	1	1	1	1	87.19	0.12
9	1	1	1	-1	-1	-1	88.02	0.11
10	-1	1	1	1	1	-1	87.94	0.58
11	1	-1	1	1	-1	1	82.21	0.08
12	-1	-1	1	-1	1	1	88.73	0.34
13	1	1	-1	-1	1	1	73.03	0.09
14	-1	1	-1	1	-1	1	87.24	0.34
15	1	-1	-1	1	1	-1	76.18	0.03
16	-1	-1	-1	-1	-1	-1	90.48	0.10

The authors used the normal probability plot of effects to select the statistically significant effects. No ANOVA results were shown and the significance level was not mentioned. From the normal effects plots, the following models (in coded values) were suggested for CWF recovery % (y_1), and wash water usage (y_2) (these were Equations 5a and 5b in the paper):

$$y_1 = 85.17 - 4.090\, x_1 + 2.749\, x_3 - 1.611\, x_4 + 1.801\, x_1x_3$$

$$y_2 = 0.309 - 0.209\, x_1 + 0.131\, x_2 + 0.070\, x_3 - 0.016\, x_1 x_2 - 0.026\, x_1x_3$$

where, the variables x_1, x_2, x_3 and x_4 represent values of A, B, C, and D, respectively. The terms x_1x_2 and x_1x_3 represent the interaction between A and B, and A and C, respectively. No goodness of fit statistics were given.

For the chemical cleaning of UF membranes, five factors were studied. The factors and levels are shown in Table 4.3.

Table 4.3: Factors and levels for the chemical cleaning of UF membranes (CC-UF).

Factor	Details	Low Level (-)	High level (+1)
A	Recirculation duration of high pH cleaning (min)	30	60
B	Concentration of high pH cleaning solution (%)	0.5	1
C	Temperature of high pH cleaning solution (°C)	25	50
D	Static soak (min)	0	30
E	Forward flush or backwash after chemical cleaning	Backwash	Forward flush

The authors first used a 2^{5-2} design followed by a complete foldover. The experimental design and results were shown in Table 4 of the paper and are reproduced here as Table 4.4. For this experiment only one response, the CWF recovery % was measured. It is labelled as y_5 in Table 4.4.

Table 4.4: Experimental design and results of chemical cleaning of UF (CC-UF). Adapted from Table 4 of Chen et al (2003).

Run	Factors					CWF %
	A	B	C	D	E	y_5
1	-1	-1	-1	1	1	72.16
2	1	-1	-1	-1	-1	81.05
3	-1	1	-1	-1	1	89.47
4	1	1	-1	1	-1	94.85
5	-1	-1	1	1	-1	96.84
6	1	-1	1	-1	1	92.39
7	-1	1	1	-1	-1	97.94
8	1	1	1	1	1	92.39
9	1	1	1	-1	-1	100.00
10	-1	1	1	1	1	91.58
11	1	-1	1	1	-1	97.83
12	-1	-1	1	-1	1	84.21
13	1	1	-1	-1	1	84.54
14	-1	1	-1	1	-1	89.69
15	1	-1	-1	1	1	78.95
16	-1	-1	-1	-1	-1	82.47

From the normal probability plot of effects, the authors suggested the follow regression equation for CWF recovery % (y_5) (Equation 7 in the paper):

$$y_5 = 89.15 - 3.409\, x_2 + 5.000\, x_3 - 3.436\, x_5 - 2.076\, x_2x_3$$

where the factors involved are B(x_2), C(x_3), and E(x_5). No ANOVA results or goodness of fit statistics were given.

Overall, the authors found that the application of factorial design to optimize physical and chemical cleaning of both UF and RO membranes was successful.

Case Study #4.2

Choi, Y.E., Y-S Yun, and J.M. Park (2002): Evaluation of factors promoting Astaxanthin production by a unicellular green alga, *Haematococcus pluvialis,* with fractional factorial design. Biotechnology Progress, 18, pp. 1170-1175.

This study evaluated seven factors that affect astaxanthin production by a unicellular green alga, *Haematococcus pluvialis* UTEX 16 using a sequential 2-level fractional factorial design. According to the authors, astaxanthin has great commercial value due to its high price. It is used mainly as a source of pigmentation in the aquaculture industry. The production of astaxanthin is a function of many factors specified by the authors in Table 1 of the paper and adapted here in Table 4.5.

Table 4.5: Specification of factors and levels used in the experiments. Table 1 of Choi et al, 2002).

Production Methods (Factors)	Variables (units)	Low level (-1)	High level (+1)
Nitrogen deficiency (N)	nitrogen concentration (mM)	4.06	0
Phosphorus deficiency (P)	phosphorus concentration (mM)	0.21	0
High irradiation (L)	photon flux density (μE $m^{-2}s^{-1}$)	100	500
Magnesium deficiency (M)	magnesium concentration (mM)	1	0
Acetate addition (A)	acetate concentration (mM)	0	15
Ferrous ion addition (F)	ferrous concentration (mM)	0	0.45
Salt addition (S)	NaCl concentration (mM)	OHM	25

Note: Plus signs represent the application of astaxanthin methods; minus signs means no use of the methods. When the astaxanthin production methods were not applied (-1), the optimal *Haematococcus* medium (OHM) was used. For the application of methods A, F, and S, CH_3COONa, $FeSO_4$ and NaCl were used, respectively. The materials and methods used in the experiments were described in detail in the paper.

The authors first used a resolution III 2^{7-4} fractional factorial design with 8 runs. The defining relationship used was the one recommended in standard textbooks. They then augmented the design to a resolution IV with a full foldover giving 16 runs. The experimental design and the responses were shown in Table 2 of the paper and are reproduced here in Table 4.6. Two responses were measured, the weight content (mg/g) and the cellular content (pg/cell). The first eight runs were based on a resolution III design, and the next eight runs were from the complete foldover.

Apparently the authors did not use any statistical software to design the experiment or analyze the results. Manual calculations and the normal probability plot of effects were used and the effects were only compared to the size of the standard error. Hence there were no ANOVA results or regression models produced. The calculated effects and standard errors for the augmented fractional factorial design with 16 runs were given in Table 3 of the paper and are shown here as Table 4.7.

Table 4.6: First fractional factorial design and responses. Table 2 of Choi et al (2002).

	Coded units of variables							Responses	
Expt No.	N	P	L	M	A	F	S	weight content (mg/g)	cellular content (pg/cell)
1	-1	-1	-1	1	1	1	-1	4.2	10.8
2	1	-1	-1	-1	-1	1	1	4.4	24.9
3	-1	1	-1	-1	1	-1	1	7.8	27.3
4	1	1	-1	1	-1	-1	-1	14.9	36.3
5	-1	-1	1	1	-1	-1	1	25.3	112.6
6	1	-1	1	-1	1	-1	-1	26.7	159.3
7	-1	1	1	-1	-1	1	-1	23.9	145.2
8	1	1	1	1	1	1	1	21.9	243.2
9	1	1	1	-1	-1	-1	1	24.3	72.1
10	-1	1	1	1	1	-1	-1	20.5	112.2
11	1	-1	1	1	-1	1	-1	10.8	22.5
12	-1	-1	1	-1	1	1	1	20.8	149.7
13	1	1	-1	-1	1	1	-1	13.5	140.1
14	-1	1	-1	1	-1	1	1	10.3	47.3
15	1	-1	-1	1	1	-1	1	23	153.2
16	-1	-1	-1	-1	-1	-1	-1	12.1	35.2

Table 4.7: Calculated effects and standard errors from first fractional factorial design based on the assumption that interactions between three or more factors are negligible. Table 3 of Choi et al (2002).

	estimate ± standard error	
Effects	weight content (mg/g)	cellular content (pg/cell)
Average	16.5 ± 1.0	93.2 ± 15.3
N	1.8 ± 1.9	26.4 ± 30.5
P	1.2 ± 1.9	19.4 ± 30.5
L	10.5 ± 1.9	67.7 ± 30.5
M	-0.3 ± 1.9	-2.0 ± 30.5
A	1.6 ± 1.9	62.5 ± 30.5
F	-5.6 ± 1.9	9.5 ± 30.5
S	1.4 ± 1.9	21.1 ± 30.5
PM+LA+FS	-0.2 ± 1.9	15.6 ± 30.5
NM+LF+AS	0.8 ± 1.9	16.7 ± 30.5
NA+PF+MS	6.1 ± 1.9	72.6 ± 30.5
NP+LS+AF	1.2 ± 1.9	13.5 ± 30.5
NL+PS+MF	-3.5 ± 1.9	-32.1 ± 30.5
NS+PL+MA	0.5 ± 1.9	12.7 ± 30.5
NF+PA+LM	-4.0 ± 1.9	-7.0 ± 30.5

According to the authors, only positive effects on the responses are important. Hence, for the weight content, from Table 4.7, the effects L, and the aliased interaction term NA+PF+MF are deemed significant since their effect estimates are larger than the standard error. Other effects were deemed not significant or they contribute a negative amount. For the response cellular content, the effects L, A, and the same aliased interaction term NA+PF+AF, are significant as they contribute positively to that response. However, the NA+PF+AF effect could also be due to the interactions of P and F or A and F, besides N and A. The authors reasoned that the NA interaction was most likely to be responsible for the positive effect of the confounded interaction NA+PF+AF.

To confirm the author's suspicion, these interactions had to be de-aliased. To achieve this, the authors performed a single-factor foldover on factor A. This means that A was no longer aliased with other factors and was also de-aliased from all interactions involving A.

The results of the new set of 8 experiments folding over on A alone was shown in Table 4 of the paper and are shown here as Table 4.8. The combined data from the first 8 runs of the first experiment (Table 4.6) and from Table 4.8 were reanalyzed. The estimated effects and standard errors were shown in Table 5 of the paper and are reproduced here as Table 4.9.

Table 4.8: Additional fractional factorial design and responses. Table 4 of Choi et al (2002).

	Coded units of variables							Responses	
Expt No.	N	P	L	M	A	F	S	weight content (mg/g)	cellular content (pg/cell)
17	-1	-1	-1	1	-1	1	-1	13.9	129.5
18	1	-1	-1	-1	1	1	1	9.3	268.8
19	-1	1	-1	-1	-1	-1	1	22	127.4
20	1	1	-1	1	1	-1	-1	20.8	135.9
21	-1	-1	1	1	1	-1	1	24.3	112.3
22	1	-1	1	-1	-1	-1	-1	27.6	119.7
23	-1	1	1	-1	1	1	-1	10.2	221.7
24	1	1	1	1	-1	1	1	2.8	35.3

Table 4.9: Calculated effects and standard errors from the additional fractional factorial design and the first half of the first fractional factorial design based on the assumption that interactions between three or more factors are negligible. Table 5 of Choi et al (2002).

	estimate ± standard error	
Effects	weight content (mg/g)	cellular content (pg/cell)
Average	16.2 ± 1.0	119.3 ± 15.3
N+PM+FS	-0.4 ± 1.9	17.1 ± 30.5
P+NM+LF	-1.4 ± 1.9	4.3 ± 30.5
L+PF+MS	8.2 ± 1.9	48.6 ± 30.5
M+NP+LS	-0.5 ± 1.9	-34.8 ± 30.5
A	-1.2 ± 1.9	56.1 ± 30.5
F+NS+PL	-9.9 ± 1.9	31.1 ± 30.5
S+NF+LM	-3.1 ± 1.9	-0.8 ± 30.5
LA	2.1 ± 1.9	24.9 ± 30.5
AS	3.4 ± 1.9	31.8 ± 30.5
NA	8.5 ± 1.9	91.7 ± 30.5
AF	1.4 ± 1.9	46.3 ± 30.5
NL+PS+MF	-0.8 ± 1.9	-25.7 ± 30.5
MA	4.8 ± 1.9	-8.9 ± 30.5
PA	0.5 ± 1.9	14.9 ± 30.5

From Table 4.9, one can see that A and all its interactions are now clear of other factors and interactions. The NA interaction effect is larger than the standard error for both responses and the AF interaction effect is larger than the standard error for the cellular content response.

Combining the results from the first and second fractional factorial designs (Tables 4.7 and 4.9), the authors concluded that for weight content, high irradiation (L) and the interaction between nitrogen deficiency and acetate addition (NA) were most effective in increasing the responses. For cellular content, NA and the interaction between acetate addition and ferrous ion addition (AF), along with single-factors of high irradiation (L) and acetate addition (A), were most effective in increasing the responses. These observations, based on the estimated effects, were confirmed by the authors using normal probability plots of effects.

The authors stated that the approach they took which composed of three resolution III 2^{7-4} parts required only 24 runs. This was much fewer than if a full factorial design was used which would have required 128 runs.

Case Study #4.3

Farris, S. and Luciano Piergiovanni (2008): Effects of ingredients and process conditions on 'Amaretti' cookies characteristics. International Journal of Food Science and Technology, 43, pp. 1395-1403.

This study was concerned with the use of fractional factorial design on the factors that determine the quality and taste of the well-known Italian 'Amaretti' cookies during their shelf life. The characteristics studied included: hardness, moisture content, water activity, and colour of the cookies. The authors considered five controllable factors in their experiment and decided to study these factors using a half fraction of a 2-level factorial design i.e. a 2^{5-1} design. The factors and levels used were given in Table 2 of the paper and are reproduced here as Table 4.10. For this experiment, the authors made some modifications to the original recipe by adding fibre and using fructose/saccharose (F/S) mixture instead of only saccharose. The level used in the original recipe was also shown in the table.

Table 4.10: Factors and their levels for both the 2^{5-1} design and the original recipe. Table 2 of Farris et al (2008).

		Symbol		Levels			
Variable	Unit	Coded	Uncoded	-1	0	+1	Original recipe
Fibre	g	X_1	Fib	20	25	30	None
F/S	g/g	X_2	F/S	0.1	0.24	0.38	Only saccharose (650)
Egg white	g	X_3	Egg	250	257.5	265	255
Temperature	°C	X_4	Temp.	165	180	195	200
Time	Min	X_5	Time	18	20	22	20

Four responses were measured in the experiment: Y_1, Hardness (N-mm) – measured using a food texture analyzer; Y_2, Moisture content (% H_2O) – measured using Gravimetric analysis; Y_3, Water activity – measured with an electronic hygrometer; and Y_4, Colour – measured using an observer reflection colorimeter. These were described in the paper.

The authors chose a resolution V 2^{5-1} design that required 16 runs. Five center points were added to measure pure error giving a total of 21 experiments. This design would allow all main effects and two-factor interactions to be estimated without bias. The experimental design and results were shown in Table 3 of the paper and are reproduced here as Table 4.11.

The authors used MODDE Version 8 software package by UMETRICS AB, Umea, Sweden for the evaluation of the raw data and regression analysis using the least squares method. A two-factor interaction model was fitted to each of the responses and only regression coefficients significant at the 5% level were selected for developing the final regression models.

The estimated coefficients of the fitted equations for the different responses were shown in Table 4 of the paper and are reproduced here as Table 4.12.

Table 4.11: Worksheet of the 2^{5-1} fractional factorial design. Table 3 of Farris et al (2008).

		Variable levels					Responses			
Std order	Run	X_1	X_2	X_3	X_4	X_5	Y_1	Y_2	Y_3	Y_4
1	6	20	0.1	250	165	22	19.81	15.00	0.698	85.46
2	5	30	0.1	250	165	18	15.30	15.27	0.722	86.42
3	1	20	0.38	250	165	18	13.70	18.33	0.775	86.00
4	16	30	0.38	250	165	22	35.30	17.47	0.761	80.66
5	20	20	0.1	265	165	18	9.90	17.00	0.745	82.91
6	21	30	0.1	265	165	22	29.95	15.16	0.698	86.65
7	13	20	0.38	265	165	22	19.42	18.99	0.759	82.61
8	17	30	0.38	265	165	18	16.99	18.75	0.777	84.12
9	4	20	0.1	250	195	18	44.27	14.96	0.723	75.18
10	7	30	0.1	250	195	22	56.32	13.59	0.664	61.22
11	10	20	0.38	250	195	22	45.97	15.50	0.714	53.24
12	18	30	0.38	250	195	18	39.02	16.63	0.724	63.04
13	8	20	0.1	265	195	22	32.71	12.31	0.712	62.49
14	11	30	0.1	265	195	18	34.66	15.33	0.708	69.41
15	15	20	0.38	265	195	18	30.10	17.08	0.741	57.75
16	3	30	0.38	265	195	22	46.73	16.01	0.704	49.86
17	19	25	0.24	257.5	180	20	31.83	15.99	0.756	74.82
18	9	25	0.24	257.5	180	20	31.20	16.58	0.725	75.48
19	14	25	0.24	257.5	180	20	29.41	16.61	0.742	71.77
20	12	25	0.24	257.5	180	20	29.66	16.64	0.737	73.33
21	2	25	0.24	257.5	180	20	31.18	16.26	0.748	75.39

Y_1 = Hardness, Y_2 = moisture content, Y_3 = water activity, Y_4 = colour

Table 4.12: Estimated significant coefficients of the fitted equations for the different responses. Table 4 of Farris et al (2008).

	Estimated coefficients			
Factors	Y_1	Y_2	Y_3	Y_4
X_0	30.6395000	-1.92335000	0.73014300	0.4690960
X_1	3.6493800	-0.0003385*	-0.0068125*	-0.004462*
X_2	0.269378*	0.00651313	0.01781250	-0.0685789
X_3	-3.0768700	0.00128602	0.0039375*	-0.0212241
X_4	10.5881000	-0.00471553	-0.01531250	-0.2626120
X_5	5.1418700	-0.00298404	-0.01281250	-0.0534031
X_1X_3	0.8756250	0.0001942*	-0.0019376*	0.0256817
X_1X_4	-0.6893750	0.00140833	-0.0044375*	-0.009472*
X_1X_5	2.6493800	0.0005585*	-0.0001875*	-0.007490*
X_2X_4	-1.0368800	-0.0007490*	-0.0083125*	-0.0358650
X_2X_5	0.8093770	0.0011490*	0.0029375*	-0.012742*
X_3X_4	-2.0956200	-0.00122528	0.0010626*	-0.010905*
X_3X_5	-0.496874*	-0.0006443*	0.0005625*	0.0292494
X_4X_5	-0.9318760	-0.00121300	0.0000625*	-0.0379559

Y_1 = Hardness, Y_2 = moisture content, Y_3 = water activity, Y_4 = colour
*Not statistically significant coefficients at $P \leq 0.05$ (or 95% CI).

The interaction terms X_1X_2 and X_2X_3 were removed beforehand because they were deemed statistically insignificant at the 5% level. However, many of the coefficients were also identified as statistically insignificant in addition to the two removed in Table 4.12. Furthermore, one can see that the coefficients given for Y_2 and Y_4 seem to be clearly wrong by orders of magnitude. The coefficients for Y_1 and Y_3, however, are correct.

The goodness-of-fit statistics for the four responses after model refinement (based on the terms in Table 4.12) were given in Table 5 of the paper, and are shown here as Table 4.13.

Table 4.13: Summary list for the four model parameters after model refinement. Table 5 of Farris et al (2008).

	Parameter				
Response	R^2	R^2adj.	Q^2	Model validity	Reproducibility
Hardness	0.977	0.991	0.930	0.794	0.992
Moisture content	0.980	0.943	0.584	0.558	0.968
Water activity	0.886	0.675	-1.023	0.529	0.828
Colour	0.995	0.986	0.965	0.941	0.980

The statistic Q^2, called the power of prediction (an uncommon term), is likely equivalent to the more commonly used term, the predicted R^2. The term model validity and reproducibility were not defined in the paper.

The ANOVA results for Y_1, Y_2, and Y_4 are shown in Table 6 of the paper. No suitable model was found for Y_3, the water activity (negative Q^2). These results used all terms except the two two-factor interactions X_1X_2 and X_2X_3. The ANOVA results are reproduced here in Table 4.14.

Table 4.14: ANOVA table for the response of hardness, moisture content and colour. Adapted from Table 6 of Farris et al (2008).

		Y_1 (Hardness)			Y_2 (moisture content)			Y_4 (colour)		
Source of variation	DF	SS	MS	F	SS	MS	F	SS	MS	F
Total	21	22553.1	1073.96		77.699	3.699		5.934	0.282	
Constant	1	19714.4	19714.4		77.684	77.684		4.621	4.621	
Tot. corrected	20	2838.71	141.936		0.001	6.82E-05		1.312	0.065	
Regression	13	2830.45	217.727	184.52	0.001	0.0001	26.83	1.306	0.100	109.78
Residual	7	8.259	1.179		2.68E-05	3.83E-06		0.006	0.0009	
Lack of fit (model error)	3	3.766	1.255	1.11	1.82E-05	6.07E-06	2.81	0.001	0.0004	0.35
Pure error (replicate error)	4	4.493	1.123		8.63E-06	2.15E-06		0.005	0.001	
$F_{0.95;\,13;7} = 3.55$		$R^2 = 0.997$			$R^2 = 0.980$			$R^2 = 0.995$		
$F_{0.95;\,3;4} = 6.59$										

A closer look at the ANOVA results showed that the results for Y_2 and Y_4 are also incorrect by an order of magnitude. It seems that the authors may have used the wrong data in their analysis. In addition, no test for curvature was done.

The final prediction models for the three responses suggested by the authors were:

$$\text{Hardness } (Y_1) = 30.64 + 3.65\, X_1 - 3.08\, X_3 + 10.59\, X_4 + 5.14\, X_5 + 0.88\, X_1X_3 - 0.69\, X_1X_4 + 2.65\, X_1X_5 - 1.04\, X_2X_4 + 0.81X_2X_5 - 2.10\, X_3X_4 - 0.93\, X_4X_5$$

$$\text{Moisture content } (Y_2) = -1.92 + 0.00651\, X_2 + 0.00129\, X_3 - 0.00471\, X_4 - 0.00298\, X_5 + 0.00141\, X_1X_4 - 0.00122\, X_3X_4 - 0.00121\, X_4X_5$$

$$\text{Colour } (Y_4) = 0.47 - 0.069\, X_2 - 0.021\, X_3 - 0.263\, X_4 - 0.053\, X_5 + 0.025\, X_1X_3 - 0.035\, X_2X_4 + 0.029\, X_3X_5 - 0.038\, X_4X_5$$

No goodness-of-fit statistics were given for these reduced models. Also, it should be noted that the models given are not hierarchical and may not lead to the correct predictions. As mentioned earlier, all the coefficients for Y_2 and Y_4 are incorrect.

Case Study #4.4

Hajeeh, M. (2003): Estimating corrosion: a statistical approach. Materials and Design, 24, pp. 509-518.

This study used a fractional factorial design to investigate the factors that affect the corrosivity of seawater on aluminum-brass (Al-brass) and carbon steel (C-S) alloy pipes. The original plan was to investigate seven factors using a half-fraction of a two-level factorial design i.e. 2^{7-1} design with 64 runs. However, the author found that since the corrosion rates for the two alloys were very different and the error variance for C-S alloy was larger than that of the Al-brass alloy, difficulties might arise in the interpretation of the estimated effects. Hence, the experiment was decomposed into two separate six-factor 2^{6-1} designs instead of using alloy type as one of the factors. The six factors and levels used in the two designs were given in Table 1 of the paper and are reproduced here as Table 4.15.

Table 4.15: Variables of the experiments and their levels. Table 1 of Hajeeh (2003).

Variable	Units	Low level	High level
Temperature (T)	°C	30	45
Oxygen (O_2)		Deaerated	Aerated
Urea (U)	ppm	0	3.5
Inhibitor (I)	ppm	0	50
Sulfide (S)	ppm	0	2
Chloride (C)	ppm	19500	23000

The 2^{6-1} design is a resolution VI design, where all main effects and two-factor interactions are not aliased with any other effects or interactions. However, three-factor interactions are aliased with other three-factor interactions. Although not mentioned in the paper, the defining relationships used for the Al-brass was I=ABCDEF, and for the C-S was I = -ABCDEF. The response measured for each experiment was the corrosion rate in mils per year (mpy). The experimental design and the corrosion rates for the Al-brass alloy and C-S alloy were shown in Tables 2 and 3 of the paper and are reproduced here as Tables 4.16 and 4.17.

Table 4.16: Experimental design and corrosion rates for Al-brass. Table 2 of Hajeeh (2003).

Expt No.	Temperature (°C)	Oxygen (ppm)	Urea (ppm)	Sulfide (ppm)	Inhibitor (ppm)	Chloride (ppm)	Corrosion rate (mpy)	Replication
1	30	D	0	0	0	19500	1.1	
2	45	A	0	0	0	19500	0.9	0.7
3	45	D	3.5	0	0	19500	0.3	
4	30	A	3.5	0	0	19500	0.6	0.9
5	45	D	0	2	0	19500	0.7	
6	30	A	0	2	0	19500	1.1	
7	30	D	3.5	2	0	19500	1.6	
8	45	A	3.5	2	0	19500	1.6	1.1
9	45	D	0	0	50	19500	0.2	
10	30	A	0	0	50	19500	1.4	
11	30	D	3.5	0	50	19500	1.1	
12	45	A	3.5	0	50	19500	0.2	0.2
13	30	D	0	2	50	19500	0.4	
14	45	A	0	2	50	19500	0.9	
15	45	D	3.5	2	50	19500	0.4	
16	30	A	3.5	2	50	19500	0.5	
17	45	D	0	0	0	23000	1.3	
18	30	A	0	0	0	23000	0.7	0.7
19	30	D	3.5	0	0	23000	0.3	
20	45	A	3.5	0	0	23000	1.3	
21	30	D	0	2	0	23000	1.1	
22	45	A	0	2	0	23000	1.1	
23	45	D	3.5	2	0	23000	0.2	
24	30	A	3.5	2	0	23000	1.0	1.6
25	30	D	0	0	50	23000	0.1	
26	45	A	0	0	50	23000	0.1	
27	45	D	3.5	0	50	23000	0.1	
28	30	A	3.5	0	50	23000	0.6	
29	45	D	0	2	50	23000	0.3	
30	30	A	0	2	50	23000	0.6	
31	30	D	3.5	2	50	23000	0.9	0.24
32	45	A	3.5	2	50	23000	0.5	

A (aerated): the environment is full of oxygen, D (deaerated): the environment does not contain any oxygen.

Table 4.17: Experimental design and corrosion rates for carbon steel (C-S) alloy. Table 3 of Hajeeh (2003).

Expt No.	Temperature (°C)	Oxygen (ppm)	Urea (ppm)	Sulfide (ppm)	Inhibitor (ppm)	Chloride (ppm)	Corrosion rate (mpy)	Replication
1	45	D	0	0	0	19500	3.1	1.8
2	30	A	0	0	0	19500	4.3	3.6
3	30	D	3.5	0	0	19500	1.0	
4	45	A	3.5	0	0	19500	6.9	3.5
5	30	D	0	2	0	19500	0.9	
6	45	A	0	2	0	19500	0.3	4.7
7	45	D	3.5	2	0	19500	2.7	
8	30	A	3.5	2	0	19500	4.0	7.1
9	30	D	0	0	50	19500	5.1	
10	45	A	0	0	50	19500	6.9	
11	45	D	3.5	0	50	19500	1.7	
12	30	A	3.5	0	50	19500	4.5	
13	45	D	0	2	50	19500	7.1	
14	30	A	0	2	50	19500	5.4	9.2
15	30	D	3.5	2	50	19500	4.1	
16	45	A	3.5	2	50	19500	10.1	
17	30	D	0	0	0	23000	0.3	
18	45	A	0	0	0	23000	8.2	6.0
19	45	D	3.5	0	0	23000	2.6	
20	30	A	3.5	0	0	23000	5.3	
21	45	D	0	2	0	23000	6.0	
22	30	A	0	2	0	23000	4.8	
23	30	D	3.5	2	0	23000	3.3	
24	45	A	3.5	2	0	23000	1.1	1.6
25	45	D	0	0	50	23000	3.6	
26	30	A	0	0	50	23000	4.0	
27	30	D	3.5	0	50	23000	1.1	
28	45	A	3.5	0	50	23000	5.9	
29	30	D	0	2	50	23000	8.8	
30	45	A	0	2	50	23000	8.1	
31	45	D	3.5	2	50	23000	4.3	
32	30	A	3.5	2	50	23000	6.6	8.7

A (aerated): the environment is full of oxygen, D (deaerated): the environment does not contain any oxygen.

The effects were estimated using the GLM procedure which is part of the Statistical Analysis System package. However, the standard deviation of the effects was calculated using the replicated observations in the experiments. The calculations were shown in the Appendices of the paper. No ANOVA results, regression models, or goodness-of-fit statistics were given.

The estimated effects for Al-brass were shown in Table 4 of the paper and are reproduced here as Table 4.18.

Table 4.18: Estimates of various effects and interactions for Al-brass alloy. (Table 4 of Hajeeh (2003).

Effect/Interaction	Estimated effect	Effect/Interaction	Estimated effect
Mean	0.7250	T x O_2 x U + S x I x C	0.2750
Temperature	-0.1875	T x O_2 x S + U x I x C	0.2125
Oxygen	0.1875	T x O_2 x I + U x S x C	-0.1875
Urea	0.0500	T x U x S + O_2 x I x C	-0.0750
Sulfide	0.1625	T x U x I + O_2 x U x S	-0.0500
Inhibitor	-0.4125	T x I x C + O_2 x U x S	0.2875
T x O_2	0.2000	O_2 x U x S + T x I x C	-0.0750
T x U	0.0625	O_2 x U x I + T x S x C	-0.3250
T x S	0.0000	O_2 x I x C + T x U x S	-0.0625
T x I	-0.1750	U x S x I + T x O_2 x C	-0.1250
O_2 x U	-0.0125	I x C	-0.0625
O_2 x S	0.0250	S x C	-0.0125
O_2 x I	-0.0250	U x C	0.0000
U x S	0.1125	O_2 x C	0.0125
U x I	0.0875	T x C	0.1375
S x I	-0.0750	Chloride	-0.1750

For the Al-brass data, the author calculated the standard deviation of effects to be 0.1027 and the 95% confidence interval as ±0.243. Hence any absolute value of estimated effect that was greater than 0.243 were considered statistically significant at the 5% level. The author identified the following effects to be statistically significant at the 5% level:

a. Inhibitor
b. Oxygen × urea × inhibitor + temperature × sulfide × chloride
c. Temperature × inhibitor × sulfide + urea × inhibitor × chloride
d. Temperature × oxygen × urea + sulfide × inhibitor × chloride
e. Temperature × inhibitor × chloride + oxygen × urea × sulfide

The author also identified those effects that were statistically significant at the 10% and 20% levels in the paper.

The estimate for the temperature × inhibitor × sulfide + urea × inhibitor × chloride interaction is missing from Table 4.18. In any case, the three-factor interaction of temperature × inhibitor × sulfide should be aliased with oxygen × urea × chloride and not urea × inhibitor × chloride. There are actually several other typographical errors or wrong estimates in Table 4.18.

For the carbon-steel alloy, the effect estimates were given in Table 6 of the paper and are reproduced here as Table 4.19. The author did not explain why the aliased terms were not shown, as in Table 4.18.

Table 4.19: Estimates of main effects and interactions for carbon-steel alloy. Table 6 of Hajeeh (2003).

Effect/Interaction	Estimated effect	Effect/Interaction	Estimated effect
Mean	9.7800	T x O_2 x U	-0.0063
Temperature	1.8440	T x O_2 x S	0.2440
Oxygen	2.0990	T x O_2 x I	0.5810
Urea	-0.8310	T x U x S	-0.0063
Sulfide	1.7190	T x U x I	0.7810
Inhibitor	1.1310	T x S x I	-0.0188
T x O_2	1.0300	O_2 x U x S	0.3188
T x U	-0.3690	O_2 x U x I	1.0810
T x S	0.1810	O_2 x S x I	0.1310
T x I	-0.8310	U x S x I	0.2810
O_2 x U	0.9310	I x C	0.5810
O_2 x S	-0.9220	S x C	-0.5810
O_2 x I	-0.8560	U x C	0.0686
U x S	-0.0188	O_2 x C	0.2688
U x I	-0.5063	T x C	0.3440
S x I	0.9940	Chloride	0.2690

The standard deviation of effects was obtained using the replicated observations and was calculated to be 0.663. The 95% confidence interval was ±1.523. Based on this interval, the author identified the following effects as significant at the 5% level:

a. Oxygen
b. Temperature
c. Sulfide.

Other effects that were statistically significant at the 20% level were identified. However, the estimates given by the author are highly suspect as the mean was estimated incorrectly. It should be 4.44 and not 9.78. All other estimates seem to be incorrect as well.

The author concluded that, for the aluminum-brass alloy, the inhibitor contributed significantly to decreasing corrosion rates, and there were also various significant three-factor interactions. For carbon-steel alloy, only oxygen, temperature, and sulfide were most significant, followed by a few two-factor interactions that were moderately significant. However, given the frequency of errors in the paper, these conclusions may not be entirely correct.

There was no mention of ANOVA assumption checks or whether there were any follow-up experiments to de-alias the three-factor interactions.

Case Study #4.5

Kazemi-Beydokhti, A., Hamed Azizi Namaghi, and Saeed Zeinali Heris (2013): Identification of the key variables on thermal conductivity of CuO nanofluid by a fractional factorial design approach. Numerical Heat Transfer, Part B, 64, pp. 480-495.

This study investigated seven parameters that are responsible for change in the thermal conductivity of nanofluids containing copper oxide nano particles. The authors started by using a resolution III 2^{7-4} fractional factorial design with eight runs and three center points and then augmented with a full-foldover design of another eight runs and three center points. The total number of runs was 22. The factors and levels used in the fractional factorial experiments were given in Table 1 of the paper and are reproduced here as Table 4.20.

Table 4.20: Factors and levels for full-foldover fractional factorial design. Table 1 of Kazemi-Beydokhti et al (2013).

		Level		
Factor	Unit	-1 (Low)	0 (Center point)	+1 (High)
[A] Temperature	C	25	35	45
[B] Particle volume fraction	%vol	2	3	4
[C] APPS	nm	30	40	50
[D] pH of nanofluid		2	7	12
[E] Elapsed time	h	0	5	10
[F] Sonication time	h	1	2	3
[G] Density of nanoparticles	kg/m^3	2000	4000	6000

The initial fractional factorial design was a $1/16^{th}$ fraction of resolution III. This means that each effect will be aliased with 15 other effects. More importantly, the main effects will be aliased with two-factor interactions. The defining relationship used for the design was recommended in standard textbooks and Design-Expert version 8.0.0.6 software which was used by the authors for experimental design and analysis of the data.

The details of the experiment and how thermal conductivity measurements were taken were explained in the paper. The response used was the ratio of the thermal conductivity of the nanofluids K_{nf} to that of the base fluid, K_f; that is, K_{nf}/K_f.

The initial experimental design and the results were shown in Table 2 of the paper and are reproduced here as Table 4.21.

Table 4.21: Design layout and experimental results of 2^{7-4} fractional factorial design. Table 2 of Kazemi-Beydokhti et al (2013).

			Factor input variable							Response
Std.	Run	Block	A	B	C	D	E	F	G	K_{nf}/K_f
1	10	1	-1	-1	-1	1	1	1	-1	1.29
2	8	1	1	-1	-1	-1	-1	1	1	1.31
3	11	1	-1	1	-1	-1	1	-1	1	1.35
4	2	1	1	1	-1	1	-1	-1	-1	1.45
5	3	1	-1	-1	1	1	-1	-1	1	1.19
6	7	1	1	-1	1	-1	1	-1	-1	1.35
7	5	1	-1	1	1	-1	-1	1	-1	1.31
8	6	1	1	1	1	1	1	1	1	1.24
9	1	1	0	0	0	0	0	0	0	1.27
10	4	1	0	0	0	0	0	0	0	1.31
11	9	1	0	0	0	0	0	0	0	1.29

The full foldover of the initial design to de-alias the main effects from the two-factor interactions were shown in Table 3 of the paper. The additional 11 runs (8 factorial + 3 center points) were put in a second block. The table is reproduced here as Table 4.22.

Table 4.22: Design layout and experimental results of 2^{7-4} full-foldover fractional factorial design. Table 3 of Kazemi-Beydokhti et al (2013).

			Factor input variable							Response
Std.	Run	Block	A	B	C	D	E	F	G	K_{nf}/K_f
12	13	2	1	1	1	-1	-1	-1	1	1.4
13	22	2	-1	1	1	1	1	-1	-1	1.23
14	17	2	1	-1	1	1	-1	1	-1	1.37
15	18	2	-1	-1	1	-1	1	1	1	1.28
16	20	2	1	1	-1	-1	1	1	-1	1.42
17	14	2	-1	1	-1	1	-1	1	1	1.32
18	19	2	1	-1	-1	1	1	-1	1	1.26
19	21	2	-1	-1	-1	-1	-1	-1	-1	1.27
20	12	2	0	0	0	0	0	0	0	1.3
21	16	2	0	0	0	0	0	0	0	1.33
22	15	2	0	0	0	0	0	0	0	1.28

The new design now becomes a resolution IV design with main effects aliased with three-factor interactions. However, the two-factor interactions are still aliased with other two-factor interactions.

The authors used the standard half-normal probability plot of effects to select the likely statistically significant terms for the model. The ANOVA results using the selected terms were shown in Table 4 of the paper and are reproduced here as Table 4.23.

Table 4.23: ANOVA for selected factorial model. Table 4 of Kazemi-Beydokhti et al (2013).

Source of variation	Sum of squares	df	Mean square	F-value	p-Value	
Block	4.545×10^{-4}	1	4.545E-04			
Model	0.077	12	6.421E-03	20.17	0.0003	Significant
A	2.000E-02	1	2.000E-02	61.56	0.0001	
B	1.000E-02	1	1.000E-02	31.41	0.0008	
C	5.625E-03	1	5.625E-03	17.67	0.0040	
D	7.255E-03	1	7.255E-03	22.69	0.0021	
E	2.500E-03	1	2.500E-03	7.85	0.0264	
F	1.000E-04	1	1.000E-04	0.31	0.5927	
G	7.225E-03	1	7.225E-03	22.69	0.0021	
AC	1.225E-03	1	1.225E-03	3.85	0.0906	
AE	6.400E-03	1	6.400E-03	20.10	0.0029	
AF	4.900E-03	1	4.900E-03	15.39	0.0057	
AG	1.100E-02	1	1.100E-02	34.63	0.0006	
BD	1.225E-03	1	1.225E-03	3.85	0.0906	
Curvature	1.467E-03	1	1.467E-03	4.61	0.0690	Not significant
Residual	2.290E-03	7	3.184E-04			
Lack of fit	1.621E-04	3	5.404E-05	0.10	0.9531	Not significant
Pure error	2.067E-03	4	5.167E-04			
Cor total	0.081	21				
Std Dev.	0.021		R^2		0.9542	
Mean	1.31		Adjusted R^2		0.8856	
C.V. %	1.64		Predicted R^2		0.6908	
PRESS	0.025		Adequate Precision		15.019	

The authors then developed the prediction equation (Equation 1 in the paper) using all the selected terms shown in the ANOVA table although some of the effects such as AC and BD were not statistically significant at the 5% level.

$$K_{nf}/K_f = 1.31 + 0.035\,A + 0.025\,B - 0.019\,C - 0.021\,D - 0.013\,E + 2.5 \times 10^{-3}\,F - 0.021\,G + 8.75 \times 10^{-3}\,AC - 0.02\,AE - 0.017\,AF - 0.026\,AG - 8.75 \times 10^{-3}\,BD$$

The authors checked the assumptions of regression and found them to be satisfactory and concluded that a satisfactory model had been obtained. No further confirmation runs were used to verify the model. Also, there was no mention of whether the correct two-factor interaction terms had been used, as they were still aliased with other two-factor interaction terms.

Case Study #4.6

Levingstone, T. J., Malika Ardhaoui, Khaled Benyyounis, Lisa Looney, and Joseph T. Stokes (2015): Plasma sprayed hydroxyapatite coatings: Understanding process relationships using design of experiments analysis. Surface and Coatings Technology, 283, pp. 29-36.

This study used a fractional factorial (2^{5-2}) design to investigate the simultaneous effects of key plasma spray process parameters on hydroxyapatite coatings for biomedical applications. Hydroxyapatite is ceramic which has a similar mineral component to bone. Hence it has many applications particularly in the field of dentistry and orthopaedics. The authors considered the effects of five plasma spray process parameters on the roughness, crystallinity, and purity of hydroxyapatite coatings. The parameters and levels used in the fractional factorial design were shown in Table 1 of the paper and are reproduced here as Table 4.24.

Table 4.24: Parameter ranges selected for the screening experiment. Table 1 of Levingstone et al (2015).

Parameter	Unit	Low level (-1)	High level (+1)
A - Current	A	450	750
B - Gas flow rate	slpm/scfh	33/70	61.4/130
C - Powder feed rate	g/min	10	20
D - Spray distance	mm	80	120
E - Carrier gas flow rate	slpm/scfh	4.7/10	9.4/20

The gas flow rates were given in two different units. It seems that that authors used the second unit which is scfh or standard cubic feet per hour.

Three responses were measured for each run combination. These were roughness (μm), crystallinity (%), and purity (%). The techniques and equipment used were described in the paper.

The experimental design and the results obtained were shown in Tables 2 and 3 in the paper and are combined here as Table 4.25. The defining relationship used for the resolution III fractional factorial (2^{5-2}) design was not mentioned in the paper but it can be deduced that it was the default used in Design-Expert 7.0 software package. Three centre points were added giving 11 runs for this screening experiment.

According to the authors, the coating from experiment N1 was very thin and measurements of crystallinity and purity could not be obtained. Furthermore, the roughness values were very low. Hence the results for N1 were not used for further analysis. Only experiments N2-N11 were used.

Table 4.25: Plasma spray screening experimental design and responses. Combined results from Tables 2 and 3 of Levingstone et al (2015).

	Variables					Responses		
Exp. Name	Current - A (A)	Gas flow rate - B (scfh)	Powder feed rate - C (g/min)	Spray distance - D (mm)	Carrier gas flow rate - E (scfh)	Roughness (μm)	Crystallinity (%)	Purity (%)
N1	450	70	10	120	20	4.1		
N2	750	70	10	80	10	10.55	87.6	99.4
N3	450	130	10	80	20	6.15	65.2	97.8
N4	750	130	10	120	10	8.65	81.3	98.9
N5	450	70	20	120	10	10.48	65.2	97.6
N6	750	70	20	80	20	13.4	77.4	97.7
N7	450	130	20	80	10	7.28	77.8	98.2
N8	750	130	20	120	20	11.03	65.8	96.4
N9	600	100	15	100	15	10.65	79.9	97.4
N10	600	100	15	100	15	9.48	54.9	95.5
N11	600	100	15	100	15	10.6	76.1	97.2

The authors fitted only a main effects model to each of the responses using a backward elimination procedure in Design-Expert 7.0. A p-value of 0.01 was used. The regression models obtained for each response in coded and actual units were given in Table 4 of the paper and are reproduced below.

Coded: Roughness = 9.45 + 1.4 A – 1.17 B + 1.10 C

Actual: Roughness = 4.257 + 9.70417 E-003 x Current – 0.039146 x Gas flow rate
+ 0.21912 x Powder feed rate.

Coded: Crystallinity = 71.83 + 6.2 A – 5.16 D – 6.14 E

Actual: Crystallinity = 91.25062 + 0.041329 x Current – 0.25797 x Spray distance
-1.22939 x Carrier gas flow rate.

Coded: Purity = 97.93 – 0.46 C – 0.34 D – 0.59 E

Actual: Purity = 102.8 – 0.09125 x Power feed rate – 0.017187 x Spray distance
-0.11875 x Carrier gas flow rate

The goodness-of-fit for each of the above models were shown in Table 5 of the paper and reproduced here as Table 4.26. The authors did not mention if assumptions of regression were checked.

Table 4.26: Statistical measures of equation adequacy. Table 5 of Levingstone et al (2015).

Statistical measure	Roughness	Crystallinity	Purity
R^2	0.95	0.96	0.91
Adjusted R^2	0.92	0.92	0.85
Predicted R^2	0.82	0.81	0.56
Adequate precision	17.776	14.902	10.44

The statistical measures given in Table 4.26 show that the fitted main effects model seems to provide a good fit to the responses obtained. Hence the authors concluded that the predictive equations developed were satisfactory and provided a better understanding of the effect of plasma spray properties on the roughness, crystallinity, and purity of hydroxyapatite coatings.

However, since the experimental design was a resolution IV design, two-factor interactions were all aliased with other two-factor interactions. It is likely that there are other statistically significant two-factor interactions not included in the models. To this end, follow up experiments were alluded to by the authors.

Case Study #4.7

Lucas, Y., Antonio Domingues, Driss Driochi, and Sylvie Treuillet (2006): Design of experiments for performance evaluation and parameter tuning of a road image processing chain. EURASIP Journal on Applied Signal Processing, Article ID 48012, pp. 1-10.

This study investigated the use of design of experiments techniques in tuning a full image processing chain (IPC) which is not a straightforward task. This was applied to a road image processing chain dedicated to road obstacles detection which has eight reconfigurable parameters and can be modified at any time. These parameters were shown in Table 1 of the paper and are reproduced here as Table 4.26. The details of the IPC and these parameters were described in the paper.

Table 4.26: Factors and levels used in the experiment. (Adapted from Table 1 of Lucas et al (2006).

Factor	Parameter	Low Level	High level
X_1	Canny-Deriche filter	0.5	1
X_2	Image amplification	33	63
X_3	Edge low threshold	5	15
X_4	Edge high threshold	15	30
X_5	Contour closing	26	30
X_6	Polygonal approximation	5	6
X_7	Little chain threshold	5	10
X_8	Slope threshold	1	3

The authors proposed a quality evaluation criterion called the covering rate (r%) as the response from various combinations of the parameters. The exact definition of r was given in the paper. Basically, the criterion r is dependent on the image content. A high score means that most of the image has been extracted, hence it was expressed as a percentage.

The authors first considered a two-level fractional factorial (2^{k-p}) design with 16 trials, then a Rechtschaffer [1] design with 37 trials, and finally a three-level factorial design (3^3) with 27 trials. Since the Rechtschaffer design is outside the scope of this Chapter and the three-level design was not analyzed by the authors, only the two-level fractional factorial design is presented here.

The experimental design and response r (%) were shown in Table 2 of the paper and reproduced here as Table 4.27.

Table 4.27: Experiment matrix-fractional factorial 2^{8-4} design*: averaged outputs. Table 2 of Lucas et al (2006).

Trail	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	r (%)
1	-1	-1	-1	-1	-1	-1	-1	-1	35.535
2	-1	-1	-1	1	1	1	-1	1	40.310
3	-1	-1	1	-1	1	1	1	-1	27.859
4	-1	-1	1	1	-1	-1	1	1	42.436
5	-1	1	-1	-1	1	-1	1	1	47.328
6	-1	1	-1	1	-1	1	1	-1	30.284
7	-1	1	1	-1	-1	1	-1	1	44.034
8	-1	1	1	1	1	-1	-1	-1	37.743
9	1	-1	-1	-1	-1	1	1	1	46.517
10	1	-1	-1	1	1	-1	1	-1	40.469
11	1	-1	1	-1	1	-1	-1	1	50.680
12	1	-1	1	1	-1	1	-1	-1	33.464
13	1	1	-1	-1	1	1	-1	-1	35.169
14	1	1	-1	1	-1	-1	-1	1	49.255
15	1	1	1	-1	-1	-1	1	-1	39.715
16	1	1	1	1	1	1	1	1	44.842

*Table 2 of Lucas et al (2006) incorrectly stated that it is a 2^{8-3} design.

To obtain the response, according to the authors, each run combination was compared to 180 input images selected from a video sequence of over 30,000 city and motorway frames. Note that the experimental design was incorrectly labelled as a 2^{8-3} design. A 2^{8-3} design would need 32 runs not 16. In fact, a 2^{8-3} does not exist. The smallest number of runs possible is a 2^{8-4} design. The authors did not indicate what design generators were used to obtain the 2^{8-4} design with 16 runs. By examining the run combinations, the authors have used the following design generators: $X_5 = X_2X_3X_4$, $X_6 = X_1X_3X_4$, $X_7 = X_1X_2X_3$, $X_8=X_1X_2X_4$. The design was a resolution IV design.

Fitting a main effects model to the experimental data, the authors obtained the following prediction equation in actual units (Equation 4 in the paper):

$Y = 51.1965 + 8.65\ X_1 - 4.08\ X_6 + 4.31\ X_8$,

where, X_1, X_2, X_3 are previously defined in Table 4.26.

The significance level used was not mentioned. They chose the 3 factor model that gave the second lowest Mallow's C_p (3.48) and second highest R^2 (0.938), instead of the four factor model which gave the lowest C_p (3,36) and highest R^2 (0.950), arguing that the additional factor provided only a marginal improvement to the fit.

The authors also compared the tuning results and computing cost of eight different tuning methods of which three were based on design of experiments. The fractional factorial design required the least number of runs, and optimal tuning using only the three identified significant parameters gave the best results. One of the reasons given for the efficiency of the experimental design approach was that the IPC is globally optimized.

[1] Rechtschaffer, R. L. (1967): Saturated fractions of 2n and 3n factorial designs. Technometrics, Vol. 9, pp. 569-575.

Case Study #4.8

Ramakrishna, D. M., T. Viraraghavan, and Yee-Chung Jin (2006): Iron oxide coated sand for arsenic removal: investigation of coating parameters using factorial design approach. Practice Periodical of Hazardous, Toxic, and Radioactive Waste Management, Vol. 10, No. 4, pp. 198-206.

This study investigated seven factors that affect the iron oxide-coated sand filtration process for removing arsenic from drinking water. The seven factors were: coating pH (pHc), temperature (T), iron concentration (Fe), number of coatings (N), aging (age), pH of the solution (pHs), and mass of the adsorbent (M). The responses measured were the percentage removal of arsenic (V) and arsenic (III). The advantages of using iron oxide-coated sand was explained in detail in the paper. The experiments were carried out at room temperature in a laboratory as explained by the authors.

Minitab 14 software was used for the design and analysis of the experiment. The authors used a resolution III seven factor two-level fractional factorial (2^{7-4}) design with two replications followed up by a full-foldover of this design, making it into a resolution IV design. 32 run combinations were used, in total. The factors and levels used were given in Table 3 of the paper and are reproduced here as Table 4.28.

Table 4.28: Design parameters and their levels. Table 3 of Ramakrishna et al (2006).

Factor	Code	Low level	High level
pHc	A	2.0	12.0
T	B	110 °C	800 °C
Fe	C	0.1 M	2 M
N	D	1	2
Age	E	4 h	12 days
pHs	F	5	9
M	G	0.1 g	1 g

The first set of experiments which consisted of 16 runs (2 replicates of a 2^{7-4} design) were shown in Table 4 of the paper and are reproduced here as Table 4.29 with corrections. In Table 4 of the paper, the responses were interchanged. That is, As(V) was listed as As(III), and As (III) was listed as As(V).

Table 4.29: Fractional factorial design used in studies on removal of As(V) and As(III): Set I. Table 4 of Ramakrishna et al (2006).

	pHc	T	Fe	N	Age	pHs	M	% removal of	
Number	A	B	C	D=AB	E=AC	F=BC	G=ABC	As(V)*	AS(III)*
1	-1	-1	1	1	-1	-1	1	90.60	58.50
2	1	1	1	1	1	1	1	4.04	19.80
3	-1	1	-1	-1	1	-1	1	51.70	52.10
4	1	-1	-1	-1	-1	1	1	59.60	20.40
5	-1	1	-1	-1	1	-1	1	60.80	44.80
6	1	-1	-1	-1	-1	1	1	57.70	22.90
7	1	1	-1	1	-1	-1	-1	49.50	12.30
8	-1	1	1	-1	-1	1	-1	8.00	1.80
9	-1	1	1	-1	-1	1	-1	12.00	0.30
10	1	1	1	1	1	1	1	0.18	9.50
11	1	-1	1	-1	1	-1	-1	60.80	45.60
12	1	-1	1	-1	1	-1	-1	86.10	47.30
13	-1	-1	1	1	-1	-1	1	98.20	73.20
14	-1	-1	-1	1	1	1	-1	65.70	32.90
15	-1	-1	-1	1	1	1	-1	74.20	25.80
16	1	1	-1	1	-1	-1	-1	57.00	37.90

*Note: As(V) was incorrectly listed as As(III), and As(III) was incorrectly listed as As(V) in Table 4 of Ramakrishna et al (2006).

The first set of experiments were not statistically analyzed because all the main effects were confounded or aliased with two-factor interaction terms. To de-alias the main effects from the two-factor interactions, a full-foldover was conducted with another 16 runs. This procedure converted the design into a resolution IV design. Now the main effects were aliased with three-factor interactions which can be assumed to have negligible effect. The additional 16 runs were given in Table 5 of the paper and are reproduced here as Table 4.30.

Table 4.30: Fractional factorial design used in studies on removal of As(V) and As(III): Set II. Table 5 of Ramakrishna et al (2006).

	pHc	T	Fe	N	Age	pHs	M	% removal of	
Number	A	B	C	D=AB	E=AC	F=BC	G=ABC	As(V)*	AS(III)*
17	1	1	-1	-1	1	1	-1	9.80	6.70
18	-1	-1	-1	-1	-1	-1	-1	43.20	15.10
19	1	-1	1	1	-1	1	-1	10.70	16.40
20	-1	1	1	1	1	-1	-1	57.20	14.40
21	1	-1	1	1	-1	1	-1	7.40	8.80
22	-1	1	1	1	1	-1	-1	48.50	47.30
23	-1	-1	1	-1	1	1	1	28.90	47.30
24	1	-1	-1	1	1	-1	1	9.70	10.20
25	1	-1	-1	1	1	-1	1	12.00	28.50
26	-1	-1	-1	-1	-1	-1	-1	54.30	32.30
27	-1	1	-1	1	-1	1	1	9.60	6.70
28	-1	1	-1	1	-1	1	1	10.20	3.90
29	1	1	-1	-1	1	1	-1	5.00	4.60
30	1	1	1	-1	-1	-1	1	17.20	29.30
31	1	1	1	-1	-1	-1	1	15.20	27.40
32	-1	-1	1	-1	1	1	1	33.30	56.70

The authors did not provide any ANOVA results but provided the estimated effects, coefficients, and p-values for the main effects of both responses in Table 6 of the paper, which is reproduced here as Table 4.31.

Table 4.31: Estimated effects, coefficients, and p-values. Table 6 of Ramakrishna et al (2006).

	As(V)			As(III)		
Term	Effect	Coeff	p-value	Effect	Coeff	p-value
Constant		37.80	0.000		26.9	0.000
pHc	-17.78	-8.89	**0.043**	-10.34	-5.17	**0.042**
Temp	-23.53	-11.77	**0.009**	-13.94	-6.97	**0.008**
Fe	-3.23	-1.62	0.701	9.16	4.58	0.070
N	0.07	0.04	0.993	-3.03	-1.52	0.536
Age	0.47	0.24	0.955	7.89	3.95	0.115
pHs	-25.98	-12.99	**0.005**	-18.23	-9.12	**0.001**
M	-5.65	-2.83	0.503	10.11	5.05	**0.047**

Note: The p-values in boldface are less than the level of significance chosen (0.05).

From Table 4.31, the authors identified pHc, temperature, and pHs as statistically significant factors for As(V) removal, and pHc, temperature, pHs, and M as statistically significant factors for

As(III) removal. They provided the following fitted regression models for the percentage removal of As(V) and As(III) (Equations 1 and 2 in the paper, respectively):

Percent removal of As(V) = 37.8 – 8.89 pHc – 11.8 T – 1.62 Fe + 0.04 N + 0.24 Age
– 12.99 pHs – 2.83 M

Percent removal of As(III) = 26.9 – 5.17 pHc – 6.97 T + 4.58 Fe – 1.52 N + 3.95 Age
– 9.12 pHs + 5.05 M

Note that the authors included all terms in the model regardless of whether they were statistically significant at the 5% level or not. No goodness-of-fit statistics were given except that both regression models were statistically significant at the 5% level.

The authors concluded that the experiments were a success and only 32 experiments were needed instead of a full factorial design which would require 2^7 or 128 experiments.

Case Study #4.9

Samad, K. A., and Norazwina Zainol (2017): The use of factorial design for ferulic acid production by co-culture. Industrial Crops and Products, 95, pp. 202-206.

This study investigated seven factors that influence the use of co-culture in the production of ferulic acid from banana stem waste. The authors used a seven-factor one-eighth fractional factorial (2^{7-3}) design. The seven factors and the levels used in the 2^{7-3} design were shown in Table 1 of the paper and are reproduced here as Table 4.32.

Table 4.32: Variables and their coded and actual levels used in the method of 2^4 fractional factorial design. Table 1 of Samad et al (2017).

				Actual values of coded levels		
No.	Variables	Coded	Type of factor	-1	+1	Units
1	Temperature	A	Numerical	26	40	°C
2	pH	B	Numerical	5.5	9.5	pH units
3	Agitation	C	Numerical	0	150	rpm
4	Water-to-substrate ratio	D	Numerical	2:1	10:1	w/w
5	Volume of inoculum	E	Numerical	2	10	% v/v
6	Fermentation time	F	Numerical	24	72	hours
7	Type of co-culture	G	Categorical	A[a]	B[a]	

[a] Co-culture A (*B cerus, B. pumilus and B. thuringiensis*) and B (*B. cereus and B. thuringiensis*).

The authors referred to the design used as a 2^4 fractional factorial design. It is more proper and conventional to write it as a 2^{7-3} design so that the number of factors involved and the fraction used are clearly indicated. This is a resolution IV design in which main effects are aliased with three-factor interactions and two-factor interactions are aliased with other two-factor interactions.

Design-Expert 8.0.6 software was used to design and analyse the data. The design had 16 run combinations. The defining relationship for the fractional factorial design was not given but upon examination of the run combinations, it is evident that the default defining relationship in Design-Expert was used.

The response variable was the amount of ferulic acid (FA) produced, in mg/kg. The experimental procedure and analytical methods used in the laboratory to obtain the data were explained in the paper. The fractional factorial design and the responses obtained were shown in Table 2 of the paper and are reproduced here as Table 4.33.

Table 4.33: The design of the 2^4 fractional factorial experiments. Table 2 of Samad et al (2017).

	Coded values of variables							
Std	A	B	C	D	E	F	G	FA (mg/kg)
1	-1	-1	-1	-1	-1	-1	-1	87.2027
2	1	-1	-1	-1	1	-1	1	40.2194
3	-1	1	-1	-1	1	1	-1	237.364
4	1	1	-1	-1	-1	1	1	423.303
5	-1	-1	1	-1	1	1	1	56.2657
6	1	-1	1	-1	-1	1	-1	114.081
7	-1	1	1	-1	-1	-1	1	488.409
8	1	1	1	-1	1	-1	-1	263.808
9	-1	-1	-1	1	-1	1	1	133.519
10	1	-1	-1	1	1	1	-1	153.829
11	-1	1	-1	1	1	-1	1	289.284
12	1	1	-1	1	-1	-1	-1	218.093
13	-1	-1	1	1	1	-1	-1	162.206
14	1	-1	1	1	-1	-1	1	97.1652
15	-1	1	1	1	-1	1	-1	290.215
16	1	1	1	1	1	1	1	350.176

According to the authors, a first order polynomial model was fitted to the experimental data. That is, only a main effects model was fitted. However, as Table 3 of the paper contains the test of significance of the regression coefficients, it seems that the authors to fitted a two-factor interaction model. Table 3 of the paper is reproduced here as Table 4.34.

In Table 4.34, four two-factor interaction effects were shown to be statistically significant at the 5% level. These were AC, AE, AF and BD. However, since the experimental design was a resolution IV design, these two-factor interactions were aliased with other two-factor interactions. These aliased terms are:

AC=BE=DG; AE=BC=DF; AF=BG=DE; and BD=CF=EG

Table 4.34: Test of significance for regression coefficients. Table 3 of Samad et al (2017).

Source	Coefficient estimate	Sum of Squares	F-value	p-value
Model	212.82	259171.29	171.31	<0.0001
A - temperature	-5.24	438.81	3.19	*0.1486
B - pH	107.26	184075.84	1338.41	<0.0001
C - Agitation	14.97	3585.38	26.07	0.007
D - Water-to-substrate ratio	-1.01	16.33	0.12	*0.7477
E- Volume of inoculum	-18.68	5581.41	40.58	0.0031
F - fermentation time	7.02	789.13	5.74	*0.0747
G - Type of co-culture	21.97	7723.90	56.16	0.0017
AC	-16.25	4223.05	30.71	0.0052
AE	13.10	2746.19	19.97	0.0111
AF	45.74	33474.66	243.39	<0.0001
BD	-32.13	16516.58	120.09	0.0004
Residual		550.13		
Cor Total		259721.42		

R^2=0.9979, * Values of p-values greater than 0.05 indicating the model terms are not significant.

The four two-factor interaction terms were probably picked because they were listed first in alphabetical order by the software in the Pareto chart to select the statistically significant terms. The authors did not explain or consider the possibility that the other aliased terms might be the correct term to use.

The authors suggested the following equations, in terms of actual factors, for the two co-cultures, A and B (Equations 2 and 3) in the paper, respectively.

Co-culture A:

$$FA_A = 120.64 - 5.24\ A + 77.73\ B + 1.22\ C + 29.87\ D - 16.09\ E - 8.69\ F - 0.03\ AC + 0.37\ AE + 0.27\ AF - 4.02\ BD$$

Co-culture B:

$$FA_B = 164.58 - 15.24\ A + 77.73\ B + 1.22\ C + 29.87\ D - 16.09\ E - 8.69\ F - 0.03\ AE + 0.27\ AF - 4.02\ BD$$

The authors stated that since the R^2 was a high value of 0.9979 from the ANOVA (not shown in the paper), the model fitted the experimental and predicted values well. Furthermore, the study showed that the fractional factorial design had the ability to examine a large number of factors in a process with a minimal number of experimental runs. There was no indication that the authors planned further experimentation to de-alias the two-factor interaction effects, in order to be more conclusive as to which two-factor interactions actually contributed significantly to the production of ferulic acid.

5. 3-LEVEL FACTORIAL DESIGNS

Six case studies are presented in this Chapter. The case studies in this Chapter use 3-level or 3^k factorial designs. The number of factors ranges from two to four.

Case Study #5.1

Ahmad, S., and Saeid A. Alghamdi (2014): A statistical approach to optimizing concrete mixture design. The Scientific World Journal, Vol. 2014, Article ID 561539, pp 1-7.

This study used a 3-level factorial design with three factors to investigate the proportioning of concrete mixtures that affects the compressive strength of concrete. The factors and levels investigated were shown in Table 1 of the paper and are reproduced here as Table 5.1.

Table 5.1: Factors and levels used in the test program. Table 1 of Ahmad and Alghamdi, 2014)

		Level		
Factor	Units	1	2	3
Cementitious materials content (Q_C)	kg/m^3	350	375	400
Water/cementitious materials ratio ($R_{w/cm}$)	by mass	0.38	0.43	0.48
Fine/total aggregate ratio ($R_{FA/TA}$)	by mass	0.35	0.4	0.45

Each factor was at three levels giving 27 (3^3) mixture combinations. Three replicates were tested for each combination and the average compressive strength (f'_c) in MPa at 28 days of the three replicates was then used as the response. The 27-run combinations, the corresponding average 28-day compressive strength, and the standard deviation of the three replicates are shown in Table 5.2 which is a consolidation of Tables 2 and 4 reported in the paper.

Statistical analyses were carried out using the Minitab 13. The ANOVA results as reported in Table 5 of the paper are based on treating each factor as a categorical variable. This is summarized in Table 5.3. Goodness-of-fit statistics were not given in the paper and there was no mention of whether the assumptions of ANOVA were fulfilled.

The authors then fitted a nonlinear regression model to the data and obtained the following equation which is Equation (1) in the paper:

$$f_c' = -61.24 - 0.056\,Q_C - 19.87\,Exp\left(2.083R_{w/cm}\right) + 183.45\,R_{FA/TA}^{0.119} \qquad (R^2 = 0.80)$$

where all terms are previously defined in Table 5.1. No other data were used for validation of the proposed prediction equation.

Table 5.2: Trial mixtures and compressive strength test results (Tables 2 and 4 of Ahmad and Alghamadi, 2014)

Mix number	$R_{w/cm}$	Q_C	$R_{FA/TA}$	f'_c (MPA)	s.d. (MPA)
1	0.38	350	0.35	39.7	1.9
2	0.38	350	0.40	38.8	1.0
3	0.38	350	0.45	39.1	0.8
4	0.38	375	0.35	34.1	1.2
5	0.38	375	0.40	38.2	1.9
6	0.38	375	0.45	40.6	2.0
7	0.38	400	0.35	34.2	1.1
8	0.38	400	0.40	39.3	1.1
9	0.38	400	0.45	39.8	1.6
10	0.43	350	0.35	27.9	1.1
11	0.43	350	0.40	37.4	1.8
12	0.43	350	0.45	38.5	1.1
13	0.43	375	0.35	31.9	0.8
14	0.43	375	0.40	37.1	1.3
15	0.43	375	0.45	33.9	0.2
16	0.43	400	0.35	26.5	1.4
17	0.43	400	0.40	30.7	1.7
18	0.43	400	0.45	36.5	1.6
19	0.48	350	0.35	30.0	1.5
20	0.48	350	0.40	32.1	1.3
21	0.48	350	0.45	30.5	0.8
22	0.48	375	0.35	20.7	1.8
23	0.48	375	0.40	27.5	0.8
24	0.48	375	0.45	29.9	0.3
25	0.48	400	0.35	25.4	1.1
26	0.48	400	0.40	31.0	0.2
27	0.48	400	0.45	25.3	0.2

Note: f’c = average 28-day compressive strength in MPa, s.d. = standard deviation of 3 replicates of each mixture in MPa.

Table 5.3: ANOVA results assuming all factors are categorical (after Table 5 of Ahmad Alghamadi, 2014).

Source	df	SS	MS	F-ratio	p-value	Significance
Q_C	2	39.672	19.836	1.986	0.199	No
$R_{w/cm}$	2	464.501	232.251	23.257	0.000	Yes
$R_{FA/TA}$	2	135.281	67.641	6.773	0.019	Yes
Q_C*$R_{w/cm}$	4	23.686	5.922	0.593	0.678	No
Q_C*$R_{FA/TA}$	4	4.993	1.248	0.125	0.969	No
$R_{w/cm}$*$R_{FA/TA}$	4	22.437	5.609	0.562	0.697	No
Error	8	79.890	9.986			
Total	26	770.460				

Note: Table 5.3 has been corrected for typographical errors. Significance level was at the 5% level.

The regression model for compressive strength was subsequently used for optimization of the concrete mixture proportions using Microsoft Excel Solver to optimize the levels of $R_{w/cm}$ and $R_{FA/TA}$ to achieve the maximum possible compressive strength at various levels of Q_C, and for optimizing the levels of $R_{w/cm}$ and $R_{FA/TA}$ to achieve different target compressive strengths at different levels of Q_C. The results are given in the paper.

The authors reported that their proposed statistical approach obtained optimum values of water/cementitious materials and fine/total aggregate ratios with higher compressive strength at a lower cementitious materials content resulting in significant cost savings in concrete production.

The reader is suggested to fit a quadratic model to the data using linear regression and compare the results with those obtained by the authors.

Case Study #5.2:

Alsobaai, Ahmed Mubarak (2013): Thermal cracking of petroleum residue oil using three level factorial design. Journal of King Saud University – Engineering Sciences, 25, pp. 21-28.

This study investigated the thermal cracking of petroleum residue in a high-pressure reactor under various conditions. Three factors were investigated, namely, temperature, reaction time, and pressure. The experimental design was a three-factor three-level factorial design with five centre points. The factors and levels used in the experiments were shown in Table 3 of the paper and are reproduced here as Table 5.4.

A total of $3^3 + 5 = 32$ run combinations were used. Five responses in weight percentage (wt%) were measured for each run combination. These were the conversion rate, X, the yield of total distillate fuels, Y, and the yield of the each of the distillate fractions, Y_1 (gasoline), Y_2 (kerosene) and Y_3 (diesel). How each yield is obtained was explained in the paper.

Table 5.4: Factors and levels used in the experiment. Table 3 of Alsobaai (2013).

Factor	Code	Unit	Low level (-1)	Medium level (0)	High level (+1)
Temperature	A	°C	400	440	480
Time	B	min	40	70	100
Pressure	C	kPa	120	150	180

Table 5.5: The experimental results, based on the 3^3 full factorial design. Table 4 of Alsobaai, (2013).

No.	Temp (°C)	Time (min)	Pressure (kPa)	X (wt%)	Y (wt%)	Y_1(wt%)	Y_2 (wt%)	Y_3 (wt%)
1	440	40	120	71	32	7.9	9.3	14.8
2	400	70	180	68	30	6.9	7.5	15.6
3	400	40	120	52	27	6.2	7.1	13.7
4	440	70	180	81	40	18.0	13.6	8.4
5	480	100	150	88	44	14.4	16.7	12.9
6	480	100	180	93	48	27.0	12.0	9.0
7	480	100	120	86	43	21.3	13.2	8.5
8	480	40	150	77	36	13.8	13.4	8.8
9*	440	70	150	82	42	17.3	16.8	7.9
10	400	40	180	56	28	5.4	7.3	15.3
11	440	40	150	70	31	8.0	8.9	14.1
12	480	70	150	90	46	22.3	13.3	10.4
13	400	70	120	64	29	8.2	8.8	12.0
14	480	70	180	89	44	23.1	12.9	8.0
15	400	100	150	72	31	8.8	8.3	13.9
16*	440	70	150	80	39	17.0	12.9	10.1
17	400	100	120	73	33	7.9	9.2	15.9
18	440	100	120	84	41	17.2	16.6	7.2
19	400	100	180	74	33	10.2	8.1	14.7
20	440	40	180	69	30	8.4	8.8	12.8
21	440	100	180	75	33	9.5	8.9	14.6
22	480	40	180	79	39	14.2	12.9	11.9
23	400	40	150	55	28	7.7	7.6	12.7
24*	440	70	150	76	35	10.0	11.2	13.8
25	440	70	150	78	38	9.9	11.0	14.1
26	480	40	120	67	34	12.1	12.4	9.5
27	480	70	120	80	40	13.8	12.7	13.5
28*	440	70	150	78	37	9.7	11.1	145.2
29	440	70	120	76	36	9.4	11.3	15.3
30	400	70	150	67	30	8.5	9.0	12.6
31	440	100	150	77	36	9.8	11.2	15.0
32*	440	70	150	75	34	9.5	11.0	13.5

*Centre points

Statistical analyses were carried out using Design-Expert 6.0.3. A general second-order response surface model was fitted to each of the responses.

The ANOVA results are shown in Tables 5 to 9 of the paper, for X, Y, Y_1, Y_2, and Y_3, respectively. Except for the conversion, X, no goodness-of-fit statistic was given for the other responses in the paper. There is also no mention of whether the assumptions of ANOVA were fulfilled for each of the responses analyzed.

The ANOVA results for the conversion, X, are shown below in Table 5.6 which is adapted from Table 5 of the paper.

Table 5.6: ANOVA results for conversion, X, (wt%).

Source of variation	Sum Squares	DF	Mean Square	F-value	Prob>F	Comment
Model	2729.05	9	303.23	27.25	<0.0001	Significant
A	1568.00	1	1568.00	140.90	<0.0001	
B	882.00	1	882.00	79.26	<0.0001	
C	53.39	1	53.39	4.80	0.0394	
A^2	20.26	1	20.26	1.82	0.1910	
B^2	97.03	1	97.03	8.72	0.0074	
C^2	1.91	1	1.91	0.17	0.6830	
AB	12.00	1	12.00	1.08	0.3103	
AC	30.08	1	30.08	2.70	0.1144	
BC	18.75	1	18.75	1.68	0.2077	
Residual	244.82	22	11.13			
Lack of fit	211.99	17	12.47	1.90	0.2469	Not significant
Pure error	32.83	5	6.57			
Corr. Total	2973.88	31				

The author reported that the full quadratic model has a R^2 value of 0.9177, a PRESS of 578.91, and predicted R^2 of 0.8053 which is in reasonable agreement with the adjusted R^2 of 0.8840. Goodness-of-fit statistics were not given for the reduced model with only statistically significant terms.

The other ANOVA results are given in the paper and will not be reproduced here. The proposed prediction equations in actual factors for X, Y, Y_1, Y_2, and Y_3 are given In Equations (5) – (9) of the paper. They are reproduced here showing the actual factors.

X = Conversion = -238.979 + 0.0193 Temp + 1.3812 Time – 0.2538 Pressure – 0.00409 Time^2

Y = Total distilled fuels = -38.4896 + 0.1458 Temp + 0.1056 Time + 0.0185 Pressure

Y_1 = Gasoline yield = -54.7414 + 0.1281 Temp + 0.0785 Time + 0.0346 Pressure

Y_2 = Kerosene yield = -17.1340 + 0.064722 Temp + 0.0306 Time – 0.0159 Pressure

Y_3 = Diesel yield = 33.3251 – 0.0471 Temp – 0.003519 Time – 0.0001852 Pressure

It should be noted that for X, the author did not refit the reduced model. The insignificant terms were simply removed from the full quadratic model. For the other responses, the author has included all three main effects even though some of them are not statistically significant.

Case Study #5.3

Amini, S., M. J. Nategh, and H. Soleimanimehr (2009): Application of design of experiments for modelling surface roughness in ultrasonic vibration turning. Proceedings Institution of Mechanical Engineers, Vol. 223, Part B: J. Engineering Manufacture, pp. 641-652.

This study investigated the influence of four parameters on surface roughness of workpieces machined using an advanced machining technique, known as ultrasonic vibration-assisted turning (UAT). UAT has been shown to have several advantages over the conventional turning (CT) approach. A model for predicting the surface roughness after using the UAT is desired. The parameters investigated were the vibration amplitude, depth of cut, feed rate, and cutting speed. A three-level full factorial design with four factors was used. The levels of the factors (parameters) used in the experiment were shown in Table 1 of the paper and are reproduced here as Table 5.7.

Table 5.7: Different levels of UAT parameters used in the experiments. Table 1 of Amini et al (2009)

Factor	Description	Level 1	Level 2	Level 3
a (μ m)	Vibration amplitude	6	12	18
d (mm)	Depth of cut	0.3	0.6	1.0
f_r (mm/rev)	Feed rate	0.11	0.20	0.40
v_c (m/min)	Cutting speed	12.3	34.0	68.1

The details on how the experiments were carried out were given in the paper. A smaller experiment involving only three factors (without vibration amplitude) was also carried out for conventional turning but will not be included here.

The experiment was a 3-level full factorial experiment. Hence the number of runs used was 3^4 = 81. The run combinations and surface roughness, R_a (μm) results were shown in Table 2 of the paper and are reproduced here as Table 5.8. Note that the levels for the depth of cut, feed rate, and cutting speed are not equally spaced. The results of the surface roughness obtained by CT are not shown in Table 5.8. Minitab (version unknown) was used for the ANOVA and regression modelling. The authors considered three different prediction models. The first model fitted was a linear model with only the main effects. A quadratic model was used next, followed by a cubic model. A summary of the goodness-of-fit statistics for the three models are shown in Table 5.9. The ANOVA results for each of the models considered were also given in the paper together with the prediction equations.

Table 5.8: Run combinations and UAT surface roughness results. Table 2 of Amini et al (2009).

No.	a (μm)	d (mm)	f_r(mm/rev)	v_c (m/min)	R_a (μm)
1	6	0.3	0.11	12.3	0.404
2	6	0.3	0.11	34.0	0.534
3	6	0.3	0.11	68.1	1.546
4	6	0.3	0.20	12.3	1.592
5	6	0.3	0.20	34.0	2.695
6	6	0.3	0.20	68.1	3.090
7	6	0.3	0.40	12.3	6.237
8	6	0.3	0.40	34.0	7.396
9	6	0.3	0.40	68.1	7.401
10	6	0.6	0.11	12.3	2.215
11	6	0.6	0.11	34.0	2.837
12	6	0.6	0.11	68.1	3.142
13	6	0.6	0.20	12.3	2.872
14	6	0.6	0.20	34.0	4.614
15	6	0.6	0.20	68.1	3.047
16	6	0.6	0.40	12.3	6.811
17	6	0.6	0.40	34.0	7.499
18	6	0.6	0.40	68.1	5.700
19	6	1.0	0.11	12.3	2.293
20	6	1.0	0.11	34.0	2.187
21	6	1.0	0.11	68.1	3.292
22	6	1.0	0.20	12.3	2.365
23	6	1.0	0.20	34.0	2.982
24	6	1.0	0.20	68.1	3.543
25	6	1.0	0.40	12.3	5.763
26	6	1.0	0.40	34.0	5.631
27	6	1.0	0.40	68.1	6.479
28	12	0.3	0.11	12.3	0.683
29	12	0.3	0.11	34.0	0.868
30	12	0.3	0.11	68.1	0.971
31	12	0.3	0.20	12.3	1.914
32	12	0.3	0.20	34.0	1.713
33	12	0.3	0.20	68.1	1.715
34	12	0.3	0.40	12.3	6.429
35	12	0.3	0.40	34.0	5.927
36	12	0.3	0.40	68.1	6.057
37	12	0.6	0.11	12.3	0.596
38	12	0.6	0.11	34.0	0.752
39	12	0.6	0.11	68.1	0.790

No.	a (μm)	d (mm)	f_r(mm/rev)	v_c (m/min)	R_a (μm)
40	12	0.6	0.20	12.3	1.595
41	12	0.6	0.20	34.0	1.644
42	12	0.6	0.20	68.1	1.889
43	12	0.6	0.40	12.3	6.808
44	12	0.6	0.40	34.0	5.636
45	12	0.6	0.40	68.1	5.980
46	12	1.0	0.11	12.3	0.788
47	12	1.0	0.11	34.0	0.763
48	12	1.0	0.11	68.1	0.840
49	12	1.0	0.20	12.3	1.750
50	12	1.0	0.20	34.0	1.906
51	12	1.0	0.20	68.1	1.757
52	12	1.0	0.40	12.3	6.700
53	12	1.0	0.40	34.0	6.955
54	12	1.0	0.40	68.1	6.616
55	18	0.3	0.11	12.3	1.330
56	18	0.3	0.11	34.0	1.855
57	18	0.3	0.11	68.1	0.729
58	18	0.3	0.20	12.3	1.645
59	18	0.3	0.20	34.0	2.948
60	18	0.3	0.20	68.1	2.180
61	18	0.3	0.40	12.3	4.910
62	18	0.3	0.40	34.0	6.404
63	18	0.3	0.40	68.1	7.155
64	18	0.6	0.11	12.3	2.885
65	18	0.6	0.11	34.0	0.743
66	18	0.6	0.11	68.1	0.774
67	18	0.6	0.20	12.3	2.812
68	18	0.6	0.20	34.0	2.295
69	18	0.6	0.20	68.1	1.682
70	18	0.6	0.40	12.3	6.311
71	18	0.6	0.40	34.0	6.664
72	18	0.6	0.40	68.1	6.148
73	18	1.0	0.11	12.3	1.530
74	18	1.0	0.11	34.0	1.501
75	18	1.0	0.11	68.1	1.371
76	18	1.0	0.20	12.3	2.580
77	18	1.0	0.20	34.0	2.134
78	18	1.0	0.20	68.1	2.252
79	18	1.0	0.40	12.3	7.595
80	18	1.0	0.40	34.0	7.615
81	18	1.0	0.40	68.1	8.019

Table 5.9: Summary of R^2 and R^2(adj) for different regression models. Table 7 of Amini et al. (2009).

Model	R^2	R^2 (adj)
Linear polynomial	0.886	0.880
Quadratic polynomial	0.933	0.919
Cubic polynomial	0.955	0.942

The authors chose the cubic polynomial (actually a reduced cubic polynomial) as the prediction model for R_a. The ANOVA results for this model were shown in Table 6 of the paper and are reproduced here as Table 5.10.

Table 5.10: ANOVA results for the cubic regression model. Table 6 of Amini et al (2009)

Source	DF	SS	MS	F	p-value
Regression	18	432.206	24.011	73.78	0.000
Residual error	62	20.177	0.325		
Total	80	452.383			

The cubic regression model given by the authors is reproduced below. This is Equation (4) in the paper:

$$\begin{aligned} R_a(\mu m) = {} & -1.00 - 0.176\, a + 6.10\, d + 13.8\, f_r + 0.0582\, v_c + 0.0196\, a^2 - 1.16\, d^2 \\ & + 37.1\, f_r^2 - 0.000172\, v_c^2 - 0.348\, ad - 1.34\, af_r \ - 0.00392\, av_c - 19.5\, df_r \\ & + 0.0166\, dv_c - 0.123\, f_r v_c + 1.74\, adf_r - 0.00079 adv_c + 0.0133\, af_r v_c \\ & - 0.0492\, df_r v_c \end{aligned}$$

Note that several terms in this model were not statistically significant and other significant terms could have been added for a better fit. The above cubic regression model was verified using four additional experimental runs not used in deriving the model. The verification results were shown in Table 8 of the paper and are reproduced here as Table 5.11.

Table 5.11: Verification test results of the cubic regression model. Table 8 of Amini et al (2009).

UAT parameters				Cubic regression model	Experimental results
a (μm)	d (mm)	f_r(mm/rev)	v_c (m/min)	R_a (μm)	R_a (μm)
6	0.5	0.14	3.78	1.38	1.37
12	0.6	0.14	8.83	1.10	1.03
12	0.7	0.14	10.80	1.19	1.37
16	0.8	0.28	17.66	3.69	3.74

The subrange results given in Table 8 of Amini et al (2009) are not shown in Table 5.11.

Based on the four verification tests, the authors concluded that the proposed cubic regression model is acceptable.

Case Study #5.4

Berrios, M., M.C. Gutierrez, M.A. Martin, and A. Martin (2009): Application of the factorial design of experiments to biodiesel production from lard. Fuel Processing Technology, 90, pp. 1447-1451.

This study considered the synthesis of biodiesel from lard using potassium hydroxide as a catalyst. A three-level full factorial design was used to investigate the effect of two factors - agitation speed and catalyst concentration, on the production of long-chain fatty acid methyl esters or FAME for short. The factors and levels used in the experiment are shown in Table 5.12.

Table 5.12: Factors and levels used in the biodiesel experiment.

Factor	Description	Units	Low level	Mid-level	High level
x_1	Agitation speed	rpm	400	600	800
x_2	Catalyst concentration	wt %	0.6	0.9	1.2

The response measured was the FAME concentration (C, % m/m) at 20 minutes. Two replications were used giving 2 x 3^2 runs or 18 experimental runs. The run combinations and experimental results were shown in Table 2 of the paper and are reproduced here as Table 5.13.

Table 5.13: Experimental matrix and FAME concentration results after 20 min. Table 2 of Berrios et al (2009).

No.	Run order	Agitation speed (rpm)	Catalyst concentration (wt%)	C (% m/m)
1	10	400	0.6	84.30
2	11	400	0.6	84.50
3	6	400	0.9	88.50
4	17	400	0.9	88.70
5	3	400	1.2	88.90
6	4	400	1.2	88.20
7	13	600	0.6	86.50
8	18	600	0.6	86.70
9	1	600	0.9	89.20
10	7	600	0.9	89.30
11	5	600	1.2	90.20
12	12	600	1.2	90.30
13	14	800	0.6	86.90
14	16	800	0.6	86.90
15	8	800	0.9	89.30
16	9	800	0.9	89.40
17	2	800	1.2	90.70
18	15	800	1.2	90.90

Note: The standard deviations are not shown in this table.

The details of how the experiments were conducted are given in the paper. A general linear model (GLM) was fitted to the data and the resulting ANOVA results are shown in Table 3 in the paper. The authors did not mention the statistical package used for the statistical analyses. On examining the reported ANOVA table, some of the sum of squares values were clearly in error. The authors fitted only a two-factor interaction model to the data. The corrected ANOVA table is shown in Table 5.14 after reanalysis using Design-Expert 12.

Table 5.14: ANOVA results for a two-factor interaction model.

Source	Sum of squares	df	Mean square	F-value	p-value
Model	58.228	3	19.409	36.74	<0.0001
Agitation speed (x_1)	8.33	1	8.33	15.77	0.0014
Catalyst concentration (x_2)	49.61	1	49.61	93.91	<0.0001
$x_1 * x_2$	0.2812	1	0.2812	0.5323	0.4778
Residual	7.40	14	0.5283		
Lack of fit	7.26	5	1.45	93.3	<0.0001
Pure error	0.14	9	0.0156		
Corrected total	65.62	17			

The authors then proposed the two-factor interaction model as the final prediction model for FAME. This is Equation (1) in Berrios et al (2009), shown below.

$$y = 78.068 + 0.007\, x_1 + 8.653\, x_2 - 0.003\, x_1 x_2$$

where, y is the FAME concentration. The coefficient of determination (R^2) was given as 0.887. It is not clear why the authors selected the two-factor interaction model. Clearly the two-factor interaction term is not statistically significant at the 5% level. Furthermore, there is significant lack of fit in the chosen model. A better model was not investigated by the authors.

To validate the model, two additional experiments were carried out. The first case used a value of 500 rpm for the agitation speed and 1 wt% for catalyst concentration obtaining a FAME concentration of 88.8%. The predicted value using the model was 88.7%. The second case used a value of 700 rpm for the agitation speed and 1 wt% for the catalyst concentration obtaining a FAME concentration of 88.8%. The predicted value was 89.4%. Based on these validation results the authors considered the prediction model to be a sufficiently accurate representation of the methyl ester production from lard for the range of factors studied.

Case Study #5.5

Comoglu, B. A., Cansun Filik Iscen, and Semra IIhan (2015): The anaerobic treatment of pharmaceutical industry wastewater in an anaerobic batch and upflow packed-bed reactor. Desalination and Water Treatment, 2015, pp 1-12.

This study concerned the treatment of pharmaceutical industry wastewater before discharge into receiving water bodies. Pharmaceutical industry wastewater is known to contain various complex organic chemicals that are toxic to the environment. One of the experiments investigated the effect of three factors, namely, basal medium, wastewater concentration, and various types of co-substrate, on COD removal. A three-level full factorial design with three factors was used. The factors and levels used in the experiment were shown in Table 1 of the paper and are reproduced here as Table 5.15.

Table 5.15: Variables and levels used in the experimental design. Table 1 of Comoglu et al (2015)

	Levels		
Variables	-1	0	1
X_1 - Basal medium (%)	5	10	15
X_2 - Wastewater (%)	25	50	75
X_3 - Cosubstrate type	Propionic acid	Glucose	Acid mix

Since this was a 3^3 full factorial experiment, 27 runs were required. The basal medium and wastewater concentrations are numeric factors while the co-substrate type is a categorical factor. The co-substrate is one of three types. The first type is a mixture of acetic-propionic-butyric acids or (ABP), each at various concentrations. The second type is glucose, and the third type is propionic acid. The other details of the experiment were given in the paper.

The response variable was the percentage of COD removed from the wastewater stream. The experiments were carried out in a batch reactor. The 27 run combinations of the experiment together with the COD removal percentage is shown in Table 2.16 which is Table 2 in the paper. All experiments were performed in duplicate but only 27 responses were given in the paper.

Statistical analyses were carried out using the SPSS statistical package. The ANOVA results and multiple comparison tests results were given in Tables 3 and 4 of the paper and are reproduced here as Table 5.17.

From the ANOVA results, the authors have treated all factors as categorical and the corrected sum of squares degrees of freedom is shown as 53, indicating that there were 54 data points used in the ANOVA. Hence the results given in Table 5.17 (Table 2 in the paper) were incomplete.

Table 5.16: Full factorial (3^3) experimental design and results of COD removal. Table 2 of Comoglu et al (2015)

No.	Basal medium (%)	Wastewater (%)	Co-substrate	COD removal (%)
1	15	75	ABP	29.44
2	15	75	Glucose	14.07
3	15	75	Propionic acid	26.52
4	15	50	ABP	30.53
5	15	50	Glucose	16.00
6	15	50	Propionic acid	80.69
7	15	25	ABP	93.49
8	15	25	Glucose	37.38
9	15	25	Propionic acid	90.20
10	10	75	ABP	28.11
11	10	75	Glucose	15.15
12	10	75	Propionic acid	37.38
13	10	50	ABP	83.46
14	10	50	Glucose	20.76
15	10	50	Propionic acid	30.20
16	10	25	ABP	89.94
17	10	25	Glucose	13.02
18	10	25	Propionic acid	72.00
19	5	75	ABP	27.08
20	5	75	Glucose	15.78
21	5	75	Propionic acid	26.20
22	5	50	ABP	80.29
23	5	50	Glucose	32.55
24	5	50	Propionic acid	44.62
25	5	25	ABP	94.38
26	5	25	Glucose	17.72
27	5	25	Propionic acid	80.67

Although it was mentioned in the paper that a regression model of the form

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 + \beta_{123} x_1 x_2 x_3 + \varepsilon$$

can be fitted to the data, no prediction equation was given in the paper.

Table 5.17: Variance analysis for COD removal. Table 3 of Comoglu et al (2015)

Source	Type III SS	df	MS	F	p-value
Corrected model	45969.788	26	1768.069	13.629	0.000*
Intercept	111623.395	1	111623.395	860.416	0.000*
Basal	122.962	2	61.481	0.474	0.628
Wastewater	15165.682	2	7582.841	58.450	0.000*
Co-substrate	17659.762	2	8829.881	68.063	0.000*
Basal-wastewater	985.515	4	246.379	1.899	0.140
Basal-cosubstrate	2293.123	4	573.281	4.419	0.007*
Wastewater-cosubstrate	5432.023	4	1358.006	10.468	0.000*
Basal-wastewater-cosubstrate	4310.76	8	538.845	4.154	0.002*
Error	3502.76	27	129.732		
Total	161095.943	54			
Corrected total	49472.548	53			

Based on the ANOVA results, with the exception of the basal medium, the authors reported that the other factors and two-way interactions between basal and co-substrate, wastewater and co-substrate, and the three-way interaction among the three factors were statistically significant at the 5% level.

In view of the missing duplicate data, it is not possible to replicate the authors' results. There was also no mention of whether the assumptions of ANOVA were fulfilled.

Case Study #5.6

Martin-Lara, M. A., I. L. Rodriguez, G. Blazquez, and M. Calero (2011): Factorial experimental design for optimizing the removal conditions of lead ions from aqueous solutions by three wastes of olive-oil production. Desalination, 278, pp. 132-140.

Three wastes, namely olive stone (OS), two-phase olive solid (OMS), and olive tree pruning waste (OTP), from olive-oil production were considered as low-cost adsorbents for lead removal from aqueous solutions. Two sets of three-level factorial design each with two factors were used for each biosorbent. The factors studied were biosorbent dosage and pH for the first set of experiments, and initial lead concentration and temperature for the second of experiments. The factors and levels used were shown in Table 1 of the paper and reproduced here as Table 5.18.

The responses measured were lead removal percentage and biosorption capacity after 120 minutes of contact time for each of OS, OMS, and OTP. The experimental details were given in the paper and statistical analyses were carried out using Statgraphics Plus 5.1 software.

The run combinations and the results for lead uptake by OS, OMS, and OTP using the factors biosorbent dosage (g/L) and pH in the first set of experiments were shown in Table 2 of the paper and reproduced here as Table 5.19. For the second set of experiments, the run combinations and results were shown in Table 4 of the paper and are reproduced here as Table 5.20. The same responses were used but with the initial lead concentration and temperature as factors. The experiments were run in random order and in duplicate. Hence there were 2 x 3^2 = 18 runs per response.

Table 5.18: Factors and levels used in the experimental design. Table 1 of Martin-Lara et al (2011)

Factor	Symbol	Low level (-1)	Center level (0)	High level (+1)
Biosorbent dosage, g/L	X_1	2	10	22
pH	X_2	4	5	6
$[Pb]_{initial}$, mg/L	X_3	10	40	70
Temperature, C	X_4	25	40	60

Note: only X_1 and X_2 were used in the first set of experiments, and only X_3 and X_4 were used in the second set of experiments. Some of the factor levels are not equally spaced.

Table 5.19: First experimental factorial design for lead uptake by OS, OMS, and OTP. Table 2 of Martin-Lara et al (2011).

Run	Factors		Biosorbent					
			Olive Stone (OS)		Two-phase olive mill solid (OMS)		Olive tree pruning (OTP)	
	Biosorbent dosage	pH	Removal (%)	Biosorption capacity (mg/g)	Removal (%)	Biosorption capacity (mg/g)	Removal (%)	Biosorption capacity (mg/g)
1	2	4	29.00	1.450	57.30	2.865	90.00	4.500
2	10	4	69.42	0.694	88.90	0.889	97.00	0.970
3	22	4	73.30	0.333	87.00	0.395	93.50	0.425
4	2	5	49.80	2.490	52.50	2.625	92.00	4.600
5	10	5	77.30	0.773	83.40	0.834	98.00	0.980
6	22	5	76.00	0.345	88.40	0.402	94.60	0.430
7	2	6	69.90	3.495	61.90	3.095	89.80	4.490
8	10	6	79.80	0.798	88.04	0.880	96.60	0.966
9	22	6	79.10	0.360	85.10	0.387	95.00	0.432
10	2	4	28.35	1.418	56.70	2.835	89.60	4.480
11	10	4	68.20	0.682	88.10	0.881	96.40	0.964
12	22	4	72.90	0.331	86.60	0.394	92.10	0.419
13	2	5	48.90	2.445	51.50	2.575	91.40	4.570
14	10	5	77.10	0.771	82.60	0.826	97.80	0.978
15	22	5	75.00	0.341	88.00	0.400	94.00	0.427
16	2	6	69.10	3.455	62.10	3.105	89.40	4.470
17	10	6	79.40	0.794	87.20	0.872	96.40	0.964
18	22	6	79.20	0.360	84.90	0.386	94.40	0.430

Note: The factors in Table 5.19 and 5.20 are listed here as actual factors instead of coded factors in Tables 2 and 3 of the paper.

Table 5.20: Second experimental factorial design for lead uptake by OS, OMS, and OTP. Table 4 of Martin-Lara et al (2011).

Run	Factors		Biosorbent					
			Olive Stone (OS)		Two-phase olive mill solid (OMS)		Olive tree pruning (OTP)	
	$[Pb]_{initial}$	Temp	Removal (%)	Biosorption capacity (mg/g)	Removal (%)	Biosorption capacity (mg/g)	Removal (%)	Biosorption capacity (mg/g)
1	10	25	77.30	0.773	83.40	0.834	98.00	0.980
2	40	25	74.50	2.980	59.02	2.362	90.90	3.636
3	70	25	49.07	3.435	44.00	3.080	86.30	6.041
4	10	40	73.00	0.730	60.00	0.600	80.60	0.806
5	40	40	70.53	2.821	55.25	2.210	88.48	3.539
6	70	40	35.17	2.462	38.00	2.660	75.83	5.308
7	10	60	76.50	0.765	52.50	0.525	77.00	0.770
8	40	60	63.20	2.528	49.48	1.979	87.50	3.500
9	70	60	44.46	3.112	37.14	2.600	78.64	5.500
10	10	25	77.10	0.771	82.60	0.826	97.80	0.978
11	40	25	74.10	2.964	58.80	2.351	90.88	3.635
12	70	25	48.61	3.403	43.89	3.072	86.14	6.030
13	10	40	72.20	0.722	58.60	0.586	80.90	0.809
14	40	40	70.25	2.810	54.95	2.198	88.20	3.528
15	70	40	34.21	2.395	37.87	2.651	75.70	5.299
16	10	60	76.10	0.761	51.40	0.514	75.60	0.756
17	40	60	63.00	2.520	49.08	1.963	85.90	3.436
18	70	60	44.09	3.086	36.91	2.584	78.41	5.489

Second-order response surface models with two independent variables (X_i and X_j) were fitted to each of the responses. The model is of the form:

$$Y = b_0 + b_iX_i + b_jX_j + b_{ii}X_i^2 + b_{jj}X_j^2 + b_{ij}X_iX_j$$

where, i and j are 1 and 2, respectively for the first set of experiments, and 3 and 4, respectively, for the second set of experiments. Note that in the paper, the models for the second set of experiments were erroneously stated in terms of 1 and 2 instead of 3 and 4.

The models' coefficients for each response were tabulated by the authors in Tables 3 and 5 for the first and second set of experiments, respectively. These are reproduced here with corrections as Tables 5.21 and 5.22. There was no indication if there was lack of fit or if the assumptions of regression were checked. The R^2 values reported were for the full models only. Some of the responses should have been log-transformed.

The authors concluded that the developed regression models for predicting lead removal and lead biosorption capacity fitted the experimental data very well.

Table 5.21: First experimental design: Response function coefficients for lead removal efficiency and lead biosorption capacity. X_1 = biosorbent dosage, and X_2 = pH. Table 3 of Martin-Lara et al (2011).

		Removal (%)		Biosorption capacity (mg/g)	
		Coefficient value	Std. Deviation	Coefficient value	Std. Deviation
	b_0	-76.1917	8.5174	-1.7152	0.2877
X_1	b_1	9.2517	0.8584	-0.0879	0.0582
X_2	b_2	27.6376	9.6156	1.0245	0.3292
X_1X_2	b_{12}	-0.8163	0.1371	-0.0466	0.0093
X^2_1	b_{11}	-0.1597	0.0205	0.0090	0.0014
X^2_2	b_{22}	-0.8775	0.1952	-0.0133	0.0023
R^2		0.961		0.957	
Standard error of est.		3.904		0.276	
Mean absolute error		2.676		0.169	

OMS		Removal (%)		Biosorption capacity (mg/g)	
		Coefficient value	Std. Deviation	Coefficient value	Std. Deviation
	b_0	118.2520	31.6512	6.3912	1.3049
X_1	b_1	6.6580	0.5599	-0.3441	0.0231
X_2	b_2	-32.0045	9.7966	-1.2772	0.5276
X_1X_2	b_{12}	-0.1596	0.0894	-0.0059	0.0036
X^2_1	b_{11}	-0.1824	0.0133	0.0105	0.0006
X^2_2	b_{22}	3.4200	1.2735	0.1383	0.0525
R^2		0.979		0.994	
Standard error of est.		2.547		0.109	
Mean absolute error		1.804		0.075	

OTP		Removal (%)		Biosorption capacity (mg/g)	
		Coefficient value	Std. Deviation	Coefficient value	Std. Deviation
	b_0	58.7363	7.0036	4.9001	0.3323
X_1	b_1	1.1972	0.1239	-0.6848	0.0058
X_2	b_2	12.2772	2.8315	0.3775	0.1343
X_1X_2	b_{12}	0.0579	0.0197	0.0005	0.0001
X^2_1	b_{11}	-0.0544	0.0029	0.0199	0.0001
X^2_2	b_{22}	-1.2667	0.2817	-0.0383	0.0133
R^2		0.974		0.999	
Standard error of est.		0.564		0.027	
Mean absolute error		0.377		0.018	

Table 5.22: Second experimental design: Response function coefficients for lead removal efficiency and lead biosorption capacity. X_3 = Initial Pb concentration, and X_4 = Temperature. Table 5 of Martin-Lara et al (2011).

OS		Removal (%)		Biosorption capacity (mg/g)	
		Coefficient value	Std. Deviation	Coefficient value	Std. Deviation
	b_0	104.7230	11.8844	1.3789	0.6524
X_3	b_3	0.4254	0.2037	0.1353	0.0111
X_4	b_4	-1.5689	0.5606	-0.0980	0.0308
X_3X_4	b_{34}	-0.0014	0.0025	-0.0001	0.00001
X^2_3	b_{33}	-0.0114	0.0021	-0.0012	0.00001
X^2_4	b_{44}	0.0172	0.0063	0.0012	0.00004
R^2		0.957		0.974	
Standard error of est.		3.975		0.210	
Mean absolute error		2.582		0.147	

OMS		Removal (%)		Biosorption capacity (mg/g)	
		Coefficient value	Std. Deviation	Coefficient value	Std. Deviation
	b_0	128.008	10.3377	0.8101	0.1668
X_3	b_3	-0.6847	0.1772	0.0801	0.0028
X_4	b_4	-2.1388	0.4877	-0.0369	0.0078
X_3X_4	b_{34}	0.0112	0.0022	-0.0001	0.00004
X^2_3	b_{33}	-0.0025	0.0018	-0.0005	0.00003
X^2_4	b_{44}	0.0146	0.0063	0.0003	0.00009
R^2		0.958		0.998	
Standard error of est.		3.451		0.055	
Mean absolute error		2.294		0.034	

OTP		Removal (%)		Biosorption capacity (mg/g)	
		Coefficient value	Std. Deviation	Coefficient value	Std. Deviation
	b_0	133.432	9.33940	1.0380	0.4051
X_3	b_3	0.1845	0.16010	0.1130	0.0069
X_4	b_4	-2.0751	0.44060	-0.0610	0.0191
X_3X_4	b_{34}	0.0066	0.00200	-0.0001	0.00009
X^2_3	b_{33}	-0.0067	0.00160	-0.0003	0.00007
X^2_4	b_{44}	0.0176	0.00500	-0.0007	0.0002
R^2		0.877		0.997	
Standard error of est.		3.121		0.136	
Mean absolute error		2.144		0.091	

6. RSM: BOX BEHNKEN DESIGNS

Eight case studies are presented in this Chapter. The case studies in this Chapter use RSM based on Box-Behnken designs (BBD). The number of factors ranges from three to six. The Box-Behnken design is a well-known classical response surface design with three levels (-1, 0, +1) and there are no extreme combinations of the factors. These designs are rotatable or near rotatable. The main drawback of BBD is that it is not amendable to sequential experimentation unlike central composite designs. These designs require more runs than central composite designs except when the number of factors are three or four.

Case Study #6.1

Aslan, N. and Y. Cebeci (2007): Application of Box-Behnken design and response surface methodology for modeling of Turkish coals. Fuel, 86, pp. 90-97.

This study applied response surface methodology to model the grinding process of some Turkish coals. Steel ball diameter, grinding time, and the Bond work index were three independent factors studied. The Box-Behnken response surface design with three factors was used to obtain the experimental data for statistical analysis and modelling. The importance of studying the grinding process and the materials and methods used were described in the paper.

The factors and levels used for the grinding experiment were shown in Table 4 of the paper and are reproduced here as Table 6.1. Note that in the original table, the levels of the ball diameter were listed as 2.5, 4.0 and 5.5, however, the text and later tables showed that the levels should be 25, 40 and 55, respectively.

Table 6.1: The levels of variables chosen for the Box-Behnken design. Corrected Table 4 of Aslan et al (2007).

Variable	Symbol	Coded variable level		
		Low (-1)	Center (0)	High (+1)
Ball diameter d, (mm)	x_1	25	40	55
Grinding time t, (min)	x_2	2	6	10
Bond work index W_i, (kWh/t)	x_3	12	17	22

The response measured from the experiment was the 80% passing size (d_{80}) in three size fractions (coarse, middle, and fine) of the coal. They were: y_1 = -3350 + 1700 (μm), y_2 = -1700 + 710 (μm), and y_3 = -710 (μm).

For three factors, the Box-Behnken design required 15 runs which included three center points. The Box-Behnken design and the experimental results were shown in Table 5 of the paper and are reproduced here as Table 6.2. MATLAB 7.1 was used for the regression analysis.

Table 6.2: Box-Behnken design with actual/coded values for three size fractions and results. Corrected Table 5 of Aslan et al (2007).

Run no.	Actual and coded level of variables			Experimental d_{80}		
	x_1 (d) (mm)	x_2 (t) (min)	x_3 (W_i) (kWh/t)	-3350+1700 (μm)	1700+710 (μm)	-710 (μm)
1	2.5 (-1)	2 (-1)	17 (0)	2550	910	250
2	5.5 (1)	2 (-1)	17 (0)	2100	900	370
3	2.5 (-1)	10 (1)	17 (0)	750	230	150
4	5.5 (1)	10 (1)	17 (0)	980	410	180
5	2.5 (-1)	6 (0)	12 (-1)	770	450	105
6	5.5 (1)	6 (0)	12 (-1)	840	520	180
7	2.5 (-1)	6 (0)	22 (1)	1070	625	185
8	5.5 (1)	6 (0)	22 (1)	1050	640	190
9	4.0 (0)	2 (-1)	12 (-1)	1100	830	120
10	4.0 (0)	10 (1)	12 (-1)	300	200	85
11	4.0 (0)	2 (-1)	22 (1)	1280	950	180
12	4.0 (0)	10 (1)	22 (1)	450	410	90
13	4.0 (0)	6 (0)	17 (0)	850	540	145
14	4.0 (0)	6 (0)	17 (0)	850	540	145
15	4.0 (0)	6 (0)	17 (0)	850	540	145

The authors fitted a full second-order regression model consisting of linear, quadratic, and two-factor interaction terms to each of the responses. The ANOVA results were not given hence there was no information on whether the estimated regression coefficients were statistical significant or not. Only the regression models were given together with the R^2 values. No other goodness-of-fit statistics were given and there was no mention of any regression assumption checks.

For y_1 = -3350 + 1700 μm size fraction, the model equation in coded units given by the authors (Equation 7 in the paper) was:

$$y_1 = 850 - 21.25\,x_1 - 568.75\,x_2 + 105\,x_3 + 447.5\,x_1^2 + 297.5\,x_2^2 - 365\,x_3^2 + 170\,x_1x_2 - 225\,x_1x_3 - 7.5\,x_2x_3$$

$R^2 = 0.96$

For y_2 = -1700 + 710 μm size fraction, the model equation in coded units given by the authors (Equation 8 in the paper) was:

$$y_2 = 540 + 33.13\,x_1 - 292.5\,x_2 + 76.88\,x_3 + 15.63\,x_1^2 + 56.88\,x_2^2 + 0.63\,x_3^2 + 47.5\,x_1x_2 - 11.25\,x_1x_3 + 22.5\,x_2x_3$$

$R^2 = 0.98$

For y_3 = - 710 μm size fraction, the model equation in coded units given by the authors (Equation 9 in the paper) was:

$$y_3 = 145 + 28.75\,x_1 - 51.88\,x_2 + 19.38\,x_3 + 69.38\,x_1^2 + 23.13\,x_2^2 - 49.38\,x_3^2 - 22.5\,x_1x_2 - 17.5\,x_1x_3 - 13.75\,x_2x_3$$

$R^2 = 0.94$

The experimental values were then compared to those obtained by the above regression models. The comparisons were shown in Table 6 of the paper and are reproduced here as Table 6.3.

Table 6.3: Experimental and predicted values of d_{80} for three size fractions. Table 6 of Aslan et al (2007).

Test No.	-3350+1700 (μm)		1700+710 (μm)		-710 (μm)	
	Experimental, d_{80} (μm)	Predicted, d_{80} (μm)	Experimental, d_{80} (μm)	Predicted, d_{80} (μm)	Experimental, d_{80} (μm)	Predicted, d_{80} (μm)
1	2550	2355	910	919	250	238
2	2100	1973	900	890	370	340
3	750	778	230	239	150	179
4	980	1175	410	400	180	192
5	770	841	450	469	105	103
6	840	814	520	490	180	188
7	1070	1066	625	578	185	169
8	1050	1050	640	688	190	199
9	1100	1224	830	802	120	134
10	300	131	200	239	85	65
11	1280	1479	950	978	180	208
12	450	296	410	371	90	69
13	850	850	540	540	145	145
14	850	850	540	540	145	145
15	850	850	540	540	145	145

Reanalyses of the results showed that the predicted R^2 for y_1 and y_3 are actually quite low, 0.242 and 0.063, respectively. Furthermore, reduced quadratic models (using only statistically significant terms at the 5% level) with a logarithmic transformation on the responses gave much better results.

Case Study #6.2

Cai, L., Haifu Wang, and Yawei Fu (2013): Freeze-thaw resistance of alkali-slag concrete based on response surface methodology. Construction and Building Materials, 49, pp. 70-76.

This study used response surface methodology based on a Box-Behnken design to investigate the freeze-thaw resistance of alkali-slag concrete (ASC). Factors studied were the activator solution-slag ratio (A/S), slag content, and sand ratio. The preparation of the ASC was described in the paper. The factors and their levels used in the experiment were shown in Table 2 of the paper and are reproduced here as Table 6.4.

The primary response of interest was the frost resistance coefficient, D_F, an internationally used evaluation index. The definition and calculation of this coefficient were given in the paper. The authors also studied the correlations between D_F and the air bubble spacing coefficient, and between D_F and the specific surface area. However, the air bubble spacing coefficient and specific surface area were not modelled using regression analysis. Hence, they will not be considered in this case study. The primary focus is on D_F.

Table 6.4: Levels of factors of RSM. Table 2 of Cai et al (2013).

Factor	Code	Levels of code		
		-1	0	1
A/S	A	0.54	0.56	0.58
slag /(g/cm^3)	B	0.4	0.42	0.44
sand ratio	C	0.32	0.34	0.36

The experimental design was based on a three-factor Box-Behnken design with five center points. The total number of runs was 17. The experimental design and results for D_F were shown in Table 3 of the paper. These are reproduced here as Table 6.5 with the results of the air bubble spacing coefficient, air bubble specific surface area, and grades of freeze-thaw resistance (all equal to F300). Design-Expert (version unknown) was used for the design and analysis of the experiment.

Table 6.5: Design of tests based on BBD and test results. Extracted from Table 3 of Cai et al (2013).

Test No.	Design of tests			
	A	B /(g/cm^3)	C	D_F /%
1	-1	-1	0	92.2
2	1	-1	0	83.1
3	1	0	-1	84.0
4	0	0	0	91.3
5	1	0	1	86.6
6	0	0	0	90.7
7	0	0	0	91.4
8	0	1	-1	90.3
9	-1	0	1	93.2
10	0	-1	1	88.4
11	1	1	0	89.3
12	-1	0	-1	91.1
13	0	-1	-1	86.2
14	-1	1	0	98.1
15	0	0	0	90.3
16	0	1	1	92.7
17	0	0	0	91.7

The authors indicated that a standard second-order response regression model consisting of linear, quadratic, and two-factor interaction terms were fitted. The ANOVA table without the individual model terms was shown in Table 4 of the paper and is reproduced here as Table 6.6. The significance tests of the regression coefficients were shown in Table 5 of the paper and are reproduced here as Table 6.7.

Table 6.6: Variance analysis of the model. Table 4 of Cai et al (2013).

Source	Sum squares	df	Mean square	F-value	p-value
Model	20547.21	8	2568.40	57.61	<0.0001
Residual error	356.67	8	44.58		
Lack of fit	227.87	4	56.97	1.77	
Pure error	128.80	4	32.20		
Total	20903.88	16			

Table 6.7: Significance test of the regression coefficients. Table 5 of Cai et al (2013).

Term	Regression coefficient	Std Dev.	Lower C.I. of 95%	Upper C.I. of 95%	P-value
A	-39.5	2.36	-44.94	-34.06	<0.0001
B	21	3.34	13.30	28.70	0.0002
C	11.63	2.36	6.18	17.07	0.0012
AB	0.75	3.34	-6.95	8.45	0.8279
A^2	-5.4	3.25	-12.90	2.10	0.1356
B^2	1.35	3.25	-6.15	8.85	0.6891
C^3	-18.15	3.25	-25.65	-10.65	0.0005
A^2B	9.25	4.72	-1.64	20.14	0.0858

Note that the authors did not include the two-factor interactions of AC and BC in the model and they added the A^2B term, which is not typical in response surface modelling. No explanation was given for the choice of model. Furthermore, the responses seem to have been multiplied by 10 before analysis. The regression model for D_F given by the authors (Equation 3) was:

$$D_F = 910.80 - 39.5A + 21.00B + 11.63C + 0.75AB - 5.40A^2 + 1.35B^2 - 18.15C^2 + 9.25A^2B$$

More accurately, the above equation is for $10D_F$. The adjusted R^2 was 0.9659. No other goodness-of-fit statistics were given. From Table 6.7, many of the model terms are not statistically significant at the 5% level. A reduced model using only statistically significant terms would have given a better model.

Case Study #6.3

Jalali, M. R. and Mohammad Amin Sobati (2017): Intensification of oxidative desulfurization of gas oil by ultrasound irradiation: Optimization using Box-Behnken design (BBD). Applied Thermal Engineering, 111, pp. 1158-1170.

This study used response surface methodology via a Box-Behnken design to investigate the effect of three influential parameters on the sulfur removal of gas oil in an ultrasound assisted oxidative desulfurization (UAOD) process. The details of the process and the experimental setup were described in the paper. The factors investigated and levels used in the experiment were shown in Table 3 of the paper and are reproduced here as Table 6.8.

Table 6.8: Experimental range and factor level of process variables applied in the experimental design. Table 3 of Jalali et al (2017).

Independent variables	Coded variables	Range and levels		
		-1	0	1
Oxidant to sulfur molar ratio	x_1	10	30	50
Formic acid to oxidant molar ratio	x_2	2	3	4
Sonication time (min)	x_3	2	16	30

A Box-Behnken response surface design for three factors and five center points was used. The response of interest was the sulfur removal percentage from the gas oil. Design-Expert 7.0.0 was used for the design and analysis of the experiment.

Table 6.9: Box-Behnken design with experimental and predicted response for UAOD of gas oil. Table 4 of Jalali et al (2017).

Run	Coded values			Real varaibles			Sulfur removal (%)			
	x_1	x_2	x_3	Oxidant to Sulfur molar ratio	Acid to oxidant molar ratio	Sonification Time (min)	Experimental	Std. Dev	Predicted	Error (%)
1	0	0	0	30	3	16	86.15	0.31	85.47	0.79
2	0	1	1	30	4	30	82.62	0.43	81.44	1.43
3	0	0	0	30	3	16	86.20	0.38	85.47	0.85
4	0	0	0	30	3	16	84.03	0.67	85.47	1.71
5	1	1	0	50	4	16	86.29	0.36	86.59	0.35
6	0	0	0	30	3	16	85.70	0.47	85.47	0.27
7	1	0	-1	50	3	2	78.37	0.72	78.17	0.26
8	1	0	1	50	3	30	88.28	0.52	89.19	1.03
9	0	0	0	30	3	16	85.66	0.41	85.47	0.22
10	-1	0	-1	10	3	2	66.06	0.62	65.17	1.35
11	1	-1	0	50	2	16	83.85	0.51	82.87	1.17
12	-1	-1	0	10	2	16	65.50	0.58	64.51	1.51
13	0	-1	-1	30	2	2	55.66	0.37	56.86	2.16
14	-1	0	1	10	3	30	80.45	0.39	80.67	0.27
15	-1	1	0	10	4	16	82.44	0.68	83.43	1.20
16	0	1	-1	30	4	2	81.40	0.33	81.32	0.10
17	0	-1	1	30	2	30	83.17	0.56	83.26	0.11

The experimental design and the results were shown in Table 4 of the paper and are reproduced here as Table 6.9. A full-second order regression model consisting of linear, quadratic, and two-factor interaction terms were fitted to the response as a function of the three factors. The ANOVA results together with the goodness-of-fit statistics were shown in Tables 5 and 6 of the paper. These tables are combined here as Table 6.10.

Table 6.10: Analysis of variance (ANOVA) for the response surface quadratic model and goodness-of-fit statistics. Tables 5 and 6 of Jalali et al (2017).

Source	Sum of squares	DF	Mean square	F-value	P-value
Model	1311.00	9	145.67	105.18	<0.0001
x_1	231.44	1	231.44	167.11	<0.0001
x_2	256.25	1	256.25	185.03	<0.0001
x_3	351.48	1	351.48	253.79	<0.0001
$x_1.x_2$	57.76	1	57.76	41.7	0.0003
$x_1.x_3$	5.01	1	5.01	3.62	0.0989
$x_2.x_3$	172.79	1	172.79	124.77	<0.0001
x_1^2	13.21	1	13.21	9.54	0.0176
x_2^2	79.66	1	79.66	57.52	0.0001
x_3^2	122.94	1	122.94	88.77	<0.0001
Residual	9.69	7	1.38		
Lack of fit	6.66	3	2.22	2.93	0.1631
Pure error	3.03	4	0.76		
Cor total	1320.70	16			

Std dev.	1.18	R-squared	0.9927
Coefficient of variation (C.V. %)	1.47	Adjusted R-squared	0.9832
Adequate precision	35.816	Predicted R-squared	0.9157

The prediction model, in terms of coded units, proposed by the authors was given as Equation 7 in the paper and is reproduced here:

$$Y\ (\text{Sulfur removal}, \%)$$
$$= 85.47 + 5.38\,x_1 + 5.66\,x_2 + 6.63\,x_3 - 3.8\,x_1x_2 - 1.12\,x_1x_3 - 6.57\,x_2x_3$$
$$- 1.77\,x_1^2 - 4.35\,x_2^2 - 5.40\,x_3^2$$

Note that the term x_1x_3 was not statistically significant at the 5% level but was included in the model. Assumptions of regression were checked and found to be fulfilled. The predicted values for each run combination of the experiment were also shown in Table 6.9, together with the absolute percentage error. The model was validated using three repeated runs at the optimum oxidation condition with x_1=46.36, x_2=3.22, and x_3=19.81 at constant temperature of 50 °C, ultrasound power per gas volume of 7.78, and 1 extraction stage. The predicted value was 89% and the average of the three experimental values was 87.01% with a standard deviation of 0.423% which was deemed accurate by the authors.

Case Study #6.4

Kilickap, E. and Mesut Huseyinoglu (2010): Selection of optimum parameters on burr height using response surface methodology and genetic algorithm in drilling of AISI 304 stainless steel. Materials and Manufacturing Processes, 25, pp. 1068-1076.

This study investigated the optimum parameters on burr height using two methodologies. One was based on response surface methodology and another on genetic algorithm. This case study will only consider the method based on response surface methodology via a three-factor Box-Behnken design. The influence of cutting parameters – cutting speed, feed rate, and the point angle was investigated. The response was the burr height. The burr height is an important consideration in drilling of mechanical components. This study used a SX XHMT vertical drilling machine and AISI 304 stainless steel for the drilling experiments. The factors and levels used in the experiment were shown in Table 5 of the paper and are reproduced here as Table 6.11.

Table 6.11: Experimental factors and their levels. Table 5 of Kilickap et al (2010).

Symbol	Drilling parameter	Level 1 (-1)	Level 2 (0)	Level 3 (+1)
A	Cutting speed, V (m/min)	4	8	12
B	Feed rate, f (mm/rev)	0.1	0.2	0.3
C	Point angle, Θ (°)	90 (1)	118 (2)	135 (3)

The three-factor Box-Behnken design consisted of 12 runs and five center points. The design and the resulting burr heights were shown in Table 6 of the paper. The table is reproduced here as Table 6.12. Design-Expert 6.0 was used for the design and analysis of the experiment.

Table 6.12: Design matrix and observed values of butt height. Table 6 of Kilickap et al (2010).

Trial No.	Cutting speed	Feed rate	Point angle	Burr height (mm)
1	-1	-1	0	0.72
2	1	-1	0	1.40
3	-1	1	0	1.24
4	1	1	0	2.08
5	-1	0	-1	1.25
6	1	0	-1	2.10
7	-1	0	1	0.82
8	1	0	1	1.45
9	0	-1	-1	0.99
10	0	1	-1	1.80
11	0	-1	1	0.74
12	0	1	1	1.42
13	0	0	0	1.44
14	0	0	0	1.44
15	0	0	0	1.44
16	0	0	0	1.44
17	0	0	0	1.44

A quadratic model was suggested by the software. The ANOVA results for the quadratic model were shown in Table 8 of the paper, together with the goodness-of-fit statistics. This table is reproduced here as Table 6.13. No attempt was made by the authors to reduce the model.

Table 6.13: ANOVA table for response surface quadratic model. Table 8 of Kilickap et al (2010).

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	2.52	9	0.28	54.35	< 0.0001	significant
A-A	1.13	1	1.13	218.6	< 0.0001	
B-B	0.90	1	0.90	175.76	< 0.0001	
C-C	0.37	1	0.37	71.02	< 0.0001	
A^2	8.059E-03	1	8.059E-03	1.57	0.2510	
B^2	0.064	1	0.064	12.53	0.0095	
C^2	0.026	1	0.026	5.07	0.0590	
AB	6.40E-03	1	6.400E-03	1.24	0.3016	
AC	0.012	1	0.012	2.35	0.1691	
BC	4.225E-03	1	4.225E-03	0.821	0.3950	
Residual	0.036	7				
Cor Total	2.55	16				
S.D.	0.072	C.V.	5.25	R^2	0.9859	Pred. R^2 0.7743
Mean	1.37	PRESS	0.58	Adj. R^2	0.9678	Adeq. Precision 25.854

The prediction equation in actual factors for the burr height (H) was given as (Equation 5 in the paper):

$$H\text{ (mm)} = -0.38 + 0.0575\,A + 8.1625\,B + 0.27625\,C + 2.7343\text{E-03}\,A^2 - 12.375\,B^2 - 0.07875\,C^2 + 0.10\,AB - 0.01375\,AC - 0.325\,BC.$$

Note that the levels for the point angle (factor C) must be entered as 1, 2, and 3, and not as the actual angles given in Table 6.11. Five of the regression coefficients were actually statistically insignificant at the 5% level. These terms could have been removed for a better model.

The authors concluded that to achieve minimum burr height, a combination of low cutting speed and low feed rate with a larger point angle must be used in the drilling process.

Case Study #6.5

Liu Junyan, Lin Li, Lizhen Zhou, Bing Li, and Zhenbo Xu (2017): Effect of ultrasound treatment conditions on *Saccharomyces cerevisiae* by response surface methodology. Microbial Pathogenesis, 111 pp. 497-502.

This study used response surface methodology via a Box-Behnken design to investigate the effect of different ultrasound treatment conditions on the inactivation of *Saccharromyces cerevisiae*, a potential pathogen in the food industry. In particular, three factors were studied – ultrasonic power level, irradiating time, and the pulse duty ratio. The authors also investigated the effect of different cell concentration, pH and temperature but these factors were not part of the experiment. The definition of terms and methods and materials used for the experiment were described in the paper. The three factors and their levels used in the experiment are summarized in Table 6.14.

Table 6.14: Factors and levels used in the experiment.

Symbol	Variables	Low (-1)	Middle (0)	High (+1)
X_1	Ultrasonic power (W)	82.3	246.8	411.4
X_2	Irradiating time (min)	4	8	12
X_3	Pulse duty ratio	0.1	0.5	0.9

Table 6.15: Box-Behnken experimental design and results. Table 1 of Liu et al (2017).

Run number	Independent variables			Response value
	Power (X_1)	Time (X_2)	Pulse duty ratio (X_3)	Y
1	246.8	4	0.1	3.11
2	82.3	12	0.5	2.11
3	246.8	8	0.5	3.09
4	82.3	4	0.5	0.56
5	246.8	12	0.1	4.23
6	246.8	4	0.9	3.55
7	246.8	8	0.5	3.12
8	246.8	12	0.9	4.4
9	246.8	8	0.5	3.35
10	82.3	8	0.1	1.67
11	82.3	8	0.9	1.56
12	411.4	12	0.5	5.97
13	411.4	8	0.9	5.48
14	246.8	8	0.5	3.21
15	411.4	4	0.5	5.25
16	411.4	8	0.1	5.65
17	246.8	8	0.5	3.22

A three-factor Box-Behnken design with five center points (total of 17 points) was used for the experiment. Design-Expert 7.0 was used for the design and subsequent analysis of the results. The response was based on a plate counting method to identify ultrasound treatment efficiency. The response Y was expressed as $-\lg(N/N_0)$, where, N_0 is the initial cell number and N is the cell number in the sample after different treatments. The average of at least three plates was used as the response. The experimental design and the results were shown in Table 1 of the paper and are reproduced here as Table 6.15.

The authors fitted a standard second-order response surface model with linear, quadratic, and two-factor interaction terms to the response. The ANOVA results were shown in Table 2 of the paper. The table is reproduced here as Table 6.16.

Table 6.16: ANOVA results for the response surface model. Table 2 of Liu et al (2017).

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	37.18	9	4.13	141.38	< 0.0001	significant
X_1	33.83	1	33.83	1157.44	< 0.0001	
X_2	2.25	1	2.25	76.89	< 0.0001	
X_3	0.014	1	0.014	0.47	0.5169	
X_1X_2	0.17	1	0.17	5.89	0.0456	
X_1X_3	9.00E-04	1	9.00E-04	0.031	0.8657	
X_2X_3	0.018	1	0.018	0.62	0.4556	
X_1^2	1.857E-03	1	1.857E-03	0.064	0.8082	
X_2^2	0.27	1	0.27	9.26	0.0188	
X_3^2	0.58	1	0.58	19.83	0.0030	
Residual	0.20	7	0.029			
Lack of Fit	0.16	3	0.054	5.24	0.0717	not significant
Pure Error	0.042	4	0.010			
Cor Total	37.39	16				
R-squared	0.9945	Predicted R-squared	0.9285			
Adjusted R-square	0.9875					

The prediction equation was given as Equation 2 in the paper as:

$$Y = 3.20 + 2.06X_1 + 0.53X_2 + 0.041X_3 - 0.21X_1X_2 - 0.015X_1X_3 - 0.068\,X_2X_3 - 0.021X_1^2 + 0.25X_2^2 + 0.37X_3^2$$

Note that the prediction equation included all terms regardless of their statistical significance. A reduced quadratic model with only statistically significant terms at the 5% level would have provided a higher predicted R^2.

The authors concluded that ultrasound power played the most important role in the irradiation process of *Saccharromyces cerevisiae* during ultrasound treatment in the food industry.

Case Study #6.6

Muthukumar, M., D. Mohan, and M. Rajendran (2003): Optimization of mix proportions of mineral aggregates using Box-Behnken design of experiments. Cement and Concrete Composites, 25, pp. 751-758.

This study investigated the optimal mix proportions of silica aggregates for use in polymer concrete using a six-factor Box-Behnken response surface design. The six factors were the six different grades of particle sizes ranging in size from 0.15 to 9.72 mm. The primary objective was to determine what combination of particle sizes would minimize the void content of the resulting concrete. The materials and methods used for the experiment were described in the paper. The factors and levels used in the experiment were shown in Table 1 of the paper and are reproduced here as Table 6.17.

Table 6.17: Levels of variables chose for the design. Table 1 of Muthukumar et al (2003).

S. no.	Grade	Particle size, mm		Input level, g (coded)		
		Min	Max	Low	Medium	High
1	A	4.76	9.52	0 (-1)	50 (0)	100 (+1)
2	B	2.38	4.76	0 (-1)	50 (0)	100 (+1)
3	C	1.19	2.38	0 (-1)	50 (0)	100 (+1)
4	D	0.60	1.19	0 (-1)	50 (0)	100 (+1)
5	E	0.30	0.60	0 (-1)	50 (0)	100 (+1)
6	F	0.15	0.30	0 (-1)	50 (0)	100 (+1)

The Box-Behnken design with the six factors used 54 run combinations (49 points and six center points). Design-Expert (version unknown) was used for the design and analysis of the experiment. The response was the void content (%). The experimental design together with the results were shown in Table 5 of the paper and are reproduced here as Table 6.18.

A second-order polynomial regression was fitted to the experimental results and the ANOVA results were shown in Table 4 of the paper (reproduced here as Table 6.19). The prediction equation (in coded factors) for the void content was the full second-order polynomial equation given in the paper as:

$$\begin{aligned}\text{Predicted void content (\%)} &= 24.81 - 1.986x_1 - 1.147x_2 - 0.132x_3 + 1.210x_4 + 1.088x_5 - 0.520x_6 \\ &+ 0.602x_1^2 + 0.571x_2^2 + 0.239x_3^2 + 0.192x_4^2 + 0.684x_5^2 + 1.401x_6^2 + 1.155x_1x_2 + 0.443x_1x_3 \\ &+ 0.032x_1x_4 - 0.791x_1x_5 - 0.12x_1x_6 + 0.899x_2x_3 + 0.030x_2x_4 - 0.712x_2x_5 - 0.609x_2x_6 - 0.375x_3x_4 \\ &- 0.981x_3x_5 - 0.939x_3x_6 - 0.281x_4x_5 - 0.580x_4x_6 + 1.336x_5x_6 \qquad (R^2 = 0.9174)\end{aligned}$$

As can be seen from the ANOVA table, more than 10 of the regression coefficients were actually not statistically significant at the 5% level yet they were included in the prediction equation. The only goodness-of-fit statistic given was the R^2 value of 0.9174. The adjusted and predicted R^2 were not reported. The comparison of the experimental and predicted values were also shown in Table 6.18.

Table 6.18: Box Behnken design for the six variables and their experimental and predicted response (% void content). Table 5 of Muthukumar et al (2003).

Run	A	B	C	D	E	F	Experimental response	Predicted response
1	-1	-1	0	-1	0	0	30.16	29.32
2	0	-1	-1	0	-1	0	25.46	25.70
3	0	1	0	0	1	1	26.37	26.90
4	0	0	-1	1	0	1	27.83	28.20
5	0	0	-1	-1	0	1	27.62	26.19
6	1	0	0	1	-1	0	24.96	25.53
7	-1	-1	0	1	0	0	31.28	31.61
8	-1	0	1	0	0	1	27.37	27.13
9	1	0	0	1	1	0	26.12	25.56
10	1	0	-1	0	0	1	24.87	25.06
11	0	0	1	-1	0	-1	27.25	26.55
12	-1	0	-1	0	0	-1	29.41	29.07
13	0	-1	0	0	1	-1	29.53	28.99
14	-1	0	0	1	-1	0	28.08	27.85
15	1	-1	0	-1	0	0	21.96	22.97
16	1	0	0	-1	-1	0	23.83	22.48
17	-1	0	-1	0	0	1	30.66	30.15
18	0	0	1	1	0	-1	28.28	29.38
19	0	-1	1	0	1	0	27.91	27.24
20	0	-1	0	0	-1	-1	28.28	28.06
21	1	0	1	0	0	1	23.79	23.80
22	-1	0	0	1	1	0	30.12	31.05
23	0	1	1	0	1	0	25.87	25.32
24	-1	1	0	1	0	0	27.66	27.07
25	0	-1	0	0	1	1	30.28	31.84
26	0	1	0	0	-1	-1	30.28	28.41
27	0	0	0	0	0	0	25.25	24.81
28	-1	0	0	-1	-1	0	23.96	24.94
29	1	1	0	-1	0	0	22.84	22.93
30	0	0	0	0	0	0	24.33	24.81
31	0	-1	0	0	-1	1	25.62	25.57
32	0	1	0	0	-1	1	23.25	23.48
33	0	-1	1	0	-1	0	25.33	25.60
34	-1	0	0	-1	1	0	29.41	29.26
35	0	0	1	-1	0	1	24.25	24.80
36	0	1	1	0	-1	0	26.08	26.53
37	0	0	0	0	0	0	24.83	24.81
38	1	0	0	-1	1	0	23.83	23.64
39	0	0	0	0	0	0	25.33	24.81
40	0	0	1	1	0	1	26.50	25.31
41	0	1	-1	0	-1	0	22.05	23.03
42	-1	1	0	-1	0	0	24.13	24.65
43	0	-1	-1	0	1	0	31.40	31.26
44	0	0	-1	1	0	-1	28.74	28.52
45	-1	0	1	0	0	-1	29.66	29.80
46	1	0	-1	0	0	-1	24.54	24.46
47	0	0	-1	-1	0	-1	22.67	24.19
48	0	1	-1	0	1	0	26.33	25.75
49	1	0	1	0	0	-1	26.12	26.96
50	0	1	0	0	1	-1	26.12	26.49
51	0	0	0	0	0	0	24.50	24.81
52	1	-1	0	1	0	0	26.33	25.39
53	0	0	0	0	0	0	24.62	24.81
54	1	1	0	1	0	0	25.04	25.47

Table 6.19: ANOVA for response surface quadratic model. Table 4 of Muthukumar et al (2003).

Source	Sum of Squares	DF	Mean Square	F-value	p-value
Model	300.71	27	11.140	10.699	< 0.0001
x_1	94.68	1	94.685	90.956	< 0.0001
x_2	31.56	1	31.556	30.313	< 0.0001
x_3	0.42	1	0.419	0.402	0.5315
x_4	35.11	1	35.114	33.731	< 0.0001
x_5	28.41	1	28.406	27.287	< 0.0001
x_6	6.48	1	6.479	6.224	0.0193
x_1^2	3.73	1	3.730	3.583	0.0695
x_2^2	3.35	1	3.348	3.216	0.0845
x_3^2	0.59	1	0.587	0.564	0.4594
x_4^2	0.38	1	0.380	0.365	0.5509
x_5^2	4.82	1	4.817	4.627	0.0409
x_6^2	20.20	1	20.200	19.404	0.0002
$x_1.x_2$	10.67	1	10.672	10.252	0.0036
$x_1.x_3$	1.57	1	1.566	1.505	0.2309
x_1x_4	0.02	1	0.016	0.016	0.9015
x_1x_5	5.01	1	5.009	4.811	0.0374
x_1x_6	0.12	1	0.115	0.111	0.7421
x_2x_3	6.46	1	6.462	6.208	0.0194
x_2x_4	0.01	1	0.007	0.007	0.9344
x_2x_5	8.12	1	8.123	7.803	0.0097
x_2x_6	2.96	1	2.965	2.848	0.1035
x_3x_4	1.13	1	1.125	1.081	0.3081
x_3x_5	7.70	1	7.703	7.399	0.0115
x_3x_6	14.10	1	14.100	13.545	0.0011
x_4x_5	0.63	1	0.633	0.608	0.4426
x_4x_6	2.69	1	2.691	2.585	0.1199
x_5x_6	14.28	1	14.285	13.722	0.0010
Residual	27.07	26	1.041		
Lack of Fit	26.24	21	1.249	7.554	0.0169
Pure Error	0.83	5	0.165		
Cor Total	327.78	53			

From Table 6.19, it can be seen that the lack of fit of the fitted model was statistically significant at 5% level. This lack of fit was not addressed in the paper. If a reduced quadratic model was fitted with only statistically significant terms, the predicted R^2 would have increased from 0.578 to 0.726.

The authors then obtained the optimal combination to give the minimum void content. This was compared to the minimum void content obtained from the 54 experiments. This comparison was shown in Table 6 of the paper and is shown here as Table 6.20.

Table 6.20: Experimental combination vs. optimized combination. Table 6 of Muthukumar et al (2003).

S. No.	Variable	Experimental combination having minimum void			Optimized combination having minimum void		
		Coded	Uncoded	%	Coded	Uncoded	%
1	A	1	100	40	0.81	90.5	39.6
2	B	-1	0	0	0.53	76.5	33.5
3	C	0	50	20	-1	0	0
4	D	-1	0	0	-1	0	0
5	E	0	50	20	-1	0	0
6	F	0	50	20	0.23	61.5	26.9
		% void	21.96			21.002	

Note that the optimum void content obtained using the prediction equation was not verified using additional experiments with the optimal combination.

Case Study #6.7

Sharifi, H., Seyed Majid Zabihzadeh, and Mohsen Ghorbani (2018): The application of response surface methodology on the synthesis of conductive polyaniline/cellulosic fiber nanocomposites. Carbohydrate Polymers, 194, pp. 384-394.

This study investigated the effects of three critical parameters (factors) on the conductivity of polyaniline/cellulosic fibers nanocomposites using response surface methodology based on a Box-Behnken design. These parameters were the surfactant type (X_A), mass ratio of fibers/aniline (X_B), and time of polymerization (X_C). The factors and their ranges were shown in Table 1 of the paper and are extracted here as Table 6.21.

Table 6.21: Factors and levels used for conductivity experiment. Extracted from Table 1 of Sharifi et al (2018).

Coded	Factors	Range and levels		
		Low (-1)	Middle (0)	Highg (+1)
A	surfactant type	anionic	non ionic	cationic
B	mass ratio of fibers/aniline	0.5	1	1.5
C	time of polymerization (hr)	4	8	12

The dependent or response variable was the conductivity (S m^{-1}) of the polyaniline/cellulosic fiber nanocomposite. The preparation of the experimental samples and measuring of the responses were explained in the paper.

A three-factor Box-Behnken design with three center points (total of 15 points) was used for the conductivity experiment. Design-Expert Version 6 was used for the design of the experiment and subsequent statistical analysis and modelling. The experimental design and results were shown in Table 1 of the paper and are reproduced here as Table 6.22.

Table 6.22: Experimental design in terms of coded factors and results of the Box-Behnken design for conductivity of samples. Extracted from Table 1 of Sharifi et al (2018).

Sample No.	Factors			Conductivity (Sm^{-1})	
	A	B	C	Experimental	Predicted
PC-1	0	-1	1	1.060	1.114
PC-2	-1	-1	0	0.550	0.601
PC-3	-1	0	-1	0.350	0.369
PC-4	1	1	0	0.940	0.889
PC-5	0	0	0	0.650	0.624
PC-6	1	0	-1	0.823	0.883
PC-7	0	0	0	0.586	0.624
PC-8	0	1	-1	0.398	0.389
PC-9	1	0	1	1.357	1.337
PC-10	-1	0	1	0.705	0.645
PC-11	-1	1	0	0.578	0.567
PC-12	0	1	1	0.446	0.517
PC-13	0	0	0	0.637	0.624
PC-14	1	-1	0	1.475	1.486
PC-15	0	-1	-1	0.540	0.469

A standard second-order polynomial equation (linear + quadratic + two-factor interaction terms) was fitted to the responses. The ANOVA results for the full model were shown in Table 2 of the paper and are reproduced here as Table 6.23.

Table 6.23: Analysis of variance (ANOVA) for the conductivity of samples from Box-Behnken. Table 2 of Sharifi et al (2018).

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	1.55	9	0.1717	32.77	0.0006
A-Surfactant type	0.73	1	0.73	138.79	< 0.0001
B-Mass ratio of fibers/anil	0.20	1	0.1994	38.05	0.0016
C-Time of polymerization	0.27	1	0.2654	50.64	0.0008
AB	0.079	1	0.07924	15.12	0.0115
AC	8.010×10^{-3}	1	8.010×10^{-3}	1.53	0.2712
BC	0.056	1	0.056	10.63	0.0224
A^2	0.19	1	0.19	37.14	0.0017
B^2	3.742×10^{-3}	1	3.742×10^{-3}	0.71	0.4367
C^2	7.532×10^{-3}	1	7.532×10^{-3}	1.44	0.2843
Residual	0.026	5	5.24×10^{-3}		
Lack of Fit	0.024	3	7.97×10^{-3}	6.96	0.1281
Pure Error	2.289×10^{-3}	2	1.144×10^{-3}		
Cor Total	1.57	14			
Std. Dev.	0.072		R^2	0.9833	
C.V. %	9.79		Adjusted R^2	0.9533	
Mean	0.74		Predicted R^2	0.7533	
PRESS	0.39		Adeq Precision	18.882	

Based on the ANOVA results for the full model, the authors considered that fitted model was statistically significant and provided a very good fit. Hence, they suggested the following prediction equation in coded factors (Equation 4 in the paper):

$$\text{Conductivity} = 0.62433 + 0.30150\, X_A - 0.15788\, X_B + 0.18213\, X_C + 0.229583\, X_A^2 \\ + 0.031833\, X_B^2 - 0.045167\, X_C^2 - 0.14075\, X_A X_B + 0.04475\, X_A X_C - 0.11800\, X_B X_C$$

The predicted conductivity for the same run combinations are also shown in Table 6.22. Note that several of the coefficients in the prediction model are not statistically significant at the 5% level. The adequacy of the model was checked by the authors and found to be satisfactory. A reduced model using only statistically significant terms would have provided a model with higher predicted R^2.

The authors then validated the prediction model using three additional experimental runs - one run for each type of surfactant. The results were shown in Table 3 of the paper are shown here as Table 6.24.

Table 6.24: Comparing the experimental and predicted values. Table 3 of Sharifi et al (2018).

Run no.	Surfactant type	Mass ratio of fibers/aniline	Time of polymerization	Conductivity		|Error (%)|
				Experiment[a]	Predicted	
1	cationic	0.7	12	1.468±0.046	1.52152	3.64
2	anionic	1.1	10	0.643±0.0195	0.59153	1.9
3	non ionic	1.4	6	0.466±0.064	0.45923	1.45

[a] Mean ± standard deviation

The interpretation of the coefficients and results were given in the paper. The optimum conditions maximizing conductivity were found to be: use of cationic surfactant, mass ratio of fibers/aniline of about 0.5, and time of polymerization of 8 hours. The maximum conductivity obtained was 1.48587 S m^{-1}. The optimization technique used was not described in the paper.

Case Study #6.8

Zhang, H., Yanli Li, and Xiaogang Wu (2012): Statistical experiment design approach for the treatment of landfill leachate by photoelectro-fenton process. ASCE Journal of Environmental Engineering, Vol. 138, No. 3, pp. 278-285.

This study investigated the influence of three different variables – ferrous ion dosage, hydrogen peroxide concentration, and current density on the photoelectro-Fenton process for the treatment of landfill leachate using response surface methodology. A Box-Behnken design was used to develop the prediction models for two responses – color removal and COD removal. According to the authors, among various advanced oxidation processes for landfill leachate treatment, the Fenton process has been widely used due to its simplicity and ease of implementation.

The factors and levels considered in the experiment are shown in Table 6.25.

Table 6.25: Factors and levels used in the landfill treatment experiment.

Factor	Factor name and units	Levels		
		Low (-1)	Middle (0)	High (+1)
x_1	Ferrous iron dosage (Fe^{2+}), mmol L^{-1}	8.75	29.75	50.75
x_2	Hydrogen peroxide concentration (H_2O_2), mmol L^{-1}	87.5	219.0	350.0
x_3	Current density, mA cm^{-2}	8.33	16.67	25.00

A Box-Behnken design with three center points (total of 15 runs) was used to generate the design points. The two responses measured were: the color removal percentage (Y_1) and the COD removal percentage (Y_2). The design matrix in coded and uncoded units and the responses were shown in Table 1 of the paper and are reproduced here as Table 6.26. Design-Expert software (version unknown) was used to design and analyze of the experiment.

Table 6.26: Design matrix in coded and uncoded units and the experimental responses. Table 1 of Zhang et al (2012).

	Uncoded and coded levels of variables			Color removal (%) (Y_1)		COD removal (%) (Y_2)	
Std Order	Fe^{2+} (mmol L^{-1}) (x_1)	H_2O_2 (mmol L^{-1}) (x_2)	Current density (mA cm^{-2}) (x_3)	Observed	Predicted	Observed	Predicted
1	8.75 (-1)	87.5 (-1)	16.67 (0)	83.5	82.4	47.9	48.47
2	50.75 (+1)	87.5 (-1)	16.67 (0)	79.6	81.2	60.5	63.79
3	8.75 (-1)	350 (+1)	16.67 (0)	94.2	92.6	61.7	58.4
4	50.75 (+1)	350 (+1)	16.67 (0)	90.8	91.9	75.8	75.3
5	8.75 (-1)	219 (0)	8.33 (-1)	84.4	85.5	50.6	50.5
6	50.75 (+1)	219 (0)	8.33 (-1)	89.4	87.9	71.4	68.5
7	8.75 (-1)	219 (0)	25 (+1)	91.9	93.4	57.6	60.5
8	50.75 (+1)	219 (0)	25 (+1)	90.0	88.9	74.6	74.7
9	29.75 (0)	87.5 (-1)	8.33 (-1)	80.2	80.2	59.1	58.6
10	29.75 (0)	350 (+1)	8.33 (-1)	90.5	90.9	65.1	68.5
11	29.75 (0)	87.5 (-1)	25 (+1)	85.4	84.9	69.4	66.0
12	29.75 (0)	350 (+1)	25 (+1)	95.0	95.1	77.0	77.5
13	29.75 (0)	219 (0)	16.67 (0)	92.4	92.5	73.4	74.1
14	29.75 (0)	219 (0)	16.67 (0)	91.2	92.5	74.4	74.1
15	29.75 (0)	219 (0)	16.67 (0)	94.0	92.5	74.4	74.1

The authors then fitted a standard second-order polynomial (linear + quadratic + two-factor interaction terms) to each of the responses. The reduced ANOVA tables for Y_1 (color removal percentage) and Y_2 (COD removal percentage) were shown as Tables 2 and 3 in the paper. These tables are reproduced here as Tables 6.27 and 6.28, respectively.

Table 6.27: Test for response function Y_1 (color removal in percentage). Table 2 of Zhang et al (2012).

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	308.53	4	77.13	19.46	0.0001
X_2	218.09	1	218.09	55.02	< 0.0001
X_3	39.43	1	39.43	9.95	0.0103
X_1^2	16.00	1	16.00	4.04	0.0723
X_2^2	38.29	1	38.29	9.66	0.0111
Residual	39.64	10	3.96		
Lack of Fit	35.77	8	4.47	2.31	0.3366
Pure Error	3.86	2	1.93		
Cor Total	348.17	14			

Using the terms in Table 6.27, the authors suggested the following prediction equation (equation 6 in the paper) for Y_1:

$$Y_1 = 91.65 + 5.22\,X_2 + 2.22\,X_3 - 2.08\,X_1^2 - 3.21\,X_2^2$$

The R^2 value for Y_1 was 0.8862, and adjusted R^2 value was 0.8406. The predicted R^2 value was not given. It should be noted that the X_1^2 term is not statistically significant at the 5% level and can be left out. If this term is included, then the X_1 term should also be included in the model for hierarchy.

Table 6.28: Test for response function Y_2 (COD removal in percentage). Table 3 of Zhang et al (2012).

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	1177.39	5	235.48	24.62	< 0.0001
X_1	520.40	1	520.40	54.41	< 0.0001
X_2	229.84	1	229.84	24.03	0.0008
X_3	132.56	1	132.56	13.86	0.0048
X_1^2	248.77	1	248.77	26.01	0.0006
X_2^2	62.08	1	62.08	6.49	0.0313
Residual	86.08	9	9.56		
Lack of Fit	85.40	7	12.20	35.79	0.0274
Pure Error	0.68	2	0.34		
Cor Total	1259.53	14			

For Y_2, the suggested prediction equation was (equation 7 in the paper):

$$Y_2 = 72.75 + 8.07\,X_1 + \ 5.36\,X_2 + 4.07\,X_3 - 8.18\,X_1^2 - 4.09\,X_2^2$$

The R^2 value for Y_2 was 0.9319, and adjusted R^2 value was 0.8940. The predicted R^2 value was not given. The coefficients of this model were all statistically significant at the 5% level and the model is hierarchical.

The assumptions of regression were checked by the authors and found to be satisfactory. The authors concluded that the photoelectron-Fenton process was capable of efficiently removing COD from landfill leachate. The results also showed that there was no interaction effect between the factors studied. The optimal conditions for color removal (of over 90%) and COD removal (of over 75%) were obtained using an overlay plot of the two response functions. One possible solution to achieve the above optimal condition was setting ferrous ion and hydrogen peroxide dosages at 38.99 and 153.2 mmol L^{-1}, respectively at a current density of 25 mA cm^{-2}. The authors did not use the desirable function approach for optimization available in Design-Expert.

7. RSM: CENTRAL COMPOSITE DESIGNS (Rotatable)

10 case studies are presented in this Chapter on the use RSM based on rotatable or near-rotatable central composite design or CCD designs. The number of factors are mostly three or four. The CCD is a well-known and popular classical response surface design with up to five levels (-α, -1, 0, +1, +α). For a rotatable design, $\alpha = \sqrt[4]{2^k}$. If $\alpha = \pm 1$, the CCD becomes a face-centered design or FCD (Chapter 8). The CCD is made up three parts – factorial points, axial points, and center points. The number of design points is $2^k + 2k + n_c$, where k is the number of factors, and n_c is the number of center points. The advantages of CCD is that it is amendable to sequential experimentation unlike the Box-Behnken design presented in Chapter 6. These designs generally require less runs than BBD especially when the number of factors are five or more because a resolution V design can be used for the factorial portion of the CCD.

Case Study #7.1

Ahmad, A. L., S. Ismail, and S. Bhatia (2005): Optimization of coagulation-flocculation process for palm oil mill effluent using response surface methodology. Environmental Science and Technology, 39, pp. 2828-2834.

This study used a three-factor near rotatable central composite design to optimize the coagulation-flocculation process for palm oil mill effluent. The factors and levels investigated were shown in Table 1 of the paper and are reproduced here as Table 7.1. The axial points for the CCD are at ±2.0 instead of 1.68 for a rotatable design.

Table 7.1: Experimental range and levels of the independent variables. Table 1 of Ahmad et al (2005).

	range and levels				
variables	-2	-1	0	1	2
A, coagulant dosage (mg/L)	0	7500	15000	22500	30000
B, flucculant dosage (mg/L)	0	125	250	375	500
C, pH	2	4	6	8	10

The coagulation-flocculation process and materials and methods used in the experiment were described in the paper. Two responses were of interest – turbidity (NTU) and water recovery (%). The objective was to seek factors setting that would minimize turbidity and maximize water recovery.

The CCD design layout in coded units and the responses were shown in Tables 2 and 3 of the paper. These tables are combined and reproduced here as Table 7.2. According to the authors, the CCD was a follow-up design after a 2-level factorial experiment with eight runs and four center points (runs 1-12). The 2-level design was then augmented with an additional six center points and two axial points (runs 13-20) giving a total of 20 design points. The additional eight runs were put in a second block. Hence, the CCD was analyzed as two blocks. Design-Expert 6.0 was used for the design and analysis of the experiment.

Table 7.2: CCD for the study of three experimental variables in coded units and results. Tables 2 and 3 of Ahmad et al (2005).

	factor			responses		
run no.	coagulant dosage (A)	flocculant dosage (B)	pH (C)	turbidity (NTU)	log10 of turbidity	water recovery (%)
1	-1	1	-1	230.35	2.3624	68.0
2	1	-1	1	92.14	1.9644	53.0
3	0	0	0	15.19	1.1816	79.8
4	-1	1	1	785.75	2.8953	52.0
5	-1	-1	1	892.40	2.9506	59.0
6	0	0	0	16.10	1.2068	74.8
7	-1	-1	-1	142.50	2.1538	74.0
8	1	1	-1	310.85	2.4926	81.0
9	0	0	0	13.91	1.1433	75.2
10	1	1	1	12.86	1.1092	68.4
11	1	-1	-1	279.15	2.4458	74.0
12	0	0	0	18.53	1.2679	77.2
13	2	0	0	32.57	1.5128	70.8
14	0	0	2	137.75	2.1391	51.0
15	0	0	0	29.53	1.4703	70.2
16	0	0	0	27.10	1.4330	74.0
17	0	-2	0	93.83	1.9723	38.0
18	0	0	-2	1409.25	3.1490	68.0
19	0	2	0	29.86	1.4751	73.6
20	-2	0	0	502.15	2.7008	92.8

A full second-order quadratic model was first fitted to the log base 10 transformations of the turbidity data. The ANOVA results were shown in Table 4 of the paper and are reproduced here as Table 7.3. Based on the ANOVA results, the authors concluded that the model was highly significant with high R^2 and adjusted R^2 values. They suggested the following regression equation as the empirical model (in terms of coded factors) for turbidity (Equation 3 in the paper):

$$\text{Turbidity} (\log_{10}) = 1.274 - 0.295A - 0.103B - 0.160C + 0.260A^2 + 0.164B^2 + 0.394C^2 - 0.120AB$$
$$-0.399AC - 0.146\ BC$$

Note that in Table 7.3, several of the terms were not statistically significant at the 5% level. Furthermore, there was also a significant lack of fit (p-value=0.0012). This was not mentioned in the paper. A reduced quadratic model with only statistically significant terms would have produced a model with a higher predicted R^2.

Table 7.3: ANOVA for response surface quadratic model (Turbidity). Table 4 of Ahmad et al (2005).

source	sum of squares	DF	mean square	F value	Prob > F
block	0.013	1	0.013		
model	7.951	9	0.883	12.296	0.0005
A	1.396	1	1.396	19.428	0.0017
B	0.17	1	0.17	2.367	0.1583
C	0.408	1	0.408	5.678	0.0410
A^2	1.617	1	1.617	22.499	0.0011
B^2	0.644	1	0.644	8.958	0.0151
C^2	3.723	1	3.723	51.812	<0.0001
AB	0.116	1	0.116	1.609	0.2365
AC	1.276	1	1.276	17.753	0.0023
BC	0.17	1	0.17	2.365	0.1585
residual	0.65	9	0.072		
lack of fit	0.64	5	0.127	47.573	0.0012
pure error	0.01	4	0.003		
cor total	8.61	19			

SD	0.27	R^2	0.9248
mean	1.95	adj R^2	0.8496
CV	13.75	pred R^2	0.5248
PRESS	4.09	adeq precision	10.47

A full second-order quadratic model was also fitted to the water recovery data. The ANOVA results were shown in Table 5 of the paper and are reproduced here as Table 7.4. Based on the ANOVA results, the authors also concluded that the model was highly significant with high R^2 and adjusted R^2 values. They suggested the following regression equation as the empirical model (in terms of coded factors) for water recovery (Equation 4 in the paper):

$$\text{Water recovery (\%)} = 74.91 - 1.288A + 5.038B - 6.162C + 1.462A^2 - 4.112B^2 + 4.112C^2 + 4.425AB - 0.325AC + 0.925\,BC$$

Note that in Table 7.4, all the terms involving factor A were not statistically significant at the 5% level. Furthermore, there was also a significant lack of fit (p-value =0.0051) and the predicted R^2 was negative indicating that the overall mean maybe a better predictor of the response than the quadratic model proposed. This was also not mentioned in the paper.

Table 7.4: ANOVA for response surface quadratic model (Water recovery %). Table 5 of Ahmad et al (2005).

source	sum of squares	DF	mean square	F value	Prob > F
block	27.648	1	27.648		
model	2311.01	9	256.779	3.504	0.0379
A	26.522	1	26.522	0.362	0.5623
B	406.022	1	406.022	5.540	0.0430
C	607.622	1	607.622	8.291	0.0182
A^2	51.334	1	51.334	0.700	0.4243
B^2	609.034	1	609.034	8.310	0.0181
C^2	405.904	1	405.904	5.538	0.0431
AB	156.645	1	156.645	2.137	0.1778
AC	0.845	1	0.845	0.012	0.9168
BC	6.845	1	6.845	0.098	0.7669
residual	659.59	9	73.288		
lack of fit	636.66	5	127.332	22.212	0.0051
pure error	22.93	4	5.732		
cor total	2998.25	19			
SD	8.56		R^2	0.7780	
mean	68.74		adj R^2	0.5559	
CV	12.45		pred R^2	-1.3271	
PRESS	6913.00		adeq precision	6.0877	

To obtain the factor settings that would minimize turbidity and maximize water recovery, the authors used an overlay plot by superimposing the contours for the various response surfaces. The optimal factors settings were as follows: coagulant dosage = 15,000 mg/L, flocculent dosage = 300 mg/L, pH = 6.0. The predicted responses were turbidity = 19 NTU and water recovery = 76%. The authors verified the results by performing three experiments at the optimal settings and obtained average values of 20 NTU for turbidity and 78% for water recovery. The authors could have used the straightforward desirability function approach for optimization available in Design-Expert.

Case Study #7.2

Bajpai, S., S. K. Gupta, Apurba Dey, M. K. Jha, Vidushi Bajpai, Saurabh Joshi, and Arvind Gupta (2012): Application of central composite design approach for removal of chromium (VI) from aqueous solution using weakly anionic resin: Modeling, optimization, and study of interactive variables. Journal of Hazardous Materials, 227-228, pp. 436-444.

This study applied response surface methodology using a rotatable central composite design to model and optimize the process parameters for the removal of Cr(VI) from aqueous streams using weakly anionic resin Amberlite IRA 96. Four process parameters were investigated – time of contact, initial solution pH, initial Cr(VI) concentration, and resin dose on Cr adsorption. The variables and levels used were shown in Table 1 of the paper and are reproduced here as Table 7.5. Table 7.5 has been modified slightly to be consistent with the order of variables used in the experimental design.

Table 7.5: Independent variables and levels. Modified Table 1 of Bajpai et al (2012).

Variables	Levels				
	-α	-1	0	1	+α
Treatment time (min)	0	30	60	90	120
pH	1	2.5	4	5.5	7
Cr(VI) concentration (mg/L)	50	162.5	275	387.5	500
Adsorbent dose (g/L)	0.2	2.65	5.1	7.55	10

The response variable was the percentage removal of Cr(VI). Design-Expert (version unknown) was used for the design and analysis of the experiment. A rotatable CCD with α = ±2 and six center points giving a total of 30 runs was used. The experimental design and results were shown in Table 2 of the paper and are reproduced here as Table 7.6. The experimental procedures and materials used were described in the paper.

The standard second-order response surface model with linear, two-factor interactions, and quadratic terms was fitted to the Cr(VI) recovery data. The ANOVA results for the full quadratic model were shown in Table 3 of the paper and are reproduced here as Table 7.7.

A quadratic regression model with all terms in actual values was selected as the prediction equation (Equation 8) in the paper and is given by:

$$\begin{aligned}\text{Removal\%} = {} & 44.21271 + 0.71240X_1 - 7.84705X_2 - 0.21915X_3 + 15.88276X_4 \\ & - 0.0130X_1X_2 + 1.17955X_1X_3 - 0.022634X_1X_4 + 0.00575924X_2X_3 \\ & - 1.09992X_2X_4 - 0.011127X_3X_4 - 0.00556975X_1^2 + 0.39154X_2^2 \\ & + 0.000236541X_3^4 - 0.22808X_4^2\end{aligned}$$

The above equation was incorrectly stated in the paper as in coded values. As can be seen in Table 7.7, nine out of 14 of the regression coefficients were actually not statistically significant at the 5% level. Furthermore, the lack of fit was statistically significant. This was not addressed in the paper. The predicted values from the regression model were also shown in Table 7.6.

Table 7.6: Independent variables and response of adsorption. Table 2 of Bajpai et al (2012).

Run	Time (min)	pH	Conc'n (mg/L)	Doses (g/L)	%removal of Cr(VI)	
	X_1	X_2	X_3	X_4	Actual	Predicted
1	60	4	500	5.1	44.98	52.45
2	90	5.5	387.5	2.65	36.97	37.10
3	60	1	275	5.1	91.56	84.26
4	90	2.5	162.5	2.65	49.46	52.18
5	90	2.5	387.5	7.55	77.98	78.65
6	90	5.5	162.5	2.65	34.76	32.76
7	60	4	275	5.1	51.98	52.18
8	120	4	275	5.1	52.86	44.18
9	60	4	275	5.1	48.64	52.18
10	60	4	275	10	61.67	69.92
11	60	4	275	5.1	49.99	56.35
12	60	4	275	5.1	46.96	52.18
13	90	2.5	387.5	2.65	55.78	56.80
14	60	4	275	5.1	52.56	52.18
15	90	5.5	387.5	7.55	42.78	42.77
16	30	2.5	162.5	7.55	95.98	88.52
17	90	5.5	162.5	7.55	58.86	50.70
18	60	4	275	0.2	20.34	23.48
19	30	5.5	387.5	7.55	40.64	27.25
20	60	7	275	5.1	8.45	17.14
21	30	5.5	387.5	2.65	30.86	14.92
22	30	2.5	162.5	2.65	51.56	47.76
23	60	4	50	5.1	78.56	75.85
24	30	5.5	162.5	2.65	30.55	26.50
25	60	4	275	5.1	56.86	52.18
26	30	2.5	387.5	2.65	32.98	33.53
27	90	2.5	162.5	7.55	97.45	105.35
28	30	5.5	162.5	7.55	54.56	49.15
29	0	4	275	5.1	0	3.07
30	30	2.5	387.5	7.55	62.87	60.78

The R^2 for the regression model was 0.88. No other goodness-of-fit statistics were given. Reanalysis of the data in Table 7.6 using Design-Expert 12 did not produce identical results to those of the paper. Only the corrected total sum squares was identical. However, the ANOVA results obtained using Design-Expert 12 were similar in magnitude to those shown in Table 7.7. Significant improvement in the predicted R^2 can be obtained if a reduced quadratic model with only statistically significant terms are used. However, the model still suffers from a significant lack of fit which needs to be addressed.

Table 7.7: ANOVA table for response surface quadratic model. Table 3 of Bajpai et al (2012).

Source	Sum of squares	df	Mean square	F value	p value > F
Model	13,080.82	14	934.34	7.70	0.0002
X_1 - time	862.69	1	862.69	7.11	0.0176
X_2 - pH	4715.57	1	4715.57	38.88	0.0001
X_3 - conc.	942.77	1	942.77	7.77	0.0138
X_4 - dose	3197.51	1	3197.51	26.36	0.0001
X_1X_2	5.38	1	5.38	0.04	0.0836
X_1X_3	284.21	1	284.21	2.34	0.1467
X_1X_4	41.89	1	41.89	0.35	0.5655
X_2X_3	18.84	1	18.84	0.16	0.6991
X_2X_4	257.11	1	257.11	2.12	0.1660
X_3X_4	170.65	1	170.65	1.41	0.2540
X_1^2	696.84	1	696.84	5.74	0.0300
X_2^2	21.52	1	21.52	0.18	0.6796
X_3^2	311.07	1	311.07	2.56	0.1301
X_4^2	51.98	1	51.98	0.43	0.5226
Residual	1819.49	15	121.3		
Lack of fit	1757.53	10	175.75	14.18	0.0046
Pure error	61.96	5	12.39		
Cor total	14,900.32	29			

The authors then used quadratic programming to determine the optimal conditions to maximize Cr(VI) removal using the developed regression model. The optimal conditions obtained using Amberlite IRA 96 resin in a batch process at temperature 30 °C were: contact time of 62.56 min, initial solution pH of 1.96, initial Cr(VI) concentration of 145.4 mg/L, and adsorbent dose of 8.51 g/L. With these conditions, the model predicted a 94.90% removal of Cr(VI). To validate the conditions obtained, an experiment with the optimal settings was conducted. The results were shown in Table 4 of the paper and are reproduced here as Table 7.8.

Table 7.8: Model validation. Table 4 of Bajpai et al (2012).

pH	Adsorbent dose (g/L)	Time (min)	Initial Cr(VI) conc'n (mg/L)	% removal	
				predicted	actual
1.96	8.51	62.5	145.4	94.99	93.26

More than one validation experiment would provide more confidence in the developed model. The authors could have used the desirability function approach for optimization that is available in Design-Expert instead of using quadratic programming.

Case Study #7.3

Eren, I. and Figen Kaymak-Erktekin (2007): Optimization of osmotic dehydration of potato using response surface methodology. Journal of Food Engineering, 79, pp. 344-352.

This study used response surface methodology with a rotatable central composite design to determine the optimum conditions to dehydrate potatoes by osmotic dehydration to maximize water loss and weight reduction, and minimize solid gain and water activity. Osmotic dehydration is gaining in popularity in food processing due to its energy efficiency and quality produced. Four factors were investigated – temperature, sucrose concentration, salt concentration, and processing time. The factors and levels used in the experiment are summarized in Table 7.9.

Table 7.9: Factors and levels used in potato dehydration experiment.

			Levels				
Variable	Name	Unit	-2	-1	0	1	2
x_1	Temperature	°C	20	30	40	50	60
x_2	Sucrose concentration	%	40	45	50	55	60
x_3	Salt concentration	%	0	3.75	7.5	11.25	15
x_4	Time	min	29.5	142	254.5	367	479.5

The materials and methods used in the osmotic dehydration process were described in the paper. The responses of interest were: water loss (WL, %), weight reduction (WR, %), solid gain (SG, %) and water activity (a_w). The definitions of the responses and how they were measured were given in the paper. The goal was to determine the optimal settings of the four process parameters (factors) to maximize WL and WR, while minimizing SG and a_w.

Design-Expert 6.01 was used in the design and analyses of the experiment, and also in subsequent optimization using the developed model.

A four-factor rotatable CCD with $\alpha = \pm 2$, and seven center points was used. The total number of runs was 31 (16 factorial points, 8 axial points, and 7 center points). The experimental matrix and results were shown in Table 1 of the paper and are reproduced here as Table 7.10.

A full standard second-order quadratic response surface model was then fitted to each of the responses. The full ANOVA table and associated regression coefficients were summarized in Table 2 of the paper and are reproduced here as Table 7.11 (typographical errors in the original table have been corrected in Table 7.11).

The full quadratic model provided a reasonably good fit to each of the responses and the lack of fit tests were all statistically insignificant at the 5% level. Reanalyses of the data in Table 7.10 gave similar results to those reported in Table 7.11 but not identical. Higher predicted R^2 values could be obtained by using reduced quadratic models with only statistically significant terms.

Table 7.10: Central composite rotatable design with experimental values of response variables. Table 1 of Eren et al (2007).

Run #	Temp (°C)	Sucrose conc. (%)	Salt Conc. (%)	Time (min)	WL (%)	SG (%)	WR (%)	a_w
1	30 (-1)	45	3.75	142	40	3.6	36.4	0.954
2	50 (+1)	45	3.75	142	46.9	4.5	42.5	0.931
3	30 (-1)	55	3.75	142	46.2	4	42.2	0.942
4	50 (+1)	55	3.75	142	54.6	5.5	49	0.919
5	30 (-1)	45	11.25	142	48.6	5	43.5	0.878
6	50 (+1)	45	11.25	142	56	6.6	49.4	0.855
7	30 (-1)	55	11.25	142	54.2	5.9	48.3	0.861
8	50 (+1)	55	11.25	142	60.5	7.1	53.4	0.828
9	30 (-1)	45	3.75	367	48.9	5.8	43.1	0.929
10	50 (+1)	45	3.75	367	52	7.4	44.6	0.919
11	30 (-1)	55	3.75	367	55.9	6.5	49.5	0.911
12	50 (+1)	55	3.75	367	60.5	8	52.5	0.896
13	30 (-1)	45	11.25	367	56.9	7	49.8	0.849
14	50 (+1)	45	11.25	367	58.6	8.2	50.4	0.838
15	30 (-1)	55	11.25	367	61.4	7.4	54	0.816
16	50 (+1)	55	11.25	367	64.8	8.6	56.2	0.798
17	20 (-2)	50	7.5	254.5	54.6	4.3	50.3	0.897
18	60 (+2)	50	7.5	254.5	62.2	7.6	54.6	0.864
19	40 (0)	40	7.5	254.5	50.3	7	43.3	0.891
20	40 (0)	60	7.5	254.5	62.8	8.1	54.7	0.846
21	40 (0)	50	0	254.5	43.4	5.1	38.3	0.957
22	40 (0)	50	15	254.5	61.9	6.7	55.1	0.778
23	40 (0)	50	7.5	29.5	40.9	3.7	37.2	0.941
24	40 (0)	50	7.5	479.5	60.6	9.3	51.3	0.871
25	40 (0)	50	7.5	254.5	60.5	7	53.4	0.878
26	40 (0)	50	7.5	254.5	60.5	6.5	53.9	0.869
27	40 (0)	50	7.5	254.5	61.5	7.1	54.4	0.874
28	40 (0)	50	7.5	254.5	60.6	6.8	53.7	0.876
29	40 (0)	50	7.5	254.5	61.9	6.4	55.5	0.878
30	40 (0)	50	7.5	254.5	59.1	6.4	52.8	0.88
31	40 (0)	50	7.5	254.5	63.7	6.8	56.9	0.881

The authors then used the desirability function approach available in Design-Expert to determine the optimum conditions for osmotic dehydration of potatoes. The goals were to maximize water loss and weight reduction and minimize solid gain and water activity. The optimal conditions were found to be: temperature of 22 °C, sucrose concentration of 54.5%, salt concentration of 14%, and time of 329 min, obtaining a water loss of 59.1 (g/100g fresh sample), weight reduction of 52.9 (g/100g fresh sample), solid gain of 6.0 (g/100g fresh sample), and water activity of 0.785. No further experiments were conducted to validate the optimum conditions obtained.

Table 7.11: ANOVA table showing the variables as linear, quadratic and interaction terms on each response variable and coefficients for the prediction models. Corrected Table 2 of Eren et al (2007).

Source	DF	Water Loss (WL)			Solid gain (SG)			Weight reduction (WR)			Water activity (a_w)		
		Coefficient	Sum of squares	p-value	Coefficient	Sum of squares	p-value	Coefficient	Sum of squares	p-value	Coefficient	Sum of squares	p-value
Intercept/Model	14	61.110	1431.26	<0.0001	6.729	59.90	<0.0001	54.380	1012.82	<0.0001	0.877	0.058	<0.0001
x_1	1	2.373	135.14	<0.0001	0.723	12.54	<0.0001	1.650	65.34	<0.0001	-0.009	0.0021	<0.0001
x_2	1	3.139	236.45	<0.0001	0.296	2.10	0.0008	2.843	193.95	<0.0001	-0.011	0.0031	<0.0001
x_3	1	3.870	359.35	<0.0001	0.578	8.01	<0.0001	3.292	260.07	<0.0001	-0.043	0.045	<0.0001
x_4	1	3.811	348.63	<0.0001	1.160	32.30	<0.0001	2.651	168.70	<0.0001	-0.015	0.0052	<0.0001
x_1^2	1	-0.753	16.22	0.0205	-0.220	1.38	0.0041	-0.533	8.14	0.0792	0.001	5E-05	0.1108
x_2^2	1	-1.211	41.97	0.0008	0.187	1.00	0.0117	-1.398	55.91	0.0001	-0.002	8E-05	0.0518
x_3^2	1	-2.190	137.21	<0.0001	-0.227	1.47	0.0032	-1.964	110.26	<0.0001	-0.002	0.0001	0.0280
x_4^2	1	-2.666	203.26	<0.0001	-0.078	0.17	0.2518	-2.588	191.54	<0.0001	0.008	0.0017	<0.0001
x_1x_2	1	0.215	0.74	0.5898	0.003	0.00	0.9765	0.213	0.72	0.5796	-0.001	3E-05	0.2123
x_1x_3	1	-0.245	0.96	0.5409	-0.032	0.02	0.7189	-0.212	0.72	0.5800	-0.001	1E-05	0.4206
x_1x_4	1	-1.005	16.16	0.0207	0.028	0.01	0.7505	-1.033	17.09	0.0143	0.003	0.0001	0.0120
x_2x_3	1	-0.520	4.39	0.1996	-0.034	0.02	0.7065	-0.490	3.84	0.2111	-0.003	0.0002	0.0073
x_2x_4	1	0.140	0.32	0.7241	-0.049	0.04	0.5812	0.190	0.58	0.6204	-0.003	0.0001	0.0153
x_3x_4	1	-0.440	3.07	0.2796	-0.202	0.65	0.0349	-0.236	0.89	0.5393	-0.002	7E-05	0.0955
Residual	16		39.23			1.97			36.24			0.0003	
Lack of fit	10		26.75	0.3944		1.49	0.2295		24.69	0.3952		0.0002	0.4624
Pure error	6		12.48			0.48			11.54			0.0001	
Total	30		1470.49			61.87			1049.06			0.058	
R^2		0.9733			0.9682			0.9655			0.995		
Adj-R^2		0.9500			0.9403			0.9352			0.9907		
Pred-R^2		0.8837			0.8507			0.8494			0.9791		
PRESS		171.07			9.23			157.95			0.0012		
CV		2.80			5.42			3.05			0.48		

Case Study #7.4

Lakshminarayanan, A. K., V. E. Annamalai, and K. Elangovan (2015): Identification of optimum friction stir spot welding process parameters controlling the properties of low carbon automotive steel joints. Journal of Materials Research and Technology, 4 (3), pp. 262-272.

This study applied response surface methodology using a rotatable central composite design to optimize the process parameters for friction stir spot welding of low carbon automotive steel joints. The goal was to develop a model for predicting the lap shear tensile strength of the welded steel joints and to determine the optimum settings of the process parameters to maximize strength. Three process parameters – rotational speed, plunge depth, and dwell time were investigated. The parameters and levels used in the experiment were shown in Table 3 of the paper and are reproduced here as Table 7.12. The details of the experimental work and materials used were described in the paper. The response of interest was the tensile shear failure load (TSFL) in kN.

7.12: Feasible limits of the process parameters and their levels. Table 3 of Lakshminarayanan et al (2015).

Parameters	Notation	Units	Levels				
			-1.682	-1	0	1	1.682
Rotational speed	N	rpm	1200	1281	1400	1519	1600
Plunge depth	P	mm	0	0.04	0.1	0.16	0.2
Dwell time	D	s	5	9	15	21	25

Design-Expert Version 8 was used for the design and analysis of the experiment and subsequent optimization process to determine the best combination of parameters to maximize TSFL. A three-factor rotatable CCD with $\alpha = \pm 1.682$ and six center points) was used. The total number of runs was 20 (8 factorial points, 6 axial points, and 6 center points. The design matrix and experimental results were shown in Table 4 of the paper and are reproduced here as Table 7.13.

Table 7.13: Design matrix and experimental results. Table 4 of Lakshminarayanan et al (2015).

Exp. No.	Coded value			Original value			TSFL (kN)
	N	P	D	N	P	D	
1	-1	-1	-1	1281	0.04	9	5.60
2	1	-1	-1	1519	0.04	9	9.50
3	-1	1	-1	1281	0.16	9	12.70
4	1	1	-1	1519	0.16	9	10.40
5	-1	-1	1	1281	0.04	21	10.30
6	1	-1	1	1519	0.04	21	15.50
7	-1	1	1	1281	0.16	21	12.10
8	1	1	1	1519	0.16	21	11.40
9	-1.682	0	0	1200	0.1	15	10.40
10	1.682	0	0	1600	0.1	15	12.80
11	0	-1.682	0	1400	0	15	9.90
12	0	1.682	0	1400	0.2	15	12.10
13	0	0	-1.682	1400	0.1	5	8.50
14	0	0	1.682	1400	0.1	25	13.00
15	0	0	0	1400	0.1	15	13.60
16	0	0	0	1400	0.1	15	13.40
17	0	0	0	1400	0.1	15	13.30
18	0	0	0	1400	0.1	15	13.50
19	0	0	0	1400	0.1	15	13.10
20	0	0	0	1400	0.1	15	13.40

A full standard second-order quadratic response surface model was then fitted to each of the responses. The ANOVA results were shown in Table 5 of the paper and are reproduced here as Table 7.14. All terms of the full quadratic model were all statistically significant at the 5% level, and the lack of fit term was not statistically significant. No goodness of fit statistics were reported in the paper.

Table 7.14: ANOVA for tensile shear failure load. Table 5 of Lakshminarayanan et al (2015).

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	96.56494	9	10.72944	557.2028	< 0.0001
N	7.52330	1	7.52330	390.7012	< 0.0001
P	6.46993	1	6.46993	335.9976	< 0.0001
D	25.51808	1	25.51808	1325.209	< 0.0001
NP	18.30125	1	18.30125	950.4233	< 0.0001
ND	1.05125	1	1.05125	54.59367	< 0.0001
PD	13.26125	1	13.26125	688.6852	< 0.0001
N^2	5.88990	1	5.88990	305.8754	< 0.0001
P^2	10.44720	1	10.44720	542.5454	< 0.0001
D^2	12.72887	1	12.72887	661.0375	< 0.0001
Residual	0.19256	10	0.019256		
Lack of Fit	0.04426	5	0.008845	0.29815	0.8949
Pure Error	0.14833	5	0.029667		
Cor Total	96.75750	19			

The full second-order quadratic regression model (in coded values) was used as the prediction model (Equation 4):

$$\text{Tensile shear failure load (TSFL, kN)} = 13.38 + 0.74N + 0.69P + 1.37D - 1.51NP + 0.36ND - 1.29PD - 0.64N^2 - 0.85P^2 - 0.94D^2$$

Reanalyses of the data in Table 7.13 using Design-Expert 12 gave very similar results to those reported in Table 7.14 but not identical. The R^2, adjusted R^2, and predicted R^2 obtained are 0.9981, 0.9964, and 0.9950, respectively.

The authors then used the prediction model to obtain the optimal factor settings using the desirability function approach available in Design-Expert. Three possible solutions were given by the software based on the desirability values. These were shown in Table 7 of the paper and reproduced here as Table 7.15.

Table 7.15: Confirmation experiments. Table 7 of Lakshminarayanan et al (2015).

Exp. No.	N (rpm)	P (mm)	D (s)		Tensile shear failure load (kN)
1	1530	0.05	21.3	Actual	14.97
	(1.09)	(-0.97)	(1.07)	Predicted	15.72
				Error %	4.96
2	1595	0.05	22.8	Actual	15.5
	(1.64)	(-0.95)	(1.32)	Predicted	16.38
				Error %	5.37
3	1591	0.05	19.64	Actual	15.73
	(1.61)	(-0.96)	(0.78)	Predicted	14.98
				Error %	4.77

Values given in braclets are the corresponding coded values.
Error% = (measured value - predicted value)/predicted value.

Three confirmation experiments were conducted using the optimal combinations shown in the table to validate the model. The predicted and observed values are also shown in Table 7.15. The percentage errors were around 5%.

The authors concluded that a maximum tensile shear fracture load 15.67 kN could be attained using a tool rotation speed of 1157 rpm, plunge depth of 0.05 mm, and dwell time of 22 s. It is not clear how these values were obtained as they were not part of the optimal combinations shown in Table 7.15.

Case Study #7.5

Mu, Y., Xian-Jun Zheng, and Han-Qing Yu (2009): Determining optimum conditions for hydrogen production from glucose by an anaerobic culture using response surface methodology (RSM). International Journal of Hydrogen Energy, 34, pp. 7959-7963.

This study applied response surface methodology using a rotatable central composite design to optimize the production of H_2 from glucose by an anaerobic culture. Three factors that influenced the hydrogen production process – temperature, pH, and glucose concentration were investigated. The factors and levels used are summarized in Table 7.16. The experimental methods and materials used in the experiment were described in the paper.

Table 7.16: Factors and levels used in the hydrogen production experiment.

Factor	Units	Levels				
		-1.68	-1	0	1	1.68
Temperature	°C	27.4	32.5	40	47.5	52.6
pH		4.24	4.75	5.5	6.25	6.76
Glucose concentration	g/L	5.8	7.5	10	12.5	14.2

The response of interest was the H_2 yield (mol-H_2/mol-glucose). The primary goal was to determine the optimal combinations of the three factors to maximize H_2 yield.

A three-factor rotatable CCD with $\alpha = \pm 1.68$ and six center points was used. The total number of runs was 20 (8 factorial points, 6 axial points, and 6 center points.) Minitab (unknown version) was used for the design and analysis of the experiment. The design matrix in coded and real values, and H_2 yield results were shown in Table 1 of the paper and are reproduced here as Table 7.17.

A full standard second-order quadratic response surface model was fitted to the response and the estimated regression coefficients and associated statistics were shown in Table 2 of the paper and are reproduced here as Table 7.18. Six of the coefficients were not statistically significant at the 5% level. However, the authors seem to have used the full model in the optimization phase.

The summarized ANOVA table for the full model was shown in Table 3 of the paper and is reproduced here as Table 7.19. The only goodness of fit statistic was the R^2 value of 0.927. There was no mention of regression diagnostics in the paper.

Table 7.17: A 20 full factorial CCD with six replicates of the centre point for H_2 yield. Table 1 of Mu et al (2009).

Run	Coded values			Real values			H_2 yield
	x_1	x_2	x_3	X_1	X_2	X_3	(mol-H_2/mol-glucose)
1	1	1	1	47.5	6.25	12.5	0.75
2	1	1	-1	47.5	6.25	7.5	0.89
3	1	-1	1	47.5	4.75	12.5	0.44
4	1	-1	-1	47.5	4.75	7.5	0.79
5	-1	1	1	32.5	6.25	7.5	0.97
6	-1	1	-1	32.5	6.25	12.5	1.07
7	-1	-1	1	32.5	4.75	12.5	0.42
8	-1	-1	-1	32.5	4.75	7.5	0.47
9	1.68	0	0	52.6	5.50	10.0	0.31
10	-1.68	0	0	27.4	5.50	10.0	1.17
11	0	1.68	0	40.0	6.76	10.0	1.00
12	0	1.68	0	40.0	4.24	10.0	0.11
13	0	0	1.68	40.0	5.50	14.2	1.76
14	0	0	-1.68	40.0	5.50	5.8	1.78
15	0	0	0	40.0	5.50	10.0	1.69
16	0	0	0	40.0	5.50	10.0	1.78
17	0	0	0	40.0	5.50	10.0	1.72
18	0	0	0	40.0	5.50	10.0	1.61
19	0	0	0	40.0	5.50	10.0	1.66
20	0	0	0	40.0	5.50	10.0	1.73

x_i is the coded value of the ith test variable.

X_i is the real value of the ith test variable.

Table 7.18: Estimated regression coefficients and corresponding standard deviation, t_{exp} and significance level for H_2 yield. Table 2 of Mu et al (2009).

Coefficient	Value	Standard deviation	t_{exp}	Significance level (%)
A_0	1.71	0.09	19.62	<0.01
A_1	-0.11	0.06	-1.91	8.5
A_2	0.22	0.06	3.88	0.3
A_3	-0.03	0.06	-0.60	56.1
A_{11}	-0.39	0.06	-6.99	<0.01
A_{22}	-0.46	0.06	-8.16	<0.01
A_{33}	-0.03	0.06	-0.51	62.2
A_{12}	-0.09	0.08	-1.23	24.8
A_{13}	-0.07	0.08	-0.90	39.2
A_{23}	0.05	0.08	0.60	56.4

t_{exp} was obtained from the t-test which indicates the significance of the regression coefficients.

Table 7.19: ANOVA analysis. Table 3 of Mu et al (2009).

Source	Degree of freedom	Sum of square	Mean square	F-value	P-value
Regression	9	5.8025	0.6447	14.18	0
Residual	10	0.4547	0.0455		
Total	19	6.2573			

$R^2 = 0.927$

The Fisher variance ratio (F-value) is the ratio of the mean square due to regression, divided by the mean square due to residual.

Reanalysis of the data in Table 7.17 using Design-Expert 12 gave almost identical results to those reported in the paper. However, the lack of fit was statistically significant (p-value = 0.0015), and Run #10 was identified as an outlier. The predicted R^2 was also quite low (0.4667). Furthermore if a reduced model with only statistically significant terms was used, the predicted R^2 would have increased by over 20%.

It is not clear exactly how the authors obtained the optimum conditions of a temperature of 38.8 °C, pH of 5.7, and glucose concentration of 9.7 g/L giving a maximum H_2 yield of 1.75 mol-H_2/mol-glucose. No additional experiments were conducted to confirm the model and optimum conditions obtained.

Case Study #7.6

Prasad, K. N., Fouad Abdulrahman Hassan, Bao Yang, Kin Weng Kong, Ramakrishnan Nagasundara Ramanan, Azrina Azlan, and Amin Ismail (2011): Response surface optimization for the extraction of phenolic compounds and antioxidant capacities of underutilised *Mangifera pajang* Kosterm peels. Food Chemistry, 1238, pp. 1121-1127.

This study used response surface methodology with a rotatable central composite design to optimize the conditions for the maximum recovery of total phenolics content (TPC) and antioxidant capacities (AC) of *Mangifera pajang* peels (MPP). *Mangifera pajang* is an underutilized fruit from East Malaysia, and is commonly referred to as brown mango or bambangan in the Malay language. Three factors – ethanol concentration, extraction temperature, and liquid-to-solid ratio were investigated. The factors and levels used were shown in Table 1 of the paper and are reproduced here as Table 7.20.

The primary objective of the study was to maximize the extraction of total phenolic content (TPC) and the antioxidant capacity (AC) from the *Mangifera pajang* peels (MPP). The materials and methods used were described in the paper.

Table 7.20: Independent variables and their coded and actual values used for optimization. Table 1 of Prasad et al (2011).

Independent variable	Units	Symbol	Coded levels				
			-1	0	1	Axial (-α)	Axial (+α)
Ethanol concentration	%	X_1	32	50	68	20	80
Temperature	°C	X_2	37	48	58	30	65
Liquid/solid ratio	mL/g	X_3	201	31.3	42.4	12.5	50

Design-Expert 6.0 software was used for the experimental design and statistical analysis of the responses. A three-factor rotatable CCD with $\alpha = \pm 1.68$ and six center points was used. The total number of experimental runs was 20 consisting of eight factorial, six axial, and six center points. The experimental design and results were shown in Table 2A of the paper and are reproduced here as Table 7.21.

Table 7.21: Three-factor central composite design used for RSM with experimental and predicted values for the independent variables. Table 2A of Prasad et al (2011).

Standard order	Factor 1 (X_1) Ethanol concentration (%)	Factor 2 (X_2) Temperature (°C)	Factor 3 (X_3) Liquid/soild ratio (mL/g)	Response 1 (Y_1) TPC (mg GAE/g)		Response 2 (Y_2) AC (absorbance in nm)	
				Experimental	Predicted	Experimental	Predicted
1	-1	-1	-1	6.39	6.04	0.165	0.166
2	1	-1	-1	7.19	7.36	0.186	0.188
3	-1	1	-1	6.41	6.51	0.135	0.135
4	1	1	-1	12.30	b	0.195	b
5	-1	-1	1	8.49	8.56	0.187	0.187
6	1	-1	1	10.20	9.88	0.183	0.183
7	-1	1	1	10.70	10.6	0.167	0.166
8	1	1	1	14.20	14.4	0.201	0.2
9a	0	0	0	13.70	13.5	0.203	0.203
10a	0	0	0	13.20	13.5	0.201	0.203
11a	0	0	0	13.40	13.5	0.206	0.203
12a	0	0	0	13.50	13.5	0.201	0.203
13	-1.68	0	0	5.97	6.11	0.142	0.143
14	1.68	0	0	10.40	10.4	0.191	0.19
15	0	-1.68	0	6.84	7.05	0.197	0.195
16	0	1.68	0	11.30	11.2	0.181	0.183
17	0	0	-1.68	6.01	6.02	0.166	0.165
18	0	0	1.68	11.50	11.6	0.185	0.187
19a	0	0	0	12.90	12.8	0.204	0.202
20a	0	0	0	13.00	12.8	0.201	0.202

a - Centre point
b - Outlier.

Note that in Table 7.21, the first column heading should have been "Run #" as the order of runs shown was not quite in standard order.

A full standard second-order quadratic response surface model was fitted to the responses but no ANOVA results were shown in the paper. However, the estimated regression coefficients of the polynomial models and some associated statistics were shown in Table 2B of the paper and are reproduced here as Table 7.22. The predicted values of the two responses using the equations shown in Table 7.22 were also shown in Table 7.21.

In Table 7.21, run #4 was identified as an outlier and no predicted values were given. Upon a closer examination of the predicted values, all center point values should have been identical, but this was not the case in Table 7.21.

Reanalysis of the data in Table 7.21 using Design-Expert 12 gave almost identical results to those reported for the second response AC (Y_2) except that the lack of fit "p-value" was 0.3266 instead of the reported 0.4764. All regression terms were statistically significant at the 5% level. However, for the response TPC (Y_1), the regression coefficients reported seemed to be off by several orders of magnitude and the lack of fit was statistically significant at the 5% level (p-value = 0.011) and not 0.1870 as reported. Furthermore, the interaction term X_2X_3 was also not statistically significant at the 5% level and should not have beeen in the model. Run #4 would not be an outlier if a reduced quadratic model with only statistically significant terms were used. Hence, the results reported in Table 7.22 were mostly incorrect likely because of typographical errors, especially for the magnitudes of the regression coefficients for TPC.

Table 7.22: Polynomial equation and statistical parameters calculated after implementation of 2nd full factorial central composite experimental design. Corrected Table 2B of Prasad et al (2011).

Response	2nd order polynomial equation	R^2	R^2 (adjusted)	Regression (p value)	Lack of fit
TPC (Y_1)	$13158.69 + 1263.38X_1 + 1235.93X_2 + 1657.63X_3 - 1615.12X_1^2 - 1298.73X_2^2 - 1409.43X_3^2 + 605.41X_1X_2 + 397.40X_2X_3$	0.9969	0.9936	0.0001	0.1870
AC (Y_2)	$0.20 + 0.014X_1 - 0.00369X_2 + 0.00651X_3 - 0.012X_1^2 - 0.00441X_2^2 - 0.00922X_3^2 - 0.00937X_1X_2 - 0.00626X_1X_3 + 0.00251X_2X_3$	0.9953	0.9900	0.0001	0.4764

Column 1 of Table 2B was incorrectly labelled as "Regression coefficient".

The authors used a combination of graphical and numerical optimization methods to determine the optimal levels of the independent variables to maximize TPC and AC. For the response TPC, the optimum values were ethanol concentration of 68%, temperature of 55 °C, and liquid/solid ratio of 32.7 mL/g. For the response AC, the optimum values were ethanol concentration of 68%, temperature of 56 °C, and liquid/solid ratio of 31.8 mL/g. The predicted responses with these settings were 14.6 mg GAE/g and 0.2065, respectively.

The response surface models were verified by conducting additional experiments at the optimum values of the independent variables and the TPC and AC obtained experimentally were 14.6 ± 0.0026 mg GAE/g and 0.2035 ± 0, respectively.

No reasons were given as to why the optimization was not set to maximize both TPC and AC simultaneously instead of individually, as was done in this study.

Case Study #7.7

Rastegar, S. O., S. M. Mousavi, S. A. Shojaosadati, and S. Sheibani (2011): Optimization of petroleum refinery effluent treatment in a UASB reactor using response surface methodology. Journal of Hazardous Materials, 197, pp. 26-32.

This study applied response surface methodology using a rotatable central composite design to optimize the treatment of petroleum effluent in a laboratory scale upflow anaerobic sludge blanket (UASB) bioreactor. The UASB bioreactor is a popular wastewater treatment used around the world for several decades. The primary goal of this study was to investigate the phenomenon of removal of petroleum refinery effluent in an UASB bioreactor. The schematic diagram of the UASB bioreactor was given in the paper and the independent variables that were adjustable on the bioreactor were the hydraulic retention time (HRT), upflow velocity (V_{up}), and the influent chemical oxygen demand (COD). The experimental ranges and levels of the three variables were shown in Table 2 of the paper and are reproduced here with corrections as Table 7.23.

Table 7.23: Experimental range and levels of independent test variables. Corrected Table 2 of Rastegar et al (2011).

Variable	Low axial ($-\alpha$)	Low factorial (-1)	Center point (0)	High factorial (+1)	High axial ($+\alpha$)
HRT (h): A	10	12	17.5	22	25
V_{up} (m/h): B	0.1	0.18	0.3	0.42	0.5
Influent COD (mg/L): C	500	642	850	1060	1200

In Table 7.23, the variables are labelled to match the layout of the experimental design shown in Table 3 of the paper. Hence, HRT is relabelled as A instead of B, influent COD is relabelled as C instead of A, and V_{up} is relabelled as B instead of C.

Two responses were of interest – COD removal (%) and the biogas production rate (L biogas/L feed d). Design-Expert 7.0 software was used for experimental design and statistical analysis. A three-factor rotatable CCD with $\alpha = \pm 1.68$ and six center points was used. The total number of experimental runs was 20 consisting of eight factorial, six axial, and six center points. The experimental design and results were shown in Table 3 of the paper and are reproduced here as Table 7.24.

A reduced quadratic response surface model was fitted to the COD removal data and a reduced cubic model was fitted to the biogas rate data. The partial ANOVA results for the two responses were shown in Table 4 of the paper and are reproduced here as Table 7.25. Only the R^2 and adjusted R^2 values were shown.

For COD removal (%), the regression model was given as (Equation 2 in the paper):

$$\text{COD removal (\%)} = 68.06 + 6.5A + 3.29B - 2.65C - 0.63AB + 0.87AC - 0.16A^2$$

Note that from Table 7.25, the terms AB, AC, and A^2 were not statistically significant at the 5% level. Hence a linear model with only A, B, and C would be more correct.

Table 7.24: Experimental plan and results. Table 3 of Rastegar et al (2011).

Run	HRT (h)	V_{up} (m/h)	Influent COD (mg/L)	COD removal (%)	Biogas rate (L biogas/L feed d)
1	22	0.42	642	78	0.18
2	22	0.18	642	72	0.14
3	17.5	0.3	851	68	0.15
4	17.5	0.1	851	61	0.1
5	17.5	0.5	851	74	0.44
6	13	0.42	642	68	0.17
7	10.0	0.3	851	55	0.21
8	22	0.42	1060	73	0.38
9	25	0.3	851	81	0.48
10	17.5	0.3	851	67	0.14
11	13	0.18	1060	54	0.52
12	17.5	0.3	851	69	0.14
13	17.5	0.3	851	68	0.14
14	17.5	0.3	1200	64	0.64
15	17.5	0.3	851	69	0.13
16	22	0.18	1060	70	0.33
17	13	0.18	642	63	0.13
18	17.5	0.3	500	73	0.07
19	17.5	0.3	851	69	0.13
20	13	0.42	1060	63	0.39

For biogas production rate, the regression model was given as (Equation 3 in the paper):

$$\text{Biogas production rate (L biogas/L feed d)} = 0.14 + 0.08A + 0.10B + 0.14C - 0.027AC - 0.02BC + 0.062A^2 + 0.065C^2 + 0.035B^2 - 0.10A^2B - 0.1AB^2$$

Note that from Table 7.25, the terms AC and BC were not statistically significant at the 5% level. Also, AB should be included for hierarchy.

Reanalyses of the data using Design-Expert 12 showed that both suggested models had statistically significant lack of fit which was not reported. Furthermore, for COD removal, if only linear terms were used, the predicted R^2 would increase from 0.858 to 0.918.

For biogas production, the reported model actually had a predicted R^2 of -0.0198. A negative predicted R^2 indicates that the model is no better than using the overall mean value. In addition, some assumptions of regression were violated. The residuals were not normally distributed and they were not homoscedastic. A logarithmic transformation of the response would provide a much better fit and would not violate any regression assumptions. However, the lack of fit would still be statistically significant.

Table 7.25: ANOVA for response surface models applied. Table 4 of Rastegar et al (2011).

Response	Model	ANOVA					
		Source	Sum of square	DF	Mean square	F value	Prob > F
COD removal (%)	Reduced quadratic model	Model	829.05	6	138.18	60.08	< 0.0001
		A	576.44	1	576.44	250.66	< 0.0001
		B	147.38	1	147.38	64.09	< 0.0001
		C	95.62	1	95.62	41.58	< 0.0001
		A^2	0.3659	1	0.37	0.1591	0.6965
		AC	6.13	1	6.13	2.66	0.1267
		AB	3.13	1	3.13	1.36	0.2647
		Residual	29.90	13	2.30		
		(R^2=0.96, R^2_{adj} = 0.94)					
Biogas production rate (L biogas/L feed d)	Reduced cubic model	Model	0.502	10	0.050	18.48	< 0.0001
		A-HRT (h)	0.036	1	0.036	13.42	0.0052
		B-Vup (m/h)	0.058	1	0.058	21.29	0.0013
		C-Influent COD	0.281	1	0.28	103.44	< 0.0001
		AC	0.006	1	0.006	2.23	0.1697
		BC	0.003	1	0.003	1.18	0.3000
		A^2	0.055	1	0.055	20.11	0.0015
		C^2	0.061	1	0.061	22.48	0.0011
		B^2	0.018	1	0.018	6.52	0.0311
		A^2B	0.034	1	0.034	12.47	0.0064
		AB^2	0.035	1	0.035	12.89	0.0058
		Residual	0.024	9	0.0025		
		(R^2=0.95, R^2_{adj} = 0.90)					

In the process optimization phase, the authors used graphical optimization to obtain the optimal variable settings for a COD removal of 75% and biogas production rate of 0.20 L biogas/L feed d. Reasons were given in the paper as to why these values for the responses were chosen. Two experimental combinations were used to validate the models. The results of the validation experiments were shown in Table 5 of the paper and are reproduced here as Table 7.26.

Table 7.26: Optimum condition verification and additional experiments. Table 5 of Rastegar et al (2011).

Influent COD (mg/L)	HRT (h)	Vup (m/h)	COD removal (%) (model)	COD removal (%) (experiment)	Biogas rate (L/L feed d) (model)	Biogas rate (L/L feed d) (experiment)
630	21.4	0.27	75.0	76.3	0.2	0.25
620	21.4	0.27	74.9	78.3	0.2	0.24

The authors could have used the desirability function approach available in Design-Expert to obtain the optimal combinations. Many other possible solutions are available to achieve the same objective.

Case Study #7.8

Singal, R., Prateek Seth, Dinesh Bangwal, and Savita Kaul (2012): Optimization of biodiesel production by response surface methodology and genetic algorithm. Journal of ASTM International, Vol. 9, NO. 5, pp. 1-7.

This study applied response surface methodology using a rotatable central composite design to investigate the effect of three parameters – reaction temperature, catalyst concentration, and molar ratio of methanol to oil on biodiesel production from alkali-catalyzed transesterification of keranja oil. Karanja is a non-edible plant commonly available in India. The parameters or factors and their levels used in the experiment are shown in Table 7.27.

Table 7.27: Factors and their coded and actual values used in the biodiesel experiment.

Factors	Units	Symbol	Coded levels				
			-1	0	1	Axial (-α)	Axial (+α)
Temperature	°C	A	45	55	65	38.18	71.82
Catalyst concentration	wt. %	B	1.00	1.25	1.50	0.83	1.67
Molar ratio	MeOH:oil	C	5	6	7	4.318	7.682

The response variable was the biodiesel yield defined as the weight of biodiesel obtained per unit weight of pretreated oil taken. The main objective was to determine the optimal factor settings to maximize biodiesel yield. The materials and methods used were described in the paper.

The experimental design was a three-factor rotatable CCD with $\alpha = \pm 1.68$ and six center points. The total number of experimental runs was 20 consisting of eight factorial, six axial, and six center points. The experimental design and results were shown in Table 1 of the paper and are reproduced here as Table 7.28. The software used for the experimental design and statistical analysis was not mentioned in the paper.

A full standard second-order quadratic response surface model was fitted to the response. The summarized ANOVA results were shown in Table 2 of the paper and are reproduced here as Table 7.29. The full second-order regression model for biodiesel yield was given as (Equation 1 of the paper):

$$\text{Biodiesel yield, } Y = 128.498 - 0.420A - 60.693B + 1.573C - 0.007A^2 + 13.98B^2 - 1.17C^2 + 0.076AB + 0.217AC + 2.31BC \qquad (R^2 = 0.9148)$$

No other goodness of fit statistics were reported besides the R^2 value of 0.9148. The p-value of individual regression term was also not reported in the paper.

Reanalyses of the data using Design-Expert 12 showed that overall ANOVA results were correct but the terms AB, BC, A^2 and B^2 were not statistically significant at the 5% level. If a reduced model with only statistically significant terms were used, the predicted R^2 would increase from about 0.40 to 0.68.

Table 7.28: Central composite design matrix. Table 1 of Singhal et al (2012).

Exp. No.	Temperature (A)	Catalyst conc. (B)	Molar ratio (C)	Yield (Y)
1	45	1	5	91.06
2	65	1	5	92.23
3	45	1.5	5	87.3
4	65	1.5	5	85.3
5	45	1	7	91.48
6	65	1	7	97.4
7	45	1.5	7	86.1
8	65	1.5	7	96.71
9	38.18	1.25	6	85.16
10	71.82	1.25	6	91.5
11	55	0.83	6	95.62
12	55	1.67	6	90
13	55	1.25	4.318	82.18
14	55	1.25	7.682	91.9
15	55	1.25	6	92
16	55	1.25	6	92.69
17	55	1.25	6	91.5
18	55	1.25	6	90.93
19	55	1.25	6	90.38
20	55	1.25	6	90.56

Table 7.29: ANOVA results for the regression model. Table 2 of Singhal et al (2012).

Source	Degree of freedom (df)	Sum of squares (SS)	Mean sum of squares (MS)	F	p
Regression	9	258.2514	28.6946	11.9357	0.0003
Residual	10	24.0410	2.4041		
Total	19	282.2924			

Although not mentioned explicitly in the paper, optimization of biodiesel yield was likely obtained using the desirability function approach with the constraints shown in Table 3 of the paper and reproduced here as Table 7.30.

Table 7.30: Constraints used to optimize biodiesel yield. Table 3 of Singhal et al (2012).

Process variable	Goal	Lower limit	Upper limit
A: Reaction temperature (°C)	In range	45	65
B: Catalyst concentration (wt. %)	In range	1.0	1.5
C: Molar ratio (MeOH:oil)	In range	5	7

The maximum biodiesel yield obtained was 97.86% at a temperature of 64.60 °C, catalyst concentration of 1.0 wt %, and a molar ratio of methanol to oil of 6.996:1. Optimization using the genetic algorithm approach apparently gave similar results.

Case Study #7.9

Wang, J.P., Yong-Zhen Chen, Xue-Wu Ge, and Han-Qing Yu (2007): Optimization of coagulation-flocculation process for a paper-recycling wastewater treatment using response surface methodology. Colloids and Surfaces A: Physicochemical Engineering Aspects, 302, pp. 204-210.

This study used response surface methodology via a near-rotatable central composite design (similar to Case Study 7.1) to investigate a coagulation-flocculation process to treat paper-recycling wastewater. The objective was to minimize turbidity and the sludge volume index. Aluminum chloride was used as the coagulant and a modified natural polymer, chitosan-g-PDMC (poly(2-methacryloyloxyethyl) trimethyl ammonium chloride) was used as the flocculant. The three factors and levels used in the experiment were shown in Table 1 of the paper and are reproduced here as Table 7.31.

Table 7.31: Levels of the variable tested in the 2^3 central composite design. Table 1 of Wang et al (2007).

Variables	Range and levels				
	-2	-1	0	1	2
X_1, coagulant dosage (mg/l)	0	500	1000	1500	2000
X_2, flocculant dosage (mg/l)	0	8	16	24	32
X_3, pH	2	4	6	8	10

The two responses of interest were the turbidity (NTU) and sludge volume index (SVI). MATLAB Version 6.5 was used for the experimental design and statistical analysis. The experimental design was a three-factor near rotatable CCD with $\alpha = \pm 2.0$ and six center points. The total number of experimental runs was 20 consisting of eight factorial, six axial, and six center points. The experimental design and results were shown in Table 2 of the paper and are reproduced here as Table 7.32.

A full standard second-order quadratic response surface model was fitted to each of the responses. The summarized ANOVA results for turbidity and for SVI were shown in Tables 3 and 4 of the paper, respectively. These tables are reproduced here as Tables 7.33 and 7.34, respectively.

For turbidity, the full second-order regression model in actual units was given as (Equation 3 of the paper):

$$\text{Turbidity} = 120.415 - 0.034\,X_1 - 0.994\,X_2 - 25.166\,X_3 + 0.0000875X_1X_2 - 0.00371X_1X_3 - 0.0562X_2X_3 + 0.0000212\,X_1^2 + 0.0280X_2^2 + 2.398X_3^2$$

From Table 7.33, the R^2 value was given as 0.898. No other goodness of fit statistics were reported and the lack of fit was not tested. Reanalysis of the turbidity data using Design-Expert 12 showed that most of the regression coefficients are not statistically significant at the 5% level and the lack of fit is highly statistically significant (p-value < 0.0001). The predicted R^2 is only 0.1512 for the full model shown. Using a reduced model with only statistically significant terms and maintaining hierarchy would improve the predicted R^2 from 0.1512 to 0.3725. However, the lack of fit would still be statistically significant.

Table 7.32: CCD and response results for the study of three experimental variables in coded units. Table 2 of Wang et al (2007).

Run	Factors			Responses	
	Coagulant dosage (X_1)	Flocculant dosage (X_2)	pH (X_3)	Turbidity (NTU)	SVI (mg/g)
1	-1	1	-1	22.6	16.3
2	1	-1	1	18	203.3
3	0	0	0	9.2	101.6
4	-1	1	1	23.8	40.7
5	-1	-1	1	30.3	48.8
6	0	0	0	7.3	89.4
7	-1	-1	-1	23.4	16.3
8	1	1	-1	26.5	16.3
9	0	0	0	8.1	81.3
10	1	1	1	15	162.6
11	1	-1	-1	28	20.3
12	0	0	0	8.5	81.3
13	2	0	0	8.5	243.9
14	0	0	2	42.9	130.1
15	0	0	0	8.1	81.3
16	0	0	0	8.9	81.3
17	0	-2	0	24.5	187
18	0	0	-2	52.1	20.3
19	0	2	0	8.1	81.3
20	-2	0	0	52.1	16.3

Table 7.33: ANOVA for turbidity. Table 3 of Wang et al (2007).

Item	Degrees of freedom	Sum of squares	Men square	F statistic	P > F
Model	9	3534.7	392.7	9.774	0.0007
Error	10	401.8	40.2		
Total	19				

$R^2 = 0.898$

Table 7.34: ANOVA for SVI. Table 4 of Wang et al (2007).

Item	Degrees of freedom	Sum of squares	Men square	F statistic	P > F
Model	9	75647.3	8405.3	6.503	0.0036
Error	10	12924.5	1292.4		
Total	19				

$R^2 = 0.854$

For SVI, the full second-order regression model in actual units was given as (Equation 4 of the paper):

$$SVI = 63.264 - 0.153X_1 - 2.981X_2 + 9.732X_3 - 0.0011X_1X_2 + 0.034X_1X_3 - 0.350X_2X_3 + 0.0000292X_1^2 + 0.130X_2^2 - 1.604X_3^2$$

The R^2 value was 0.854. Similar to turbidity, no other goodness of fit statistics were reported. Reanalysis of the SVI data using Design-Expert 12 showed that only factor X_1, X_3, and interaction of X_1 and X_3 are statistically significant at the 5% level. Furthermore, the predicted R^2 for the full model is negative (-0.1527) indicating that the model is no better than the overall mean. The lack of fit is also statistically significant (p-value = 0.0006). If a reduced model with only statistically significant terms were used, the predicted R^2 would improve to 0.592.

The authors then used each regression equation to perform optimization. For minimum turbidity, the optimal conditions of the factors were coagulant dosage of 877 mg/l, flocculant dosage of 22.6 mg/l, and pH of 6.2, giving a turbidity value of 8.6 NTU.

For minimum SVI, the optimal conditions of the factors were coagulant dosage of 338 mg/l, flocculant dosage of 19.1 mg/l, and pH of 4.5 giving a SVI of 30.9 ml/g. These results were verified from additional experimental runs shown in Table 5 of the paper and are reproduced here as Table 7.35.

Table 7.35: Measured and calculated values for the confirmation experiments. Table 5 of Wang et al (2007).

Run	Conditions	Parameter	Measured	Calculated
21	Coagulant dosage = 877 mg/l Flocculant dosage = 22.6 mg/l pH = 6.2	Turbidity (NTU)	8.7 ± 0.3	8.6
22	Coagulant dosage = 338 mg/l Flocculant dosage = 19.1 mg/l pH = 4.5	SVI (ml/g)	32.5 ± 2.4	30.9

The authors then used multiple response optimization based on the desirability function approach to determine the factor values to minimize both turbidity and SVI simultaneously. The optimal factor values were a coagulant dosage of 759 mg/l, flocculant dosage of 22.3 mg/l, and pH of 5.4. The corresponding turbidity and SVI obtained were 12.3 NTU and 48.0 ml/g, respectively.

Given the incorrect choice of models and the significant lack of fit of the chosen models, it is not clear whether the results obtained are indeed accurate.

Case Study #7.10

Yuan, X., Jia Liu, Guangming Zeng, Jingang Shi, Jingyi Tong, and Guohe Huang (2008): Optimization of conversion of waste rapeseed oil with high FFA to biodiesel using response surface methodology. Renewable Energy, 33, pp. 1678-1684.

This study used response surface methodology based on a 2^4 full-factorial rotatable central composite design to investigate the conversion to biodiesel of waste rapeseed oil with high free fatty acids as raw material to biodiesel. The goal was to maximize the conversion and to gain insights into the process affecting biodiesel production. The four factors and levels investigated were shown in Table 1 of the paper and are reproduced here as Table 7.36.

Table 7.36: Experimental range and levels of the independent variables. Table 1 of Yuan et al (2008).

Variables	Symbol coded	Range and levels				
		-2	-1	0	1	2
Methanol/oil molar ratio	X_1	4:1	5:1	6:1	7:1	8:1
Catalyst concentration (%)	X_2	0.4	0.8	1.0	1.2	1.6
Reaction time (min)	X_3	45	55	65	75	85
Temperature (°C)	X_4	25	35	45	55	65

The materials and methods used in the experiment were described in the paper. Design-Expert 6.0 was used for the experimental design and statistical analysis of the data obtained. The response variable was the conversion to biodiesel (wt. %). A rotatable CCD with $\alpha = \pm 2$ and six center points giving a total of 30 runs was used. The experimental design and results were shown in Table 2 of the paper and are reproduced here as Table 7.37.

The standard second-order response surface model with linear, two-factor interactions, and quadratic terms was fitted to the response. The summarized ANOVA results together with the goodness of fit statistics for the full quadratic model were shown in Table 3 of the paper and are reproduced here as Table 7.38.

The estimated regression coefficients and associated statistics for the full model were shown in Table 4 of the paper and are reproduced here as Table 7.39. The second-order regression equation for biodiesel conversion was given as (Equation 2 of the paper):

$$Y = 83.03 + 0.80X_1 + 0.67X_2 - 0.17X_3 + 0.58X_4 - 0.68X_1^2 - 5.05X_2^2 -1.322X_3^2 - 0.62X_4^2$$
$$-0.30X_1X_2 + 0.65X_1X_3 - 0.39X_1X_4 - 1.18X_2X_3 + 6.875 \times 10^{-3}X_2X4 + 0.027X_3X_4$$

Note that while the full model was highly statistically significant, several of the regression coefficients (X_1X_2, X_1X_3, X_1X_4, X_2X_4, and X_3X_4), were not statistically significant at the 5% level. Furthermore, there was statistically significant lack of fit (p-value=0.0411) of the full model.

Table 7.37: Full factorial central composite design matrix of four variables in coded and natural units along with the observed responses. Table 2 of Yuan et al (2008).

No.	X_1	X_2	X_3	X_4	Methanol/oil molar ratio	Catalyst concentration (%)	Reaction time (min)	T (°C)	Conversion to biodiesel (wt%)
1	-1	-1	-1	-1	5	0.8	55	35	71.28
2	1	-1	-1	-1	7	0.8	55	35	74.50
3	-1	1	-1	-1	5	1.2	55	35	76.82
4	1	1	-1	-1	7	1.2	55	35	77.07
5	-1	-1	1	-1	5	0.8	75	35	73.16
6	1	-1	1	-1	7	0.8	75	35	76.72
7	-1	1	1	-1	5	1.2	75	35	70.35
8	1	1	1	-1	7	1.2	75	35	76.06
9	-1	-1	-1	1	5	0.8	55	55	74.58
10	1	-1	-1	1	7	0.8	55	55	76.10
11	-1	1	-1	1	5	1.2	55	55	78.64
12	1	1	-1	1	7	1.2	55	55	78.10
13	-1	-1	1	1	5	0.8	75	55	74.64
14	1	-1	1	1	7	0.8	75	55	78.30
15	-1	1	1	1	5	1.2	75	55	74.87
16	1	1	1	1	7	1.2	75	55	76.71
17	-2	0	0	0	4	1.0	65	45	80.06
18	2	0	0	0	8	1.0	65	45	80.04
19	0	-2	0	0	6	0.6	65	45	60.89
20	0	2	0	0	6	1.4	65	45	64.27
21	0	0	-2	0	6	1.0	45	45	76.95
22	0	0	2	0	6	1.0	85	45	78.04
23	0	0	0	-2	6	1.0	65	25	80.81
24	0	0	0	2	6	1.0	65	65	79.80
25	0	0	0	0	6	1.0	65	45	82.29
26	0	0	0	0	6	1.0	65	45	84.05
27	0	0	0	0	6	1.0	65	45	83.39
28	0	0	0	0	6	1.0	65	45	82.50
29	0	0	0	0	6	1.0	65	45	82.55
30	0	0	0	0	6	1.0	65	45	83.40

Table 7.38: Analysis of variance (ANOVA) for the quadratic model. Table 3 of Yuan et al (2008).

Sources of variations	Sum of squares	Degrees of freedom	Mean square	F-value	Prob > F
Model	778.17	14	55.82	30.82	<0.0001
Residual	27.05	15	1.80		
Lack of fit	24.68	10	2.47	5.22	0.0411
Pure error	2.37	5	0.47		
Total	805.22	29			

CV = 1.75%, R^2 = 0.9664, R = 0.9830, Adj. R^2 = 0.9351.

Table 7.39: The least squares fit and parameter estimates (significance of regression coefficients). Table 4 of Yuan et al (2008).

Model Term	Parameter estimate	Standard error	Computed t-value	P-value
Intercept	83.03	0.55	150.96	
X_1	0.80	0.27	2.96	0.0180
X_2	0.67	0.27	2.481	0.0276
X_3	-0.17	0.27	-0.63	0.5474
X_4	0.58	0.27	2.15	0.0502
X_1^2	-0.68	0.26	-1.15	0.0180
X_2^2	-5.05	0.26	-3.47	<0.0001
X_3^2	-1.32	0.26	0.02	0.0001
X_4^2	-0.62	0.26	0.08	0.0294
X_1X_2	-0.29	0.34	-2.62	0.3905
X_1X_3	0.65	0.34	-19.42	0.0727
X_1X_4	-0.39	0.34	-5.08	0.2657
X_2X_3	-1.18	0.34	-2.38	0.0030
X_2X_4	6.875E-03	0.34	-0.88	0.9839
X_3X_4	0.027	0.34	1.91	0.9373

A reduced quadratic model with only statistically significant terms and terms to maintain hierarchy would have provided a better model with a higher predicted R^2. That is, predicted R^2 (full model) = 0.8192, predicted R^2 (reduced model) = 0.8590.

The optimal values of the factors in coded units were $X_1 = 0.50$, $X_2 = 0.05$, $X_3 = 0.04$, and $X_4 = 0.32$ which corresponded to the actual units of methanol/oil molar ratio of 6.5:1, catalyst concentration of 1.0%, reaction time of 65.4 min, and temperature of 48.2 °C. The maximum biodiesel conversion at these factor values was 83.34%. The method used for optimization was not discussed in the paper.

8. RSM: CENTRAL COMPOSITE DESIGNS (Face-centered)

10 case studies are presented in this Chapter that use Response Surface Methodology that is based on face-centered Central Composite Designs or FCD designs. Face-centered designs are special cases of CCD where the axial points are at the -1 and +1 levels. The number of factors ranges from two to five.

Case Study #8.1

Alvarez, M. J., N. Gil-Negrete, L. Ilzarbe, M. Tanco, E. Viles, and A. Asensio (2009): A computer experiment application to the design and optimization of a capacitive accelerometer. Applied Stochastic Models in Business and Industry, 25, pp. 151-162.

This study used computer experiments based on finite element analyses to investigate the geometrical factors that affect the performance of a capacitive accelerometer for measuring accelerations acting on buildings. The five factors chosen were: the thickness of the seismic mass, X_1; the thickness of the beams, X_2; the area of the seismic mass, X_3; the length of the beams, X_4; and the width of the beams, X_5. The response was the natural frequency of the accelerometer.

The authors used response surface methodology and a second-order polynomial to model the natural frequency as a function of the five factors. A face-centered central composite design (FCD) was chosen and the factors and levels were shown in Table I of the paper and are reproduced here as Table 8.1.

The FCD with five factors require 32 factorial points, 10 axial points, and one center point. Only one center point was used because since this was a computer based experiment, identical results would be produced on replicated runs. The experimental design and responses are shown in Table II of the paper and are shown here as Table 8.2. Minitab software was used for statistical analyses.

Table 8.1: Factor and levels. Table I of Alvarez et al (2009).

Level	X_1: thickness of seismic mass (μm)	X_2: thickness of beams (μm)	X_3: area seismic mass (μm) x (μm)	X_4: length of beams (μm)	X_5: width of beams (μm)
1	433	17.5	3050 x 3050	500	900
0	400	12.5	2050 x 2050	1100	580
-1	367	7.5	1050 x 1050	1700	260

Table 8.2: Experimental matrix and response values for FCD design. Table II of Alvarez et al (2009).

Test	Thickness seismic mass X_1	Thickness of beams X_2	Area seismic mass X_3	Length of beams X_4	Width of beams X_5	y= Frec. (Hz)	ln y
1	-1	-1	-1	-1	-1	5118.3	8.54057
2	1	-1	-1	-1	-1	4961.9	8.50954
3	-1	1	-1	-1	-1	17470.0	9.76824
4	1	1	-1	-1	-1	16957.0	9.73844
5	-1	-1	1	-1	-1	1562.4	7.35398
6	1	-1	1	-1	-1	1462.2	7.28770
7	-1	1	1	-1	-1	5397.4	8.59367
8	1	1	1	-1	-1	5053.2	8.52778
9	-1	-1	-1	1	-1	839.3	6.73257
10	1	-1	-1	1	-1	814.0	6.70196
11	-1	1	-1	1	-1	2901.8	7.97309
12	1	1	-1	1	-1	2818.2	7.94385
13	-1	-1	1	1	-1	244.2	5.49799
14	1	-1	1	1	-1	228.5	5.43154
15	-1	1	1	1	-1	861.0	6.75809
16	1	1	1	1	-1	806.1	6.69221
17	-1	-1	-1	-1	1	9458.6	9.15468
18	1	-1	-1	-1	1	9171.6	9.12387
19	-1	1	-1	-1	1	32463.0	10.38786
20	1	1	-1	-1	1	31522.0	10.35844
21	-1	-1	1	-1	1	2739.1	7.91538
22	1	-1	1	-1	1	2564.3	7.84944
23	-1	1	1	-1	1	9417.4	9.15031
24	1	1	1	-1	1	8825.3	9.08538
25	-1	-1	-1	1	1	1489.1	7.30593
26	1	-1	-1	1	1	1445.9	7.27649
27	-1	1	-1	1	1	5039.4	8.52504
28	1	1	-1	1	1	4906.0	8.49821
29	-1	-1	1	1	1	482.1	6.17815
30	1	-1	1	1	1	451.5	6.11258
31	-1	1	1	1	1	1691.0	7.43308
32	1	1	1	1	1	1585.2	7.36847
33	-1	0	0	0	0	2408.4	7.78672
34	1	0	0	0	0	2276.4	7.73035
35	0	-1	0	0	0	1099.2	7.00234
36	0	1	0	0	0	3825.7	8.24950
37	0	0	-1	0	0	5194.9	8.55543
38	0	0	1	0	0	1493.1	7.30861
39	0	0	0	-1	0	7080.6	8.86511
40	0	0	0	1	0	1209.4	7.09788
41	0	0	0	0	-1	1559.9	7.35238
42	0	0	0	0	1	2935.2	7.98453
43	0	0	0	0	0	2337.6	7.75688

The estimated regression coefficients and statistical significance of a full quadratic model were shown in Table III of the paper and are reproduced here as Table 8.3. The polynomial prediction equation in coded factors for the logarithm of the natural frequency using only statistically significant terms at the 5% level was given as:

$$\begin{aligned}\ln \mathrm{y} = 7.7587 - 0.02409\, x_1 + 0.61991\, x_2 - 0.60441\, x_3 - 0.90245\, x_4 + 0.30307\, x_5 \\ - 0.13301\, x_2^2 + 0.17309\, x_3^2 + 0.22256\, x_4^2 - 0.0948\, x_5^2 + 0.0104\, x_3\, x_4\end{aligned}$$

The model has an adjusted R^2 of 0.999, and all regression assumptions were checked and fulfilled.

Table 8.3: Estimated regression coefficients. Table III of Alvarez et al (2009).

	Term	Coeff	SE coeff	T	P
	Constant	7.75870	0.01033	751.457	0.000
x_1	Thickness of seismic mass	-0.02409	0.00479	-5.029	0.000
x_2	Thickness of beams	0.61991	0.00479	129.389	0.000
x_3	Area of seismic mass	-0.60441	0.00479	-126.154	0.000
x_4	Length of beams	-0.90245	0.00479	-188.362	0.000
x_5	Width of beams	0.30307	0.00479	63.257	0.000
x_1^2	Thickness of mass x thickness of mass	-0.00040	0.01783	-0.022	0.982
x_2^2	Thickness of beams x thickness of beams	0.13301	0.01783	-7.461	0.000
x_3^2	Area seismic mass x area seismic mass	0.17309	0.01783	9.709	0.000
x_4^2	Length of beams x length of beams	0.22256	0.01783	12.484	0.000
x_5^2	Width of beams x width of beams	-0.09048	0.01783	-5.075	0.000
x_1x_2	Thickness s. mass x thickness of beams	0.00030	0.00494	0.060	0.952
x_1x_3	Thickness of s. mass x area seismic mass	-0.00901	0.00494	-1.825	0.082
x_1x_4	Thickness of s. mass x thickness of beams	0.00017	0.00494	0.035	0.973
x_1x_5	Thickness of s. mass x width of beams	0.00024	0.00494	0.048	0.962
x_2x_3	Thickness of beams x area seismic beams	0.00421	0.00494	0.852	0.403
x_2x_4	Thickness of beams x length of beams	0.00250	0.00494	0.506	0.618
x_2x_5	Thickness of beams x width of beams	-0.00154	0.00494	-0.312	0.758
x_3x_4	Area of seismic mass x length of beams	0.01040	0.00494	2.107	0.047
x_3x_5	Area of sesmic mass x width of beams	0.00711	0.00494	1.440	0.164
x_4x_5	Length of beams x width of beams	0.00816	0.00494	1.653	0.113

The ANOVA results, showing the contribution of the various order of terms, considering only the statistically significant terms, were shown in Table IV of the paper. The table is reproduced here as Table 8.4.

Table 8.4: Analysis of variance for ln y. Table IV of Alvarez et al (2009).

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	10	56.6842	56.6842	5.6684	7443.81	0.000
Linear	5	56.3190	56.3190	11.2638	14791.72	0.000
Square	4	0.3617	0.3617	0.0904	118.74	0.000
Interaction	1	0.0035	0.0035	0.0035	4.55	0.041
Residual error	32	0.0244	0.0244	0.0008		
Total	42					

$R^2 = 0.999$

From Table 8.4, it shows that linear terms have more significant contribution than quadratic terms, and interaction terms have the least contribution. Furthermore, among the five factors, the seismic mass has the lowest influence on the natural frequency.

The authors did not provide any additional experimental runs that were not used for model development, to verify the model.

Case Study #8.2

Chiang, Ko-Ta, and Fu-Ping Chang (2006): Application of response surface methodology in the parametric optimization of a pin-fin type heat sink. International Communications in Heat and Mass Transfer, 33, pp. 836-845.

This study used response surface methodology to find optimal values for the design parameters of a pin-fin type heat sink (PFHS) to achieve high thermal performance (or cooling efficiency). These heat sinks are widely used to provide cooling of electronic components. Experiments were performed using a face-centered central composite design (FCD) with four factors. The factors and levels used in the experiment were given in Table 1 of the paper and are shown here in Table 8.5.

Table 8.5: Design scheme of designing parameters and their levels. Table 1 of Chiang et al (2006).

			Levels	
Symbol	Factor	Unit	Low (-1)	High (+1)
A	Fin Height, H	mm	50	60
B	Pin diameter, D	mm	4	6
C	Longitudinal pitch, S1	mm	6	10
D	Transverse pitch, S2	mm	8	12

The two responses of relevance in this experiment were the thermal resistance, Rth (°C/W) and pressure drop, ΔP (Pa). The design and other technical details of the heat sink were described in detail in the paper.

The FCD had a total of 30 experimental runs (16 factorial points, eight axial points, and six center points). The experimental design and measured responses were given in Table 2 of the paper and are reproduced here as Table 8.6. The software used for the design and analysis of the experiment was not mentioned.

Table 8.6: Design of experimental matrix and results for the PFHS performance characteristics. Table 2 of Chiang et al (2006).

	Design parameters				Experimental results	
Exp No.	(A) Fin height	(B) Pin diameter	(C) Long. Pitch	(D) Trans. Pitch	Therm resist, R_{th} (°C/W)	Pressure drop, ΔP (Pa)
1	50	4	6	8	0.258	28.3
2	60	4	6	8	0.213	23.8
3	50	6	6	8	0.243	26.8
4	60	6	6	8	0.201	22.6
5	50	4	10	8	0.241	26.6
6	60	4	10	8	0.198	22.3
7	50	6	10	8	0.217	24.2
8	60	6	10	8	0.193	21.8
9	50	4	6	12	0.235	26.2
10	60	4	6	12	0.204	22.9
11	50	6	6	12	0.231	25.6
12	60	6	6	12	0.201	20.9
13	50	4	10	12	0.236	26.1
14	60	4	10	12	0.198	21.8
15	50	6	10	12	0.212	23.7
16	60	6	10	12	0.189	21.4
17	50	5	8	10	0.231	25.6
18	60	5	8	10	0.203	22.8
19	55	4	8	10	0.224	24.9
20	55	6	8	10	0.211	23.6
21	55	5	6	10	0.235	25.2
22	55	5	10	10	0.214	23.9
23	55	5	8	8	0.223	24.8
24	55	5	8	12	0.218	23.7
25	55	5	8	10	0.224	24.8
26	55	5	8	10	0.232	24.6
27	55	5	8	10	0.223	25.1
28	55	5	8	10	0.222	24.7
29	55	5	8	10	0.227	24.4
30	55	5	8	10	0.223	24.5

The authors fitted a second-order polynomial regression model to each of the responses. The full ANOVA results including all terms in the models were given in Tables 3 and 5 of the paper for the thermal resistance and pressure drop, respectively. The authors also showed ANOVA tables resulting from using the backward elimination procedure to reduce the model. These reduced ANOVA tables for thermal resistance and pressure drop were shown as Tables 4 and 6 in the paper and are reproduced here as Tables 8.7 and 8.8, respectively. It is of interest to note that after using backward elimination, there are still statistically insignificant terms left in the ANOVA tables. It is also likely that statistically significant terms have been incorrectly left out of the tables. Hence the models developed by the authors may not be the best models.

Table 8.7: ANOVA table for the thermal resistance (after backward elimination). Table 4 of Chiang et al (2006).

Source	Sum of squares	DF	Mean square	f-value	Prob>F	
Model	0.00694	5	0.001389	40.3058	<0.0001	Significant
A	0.00513	1	0.005134	148.9730	<0.0001	
B	0.00066	1	0.000660	19.1520	0.0002	
C	0.00084	1	0.000841	24.3877	<0.0001	
D	0.000221	1	0.000221	6.3980	0.0184	
AB	9.03E-05	1	9.03E-05	2.6187	0.1187	
Residual	0.000827	24	3.45E-05			
Lack of fit	0.000756	19	3.98E-05	2.8098	0.1279	Not significant
Pure error	7.083E-05	5	1.42E-05			
Cor. Total	0.007772	29				
Standard deviation = 0.005870			R^2 = 0.89358			
Mean = 0.219333			R^2 adjusted = 0.87141			
Coefficient of variation = 2.676572			Predicted R^2 = 0.82395			
PRESS = 0.001368			Adequate precision = 25.3505			

Table 8.8: ANOVA table for the pressure drop (after backward elimination). Table 6 of Chiang et al (2006).

Source	Sum of squares	DF	Mean square	f-value	Prob>F	
Model	79.69944	5	15.93988	65.7850	<0.0001	Significant
A	59.76888	1	59.76888	246.6720	<0.0001	
B	8.405	1	8.40500	34.6880	<0.0001	
C	6.12500	1	6.12500	25.2780	<0.0001	
D	4.40055	1	4.40055	18.1610	0.0003	
CD	1.00E+00	1	1.00000	4.1270	0.0534	
Residual	5.81522	24	0.24230			
Lack of fit	5.506888	19	0.28984	4.7000	0.0572	Not significant
Pure error	0.308330	5	0.061666			
Cor. Total	85.512466	29				
Standard deviation = 0.492240			R^2 = 0.93199			
Mean = 24.253333			R^2 adjusted = 0.91783			
Coefficient of variation = 2.029579			Predicted R^2 = 0.87782			
PRESS = 10.447778			Adequate precision = 32.55552			

From Table 8.7, the final response equations for thermal resistance in terms of coded and actual factors were given as Equations 5 and 6, respectively, in the paper. They are reproduced here below:

Coded factors:

Thermal resistance = 0.2193 – 0.0169 A – 0.0061 B – 0.0068 C – 0.0035 D + 0.0024 AB

Actual factors:

Thermal resistance = 0.6108 – 0.0058 H – 0.0322 D – 0.0034 S1 – 0.0018 S2 + 0.0005 HD.

From Table 8.8, the final response equations for pressure drop in terms of coded and actual factors were given as Equations 7 and 8, respectively, in the paper. These equations are reproduced below:

Coded factors:

Pressure drop = 24.2533 – 1.8222 A – 0.6833 B – 0.5833 C – 0.4944 D + 0.25 CD

Actual factors:

Pressure drop = 57.52 – 0.3644 H – 0.6833 D – 0.9166 S1 – 0.7472 S2 + 0.0625 S2.

The authors then went on to find the optimal values for the fin parameters [H, D, S1, S2] to minimize thermal resistance and pressure drop using a sequential approximation optimization method. They also set the constraints for the parameters as: $50 \leq H \leq 60$ mm, $D \leq 6$ mm, $6 \leq S1 \leq 8$, $8 \leq S2 \leq 12$ mm, and mass of the PFHS ≤ 620 g.

The optimal settings obtained were shown in Table 7 of the paper and are shown here as Table 8.9.

Table 8.9: The optimal designing parameters. Table 7 of Chiang et al (2006).

Parameters	Unit	Optimal value
Fin height, H	mm	60
Pin diameter, D	mm	4.76
Longitudinal pitch, S1	mm	10
Transverse pitch, S2	mm	11.96
Predicted value		
Thermal resistance, R_{th}	°C/W	0.192
Pressure drop, ΔP	Pa	21.71

To verify the adequacy of the developed models, three confirmation runs were performed. The results obtained for the thermal resistance and pressure drop using the regression models were then compared to those obtained experimentally. These comparisons were shown in Table 8 of the paper and are reproduced here as Table 8.10. However, no confirmation runs were done at the optimal settings.

Table 8.10: Confirmation experiments. Table 8 of Chiang et al (2006).

Exp No.	Designing parameters				Thermal resistance, R_{th}			Pressure drop, ΔP		
	A	B	C	D	Exp.	Predicted	Error (%)	Exp.	Predicted	Error (%)
1	50	4	6	12	0.235	0.247	-5.11	26.2	26.5	-1.15
2	55	5	8	10	0.224	0.219	2.23	24.8	24.2	2.42
3	60	6	10	8	0.193	0.202	4.66	21.8	22.7	-4.13

Based on the confirmation runs, the authors concluded that the developed models were accurate, even though no confirmation runs were performed at the optimal settings.

Case Study #8.3

Jenarthanan, M. P. and R. Jeyapaul (2014): Machinability study of carbon fibre reinforced polymer (CFRP) composites using design of experiment technique. Pigment & Resin Technology, 43/1, pp. 35-44.

This study investigated the influence of three input CNC machining parameters on the surface delamination of carbon fibre reinforced polymer (CFRP) when milled by a machine with a solid carbide end mill coated with crystalline diamond. The input parameters were the cutting speed, feed rate, and depth of cut. A second-order response surface model based on a face-centred central composite design (FCD) was used. The FCD had eight factorial points, six axial points, and six center points giving a total of 20 run combinations. The response measured was the delamination factor, F_d. The materials and experimental procedure used were described in the paper. The factors and their levels used were given in Table II of the paper and are reproduced here as Table 8.11.

Table 8.11: Process control parameters and their levels. Table II of Jenarthanan et al (2014).

				Levels		
Process parameters	Units	Notattion	Variable	-1	0	1
Feed rate	mm/rev	f	A	0.04	0.08	0.12
Cutting speed	m/min	V	B	50	75	100
Depth of cut	mm	D	C	0.05	0.15	0.25

The design and analysis of the experimental data were carried out using Design-Expert 8.0 software. The experimental layout in coded and actual factors and the results were shown in Table III of the paper and are reproduced here as Table 8.12.

The authors fitted a full second-order (main effects, two-factor interactions, and quadratic terms) model to the responses. The ANOVA results were shown in Table IV of the paper and are shown here as Table 8.13.

Table 8.12: Layout of full factorial face-centred central composite design with results. Table III of Jenarthanan et al (2014).

	Coded variables			Uncoded variables			Delamination factor
Run	A	B	C	f	V	D	Fd
1	0	0	0	0.08	75	0.15	1.0209
2	-1	-1	-1	0.04	50	0.05	1.0113
3	1	0	0	0.12	75	0.15	1.0537
4	1	1	1	0.12	100	0.25	1.0654
5	1	-1	-1	0.12	50	0.05	1.0426
6	0	0	0	0.08	75	0.15	1.0319
7	-1	-1	1	0.04	50	0.25	1.0063
8	0	0	1	0.08	75	0.25	1.0337
9	0	0	-1	0.08	75	0.05	1.0238
10	-1	1	-1	0.04	100	0.05	1.0051
11	0	1	0	0.08	100	0.15	1.0408
12	1	-1	1	0.12	50	0.25	1.0436
13	0	0	0	0.08	75	0.15	1.0319
14	-1	1	1	0.04	100	0.25	1.0186
15	1	1	-1	0.12	100	0.05	1.0579
16	0	0	0	0.08	75	0.15	1.0319
17	0	0	0	0.08	75	0.15	1.0319
18	0	-1	0	0.08	50	0.15	1.0258
19	0	0	0	0.08	75	0.15	1.0319
20	-1	0	0	0.04	75	0.15	1.0055

Table 8.13: ANOVA for delamination. Table IV of Jenarthanan et al (2014).

Source	Sum of squares	df	Mean squares	F-value	p-value	Effect	% contribution
Model	5.533×10^{-03}	9	6.148×10^{-04}	386.5	<0.0001	Significant	99.71
A	4.844×10^{-03}	1	4.844×10^{-03}	3045.7	<0.0001	Significant	87.28
B	5.314×10^{-04}	1	5.314×10^{-04}	334.1	<0.0001	Significant	9.56
C	1.129×10^{-04}	1	1.129×10^{-04}	71	<0.0001	Significant	2.03
AB	4.961×10^{-06}	1	4.961×10^{-06}	3.1	0.1078	Insignificant	0.08
AC	6.661×10^{-06}	1	6.661×10^{-06}	4.2	0.0679	Insignificant	0.11
BC	3.612×10^{-07}	1	3.612×10^{-07}	0.2	0.6439	Insignificant	0.00
A^2	1.903×10^{-06}	1	1.903×10^{-06}	1.2	0.2997	Insignificant	0.03
B^2	3.120×10^{-05}	1	3.120×10^{-05}	19.6	0.0013	Significant	0.55
C^2	3.841×10^{-06}	1	3.841×10^{-06}	2.4	0.1512	Insignificant	0.07
Error	1.591×10^{-05}	10	1.591×10^{-06}			Insignificant	0.29
Total	5.549×10^{-03}	19					
Notes: R^2 = 99.91; adjusted R^2 = 99.46							

From Table 8.13, many of the model terms were not statistically significant at the 5% level. Only the main effects A, B, C, and the quadratic term B^2 were statistically significant. However, the authors suggested a final model with all terms regardless of statistical significance. The final model in terms of coded factors was:

$$\begin{aligned}\text{Delamination Factor } (F_d) = {} & 1.03 + 0.022\ A + 7.290 \times 10^{-03}\ B + 3.360 \times 10^{-03}\ C \\ & + 7.875 \times 10^{-04}\ AB + 9.125 \times 10^{-04}\ AC - 2.125 \times 10^{-04}\ BC \\ & - 8.318 \times 10^{-04}\ A^2 + 3.368 \times 10^{-03}\ B^2 - 1.182 \times 10^{-03}\ C^2\end{aligned}$$

And, the final model in terms of actual factors was:

$$\begin{aligned}\text{Delamination Factor } (F_d) = {} & 0.99042 + 0.54015\ \text{feedrate} - 5.67014 \times 10^{-04}\ \text{cuttingspeed} \\ & + 0.057180\ \text{depthofcut} + 7.87500 \times 10^{-04}\ \text{feedrate x cuttingspeed} \\ & + 0.22813\ \text{feedrate x depthofcut} \\ & - 8.5000 \times 10^{-05}\ \text{cuttingspeed x depthofcut} \\ & + 5.38909 \times 10^{-06}\ (\text{cuttingspeed})^2 - 0.11818\ (\text{depthofcut})^2\end{aligned}$$

There is a squared term $(\text{feedrate})^2$ missing from the model with actual factors. This term may have been accidentally left out by the authors.

The adjusted R^2 for the model was 99.46 which was an excellent fit to the data. The authors also ran 20 additional confirmation experiments to validate the model. The results from experiments were compared those obtained from the regression model. These comparison results were shown in Table VI of the paper. The error was found to be within ±0.3 percent. Since the factor settings were not given, it is not possible to reproduce the results shown here.

Reanalyses of the published data using both Design-Expert 12 and Minitab 18 did not reproduce the results reported by the authors. Some of the F-values differed by a factor of 10. Hence, it is possible the ANOVA results shown were incorrect. If this is so, then the models developed were also incorrect.

Case Study #8.4

Kwon, J-H, Sang-Moon Hwang, Chang-Min Lee, Kwang-Suk Kim, and Gun-Yong Hwang (2009): Application of response surface methodology (RSM) in microspeaker design used in mobile phones. IEEE Transactions on Magnetics, Vol. 45, N. 10, pp. 4550-4553.

This study investigated the design of a microspeaker used in mobile phones using response surface methodology based on a face-centered central composite design. The parameters that influence a magnetic circuit design of the microspeaker included the dimensions of the top plate, yoke, and radius of the inner magnet. The design drawing and constraints were given in the paper. The factors and levels used in the study are summarized in Table 8.14. The responses were the mean and variance of the electromagnetic force obtained using finite element analysis. Hence this is a computer based experiment. The goal was to design a microspeaker with a high mean electromagnetic force but with minimum variance.

Table 8.14: Factors and levels used in the experiment.

			Levels		
Factor	Description	Unit	-1	0	1
x_1	Radius of inner magnet	mm	3.3	3.8	4.3
x_2	Thickness of yoke	mm	0.2	0.3	0.4
x_3	Thickness of top plate	mm	0.2	0.3	0.4

The authors used a three-factor full-factorial face-centered central composite design (FCD) with eight factorial points, six axial points, and one center point (total of 15 run combinations). The FCD design together with the mean and variance of the electromagnetic force were given in Table 1 of the paper and are reproduced here as Table 8.15.

Table 8.15: Experimental design matrix and results. Table 1 of Kwon et al (2009).

	Coded factors			Responses	
Std Ord	x1	x2	x3	Mean (mN)	Variance {x10^{-6})
1	-1	-1	-1	45.9	16.6
2	1	-1	-1	47.1	26.5
3	-1	1	-1	46.8	17.3
4	1	1	-1	48.3	27.0
5	-1	-1	1	45.3	12.9
6	1	-1	1	43.8	58.5
7	-1	1	1	46.8	13.6
8	1	1	1	45.6	62.7
9	-1	0	0	48.3	12.4
10	1	0	0	48.0	48.5
11	0	-1	0	48.7	24.2
12	0	1	0	49.9	24.7
13	0	0	-1	48.4	14.3
14	0	0	1	46.7	35.2
15	0	0	0	49.4	24.6

Full second-order regression models were fitted to the two responses. No ANOVA results were given in the paper. Only the fitted regression models in terms of coded factors for the mean and variance of the electromagnetic force were shown (Equation 4 and 5, respectively). These equations are:

$$F_{mean} = 49.4 - 0.03\,x_1 + 0.66\,x_2 - 0.83\,x_3 - 1.25\,\mathrm{x}_1^2 - 0.10\,\mathrm{x}_2^2 - 1.85\,\mathrm{x}_3^2 + 0.075\,\mathrm{x}_1\mathrm{x}_2 - 0.675\,\mathrm{x}_1\mathrm{x}_3 + 0.15\,\mathrm{x}_2 x_3$$

$$F_{variance} = 24.98 + 15.04\,x_1 + 0.66\,x_2 + 8.12\,x_3 + 5.38\,\mathrm{x}_1^2 - 0.62\,\mathrm{x}_2^2 - 0.32\,\mathrm{x}_3^2 + 0.4125\,\mathrm{x}_1\mathrm{x}_2 + 9.3875\,\mathrm{x}_1\mathrm{x}_3 + 0.4625\,\mathrm{x}_2 x_3$$

There was no indication as to which of the regression coefficients were statistically significant. Not goodness-fit-statistics were given and no assumptions checks were mentioned.

Using the developed regression models, the authors obtained the following dimensions (in coded factors) that minimized variance and kept the mean value above 99% of its possible maximum:

$x_1 = -0.41$, $x_2 = 1$, and $x_3 = -0.5$

There was also a limitation in the manufacturing process in that the thicknesses of the yoke sand top plate (x_2 and x_3, respectively) can only be produced in intervals of 0.5 mm.

The authors concluded that the combined use of RSM and finite element analysis in the design of the microspeaker had produced a design that minimized variance while maximizing electromagnetic force.

Case Study #8.5

Mohajeri, L., Hamidi Abdul Aziz, Mohamed Hasnain Isa, and Mohammad Ali Zahed (2010): A statistical experiment design approach for optimizing biodegradation of weathered crude oil in coastal sediments, Bioresource Technology, 1011, pp. 893-900.

This study used a face-centered central composite design (FCD) to develop a second-order response surface equation to model the percentage removal of weathered crude oil (WCO) as a function of four independent variables – initial oil concentration, biomass, nitrogen and phosphorus concentrations. The factors and levels used in the experiment were given in Table 2 of the paper and are reproduced here as Table 8.16.

Table 8.16: Coded and actual values of variables used in the response surface study. Table 2 of Mohajeri et al (2010).

Factor	Symbol	Coded levels of variables		
		Low level (-1)	Center (0)	High level (+1)
Oil (g)	A	2	16	30
Biomass	B	0	1	2
Nitrogen (g)	C	0.2	1.6	3
Phosphorus (g)	D	0.02	0.16	0.3

The FCD consisted of 16 factorial points, eight axial points, and six center points. Design-Expert 6.07 was used for the experimental design and subsequent statistical analysis and optimization. The background of the study and the laboratory methods used were given in the paper. The experimental matrix and the results were shown in Table 3 and 4 of the paper and are combined here as Table 8.17. Three control experiments were also conducted but were not used as part of the experimental design and analysis.

Table 8.17: Experimental matrix for central composite design (CCD) for overall optimization and results of WCO removal. Tables 3 and 4 of Mohajeri et al (2010).

Run no.	Point type	Factors				Percent WCO removal		
		Oil (g)	Biomass	N (g)	P (g)	Observed	Predicted	StD
1	Fact	2	0	0.2	0.02	47.01	50.59	2.53
2	Fact	30	0	0.2	0.02	15.62	16.79	0.83
3	Fact	2	2	0.2	0.02	67.85	62.48	3.79
4	Fact	30	2	0.2	0.02	19.78	19.55	0.16
5	Fact	2	0	3	0.02	54.06	49.98	2.88
6	Fact	30	0	3	0.02	51.25	50.71	0.38
7	Fact	2	2	3	0.02	60.24	61.87	1.15
8	Fact	30	2	3	0.02	53.57	53.47	0.07
9	Fact	2	0	0.2	0.16	58.96	58.12	0.60
10	Fact	30	0	0.2	0.16	25.63	24.32	0.93
11	Fact	2	2	0.2	0.16	66.33	70.01	2.60
12	Fact	30	2	0.2	0.16	27.27	27.07	0.14
13	Fact	2	0	3	0.16	63.01	64.75	1.23
14	Fact	30	0	3	0.16	66.02	65.48	0.38
15	Fact	2	2	3	0.16	77.13	76.64	0.35
16	Fact	30	2	3	0.16	69.87	68.24	1.16
17	Axial	2	1	1.6	0.09	76.58	78.98	1.70
18	Axial	30	1	1.6	0.09	52.23	57.88	4.00
19	Axial	16	0	1.6	0.09	68.75	69.56	0.57
20	Axial	16	2	1.6	0.09	74.17	76.88	1.92
21	Axial	16	1	0.2	0.09	43.89	43.41	0.34
22	Axial	16	1	3	0.09	59.68	63.69	2.83
23	Axial	16	1	1.6	0.02	51.85	55.78	2.78
24	Axial	16	1	1.6	0.16	67.33	66.93	0.29
25	Center	16	1	1.6	0.09	71.86	68.43	2.42
26	Center	16	1	1.6	0.09	69.08	68.43	0.46
27	Center	16	1	1.6	0.09	72.38	68.43	2.79
28	Center	16	1	1.6	0.09	68.33	68.43	0.07
29	Center	16	1	1.6	0.09	70.15	68.43	1.21
30	Center	16	1	1.6	0.09	73.9	68.43	3.87
31	Control	2	0	0	0	12.6	-	-
32	Control	16	0	0	0	10.9	-	-
33	Control	30	0	0	0	9.1	-	-

The authors fitted a full second order regression model to the WCO removal percentage data and the ANOVA results, showing only the statistically significant terms at the 5% level, were given in Table 5 of the paper. The table is reproduced here as Table 8.18.

Table 8.18: Analysis of variance for response surface quadratic model terms. Table 5 of Mohajeri et al (2010).

Source	Sum of Squares	df	Mean Square	F-value	p-value	Remarks
Model	8106.65	10	810.67	69.04	< 0.0001	significant
A-A	2004.08	1	2004.08	170.68	< 0.0001	significant
B-B	241.27	1	241.27	20.55	0.0002	significant
C-C	1850.14	1	1850.14	157.57	< 0.0001	significant
D-D	559.12	1	559.12	47.62	< 0.0001	significant
B^2	65.1	1	65.1	5.54	0.0294	significant
C^2	629.04	1	629.04	53.57	< 0.0001	significant
D^2	142.34	1	142.34	12.12	0.0025	significant
AB	83.45	1	83.45	7.11	0.0153	significant
AC	1192.32	1	1192.32	101.55	< 0.0001	significant
CD	52.49	1	52.49	4.47	0.0479	significant
Residual	223.09	19	11.74			
Lack of Fit	200.51	14	14.32	3.17	0.1044	not significant
Pure Error	22.58	5	4.52			
Cor Total	8329.74	29				

The regression model based on only statistically significant terms is given by (Equation 3 in the paper):

$$\text{WCO removal (percent)} = 68.43 - 10.55\,A + 3.66\,B + 10.14\,C + 5.57\,D + 4.79\,B^2 - 14.89\,C^2 - 7.08D^2 - 2.28\,AB + 8.63\,AC + 1.81\,CD$$

The model has a R^2 of 0.9732, adjusted R^2 of 0.9591, and predicted R^2 of 0.9336. The interpretation of the interaction terms were given in the paper.

The authors used the developed regression model to obtain factor settings to maximize WCO removal for initial oil concentrations of 2, 16, and 30 g/kg. The results comparing observed and predicted values were given in Table 6 of the paper and are reproduced here as Table 8.19.

Table 8.19: Optimum conditions found by Design-Expert for the WCO bioremediation. Table 6 of Mohajeri et al (2010).

				WCO percent removal			
Oil (g)	Biomass	N (g)	P (g)	Observed	Predicted	Percent error	StD
2	2	0.680	0.140	83.13	85.01	-2.26	1.33
16	2	2.164	0.231	78.06	80.59	-3.24	1.79
30	2	2.520	0.250	69.92	71.54	-2.32	1.15

The authors also compared the unoptimized values at for 2, 16, and 30 g/kg (77.13%, 74.17%, and 69.87%, respectively) of initial oil concentration with the optimized values (83.13%, 78.06%, and 69.92%, respectively).

Case Study #8.6

Noordin, M. Y., V.C. Venkatesh, S. Sharif, S. Elting, and A. Abdullah (2004): Application of response surface methodology in describing the performance of coated carbide tools when turning AISI 1045 steel. Journal of Materials Processing Technology, 145, pp. 46-58.

This study investigated the factors that influence the surface roughness and tangential force of a multilayer tungsten carbide tool when turning AISI 1045 steel. The factors investigated were the cutting speed, feed, and the side cutting edge angle (SCEA) of the cutting edge. The experiments were based on a face-centered central composite design (FCD). The factors and levels used in the experiment were shown in Table 1 of the paper and are reproduced here as Table 8.20.

Table 8.20: Factors and levels for response surface study. Table 1 of Noordin et al (2004).

Factor	Low level (-1)	High Level (+1)
A - cutting speed (m/min)	240	375
B - feed (mm/rev)	0.18	0.28
C - SCEA (°)	-5	0

The FCD consisted of 16 experimental points (eight factorial points, six axial points, and two center points). The responses measured were the surface roughness, R_a (μ) of the turned surface, and the main cutting force, i.e. the tangential force, F_c (N). The experimental design and results were shown in Tables 2 and 3 of the paper. The tables are combined here as Table 8.21.

Table 8.21: Completed design layout and experimental results. (Tables 2 and 3 of Noordin et al (2004).

		Factor				
Std No.	Run	A-cutting speed (m/min)	B - feed (mm/rev)	C - SCEA (°)	Surface roughness, R_a (μ)	Tangential force, F_c (N)
1	8	240	0.18	-5	1.68	395.98
2	9	375	0.18	-5	1.40	372.24
3	12	240	0.28	-5	3.18	550.14
4	15	375	0.28	-5	2.95	525.85
5	13	240	0.18	0	1.20	372.83
6	3	375	0.18	0	1.42	366.48
7	11	240	0.28	0	3.80	559.22
8	2	375	0.28	0	3.25	553.50
9	14	240	0.23	-3	2.14	443.10
10	16	375	0.23	-3	2.08	432.27
11	6	300	0.18	-3	1.14	351.14
12	7	300	0.28	-3	2.99	540.77
13	1	300	0.23	-5	2.17	440.92
14	4	300	0.23	0	2.32	465.40
15	10	300	0.23	-3	1.76	436.10
16	5	300	0.23	-3	1.74	428.88

Design-Expert version 6 was used for the design of the experiment and subsequent analysis of the results.

A full-second order regression model was fitted to each of the responses. The ANOVA tables for the reduced models (using only statistically significant terms at the 5% level) for R_a and F_c were shown in Tables 5 and 6 of the paper, respectively. These tables are reproduced here as Tables 8.22 and 8.23, respectively.

Table 8.22: Resulting ANOVA table (partial sum of squares) for reduced quadratic model (response: surface roughness, R_a). Table 5 of Noordin et al (2004).

Source	Sum of Squares	df	Mean Square	F-value	p-value	Remarks
Model	9.47	4	2.37	62.07	< 0.0001	significant
B	8.82	1	8.82	231.11	< 0.0001	
C	0.037	1	0.037	0.9755	0.3445	
C^2	0.45	1	0.45	11.85	0.0055	
BC	0.24	1	0.24	6.22	0.0298	
Residual	0.42	11	0.038			
Lack of Fit	0.42	10	0.042	209.7	0.0537	not significant
Pure Error	0.0002	1	0.0002			
Cor Total	9.89	15				
S.D.	0.20		R^2	0.9576		
Mean	2.20		Adjusted R^2	0.9421		
C.V. %	8.87		Predicted R^2	0.8976		
PRESS	1.01		Adeq Precisior	22.35		

Table 8.23: Resulting ANOVA table (partial sum of squares) for reduced quadratic model (response: tangential force, F_c). Table 6 of Noordin et al (2004).

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	7.86E+04	5	1.57E+04	168.78	< 0.0001	significant
A	482.40	1	482.4	5.18	0.0462	
B	7.62E+04	1	7.62E+04	817.91	< 0.0001	
C	104.33	1	104.33	1.12	0.3149	
C^2	1668.17	1	1668.17	17.9	0.0017	
BC	485.13	1	485.13	5.21	0.0457	
Residual	931.9	10	93.19			
Lack of Fit	905.84	9	100.65	3.86	0.3769	not significant
Pure Error	26.06	1	26.06			
Cor Total	79575.63	15				
S.D.	9.65		R^2	0.9883		
Mean	452.18		Adjusted R^2	0.9824		
C.V. %	2.13		Predicted R^2	0.9714		
PRESS	2279		Adeq Precisior	35.91		

For R_a, from Table 8.22, only terms B, C, BC, and C^2 were statistically significant at the 5% level. Using these terms, the regression equations for the surface roughness, R_a, in terms of coded and actual factors were given in Equations 1 and 3 of the paper, respectively. They are:

Coded factors:

$$R_a = 1.97 + 0.94\ B + 0.06\ C^2 + 0.17\ BC$$

Actual factors:

$$R_a = -2.714 + 22.228\ \text{feed} + 2.88 \times 10^{-4}\ \text{SCEA} + 0.0583\ \text{SCEA}^2 + 1.372\ \text{feed} \times \text{SCEA}$$

The model for R_a has an adjusted R^2 of 0.9421 and predicted R^2 of 0.8976.

For F_c, from Table 8.23, the statistically significant terms were A, B, C, C^2 and BC. Although C was not statistically significant at the 5% level, it was included because BC and C^2 were in the model. The regression models for the tangential force, Fc, in terms of coded and actual factors, were given in Equations 2 and 4 of the paper, respectively. They are:

Coded factors:

$$F_c = 437.96 - 6.93\ A + 87.39\ B + 3.23\ C + 22.15\ C^2 + 7.76\ BC$$

Actual factors:

$$F_c = 57.237 - 0.103\ \text{cutting speed} + 1902.95\ \text{feed} + 4.737\ \text{SCEA} + 3.543\ \text{SCEA}^2 + 62.05\ \text{feed} \times \text{SCEA}$$

The model for F_c has an adjusted R^2 of 0.9824 and predicted R^2 of 0.9714.

To validate the models, the authors completed another six confirmation runs using different combinations of the three factors. The experimental and model results were compared in Table 7 of the paper and are reproduced here as Table 8.24.

Table 8.24: Confirmation experiments. Table 7 of Noordin et al (2004).

No.	SCEA	Feed	Cutting speed	Surface roughness				Tangential force			
				Actual R_a	Predicted R_a	Residual	Error (%)	Actual F_c	Predicted F_c	Residual	Error (%)
1	-3	0.18	300	1.13	1.07	0.06	5.31	356.6	353.13	3.47	0.97
2	-3	0.23	375	2.06	1.98	0.08	3.88	440.78	431.26	9.52	2.16
3	-5	0.28	375	2.98	3.04	-0.06	-2.01	528.21	529.57	-1.37	-0.26
4	0	0.28	300	3.46	3.51	-0.05	-1.45	569.37	559.25	10.12	1.78
5	-3	0.18	375	1.16	1.07	0.09	7.76	343.56	345.42	-1.87	-0.54
6	-5	0.28	300	2.91	3.04	-0.13	-4.47	545.81	537.28	8.53	1.56

The authors concluded that the proposed models provided reasonably accurate predictions when compared to the experimental results.

Case Study #8.7

Qian, Fuping and Mingyao Zhang (2005): Study of the natural vortex length of a cyclone with response surface methodology. Computers and Chemical Engineering, 29, pp. 2155-2162.

This study investigated the natural vortex length, an important parameter of a cyclone, as a function of various geometries of a cyclone using response surface methodology. The natural vortex length of cyclones were obtained by numerical simulations using a commercial computational fluid dynamics (CFD) code, Fluent 6.1. A face-centered central composite design (FCD) was used to obtain the run combinations to fit a second-order regression model to the natural vortex lengths. Five factors, all in dimensionless form, were used in the experiment. The theory behind cyclones and how the dimensionless terms were derived were given in the paper. The factors and levels used in the FCD were shown in Table 1 of the paper and are reproduced here as Table 8.25.

Table 8.25: Low and high level settings of the factors used in the response surface model (Hoekstra, 2000). Table 1 of Qian et al (2005).

Factor	x_i	X_{iL}	X_{iH}
De/D	X_1	0.30	0.70
a/D	X_2	0.30	0.80
b/D	X_3	0.15	0.35
(h-S)/D	X_4	0.50	2.50
ln Re	X_5	10.2	12.9

In Table 8.25, *De* is the diameter of the cyclone vortex finder, *D* is the diameter of the cyclone body, *a* is the height of the cyclone inlet, *b* is the width of the cyclone inlet, *h* is the length of the cyclone cylinder, *S* is the deepness of the vortex finder insertion, and *Re* is the well-known dimensionless Reynold's number. All these terms were described in a schematic drawing of the cyclone in the paper. Dividing by *D*, all factors involving length become dimensionless. The response was the natural vortex length which was also expressed in dimensionless form as *l/D*.

Minitab 14 software was used for designing and analysing the data. The FCD had 43 runs in total which consisted of 32 full-factorial points, 10 axial points, and one center point since this was a computer-based experiment with no random errors. The experimental design and results were shown in Table 2 of the paper and are reproduced here as Table 8.26.

The authors fitted a full second-order regression model to the dimensionless vortex length (*l*/*D*) data and the breakdown of the analysis of variance in terms of linear, squared, and interaction terms was shown in Table 3 of the paper is shown here as Table 8.27. The estimated regression coefficients and test statistics for the coded factors were shown in Table 4 of the paper. The coefficients in terms of actual factors (uncoded units) were also shown in Table 5 of the paper. These tables are reproduced as Tables 8.28 and 8.29, respectively.

Table 8.26: Central composite design of Y = *l*/D. Table 2 of Qian et al (2005).

No.	X_1	X_2	X_3	X_4	X_5	Y
1	0.3	0.3	0.15	0.5	10.20	5.000
2	0.7	0.3	0.15	0.5	10.20	4.010
3	0.3	0.8	0.15	0.5	10.20	4.300
4	0.7	0.8	0.15	0.5	10.20	2.950
5	0.3	0.3	0.35	0.5	10.20	4.200
6	0.7	0.3	0.35	0.5	10.20	3.850
7	0.3	0.8	0.35	0.5	10.20	3.500
8	0.7	0.8	0.35	0.5	10.20	3.000
9	0.3	0.3	0.15	2.5	10.20	4.750
10	0.7	0.3	0.15	2.5	10.20	4.500
11	0.3	0.8	0.15	2.5	10.20	4.550
12	0.7	0.8	0.15	2.5	10.20	4.300
13	0.3	0.3	0.35	2.5	10.20	3.950
14	0.7	0.3	0.35	2.5	10.20	3.750
15	0.3	0.8	0.35	2.5	10.20	4.000
16	0.7	0.8	0.35	2.5	10.20	3.800
17	0.3	0.3	0.15	0.5	12.90	5.675
18	0.7	0.3	0.15	0.5	12.90	4.950
19	0.3	0.8	0.15	0.5	12.90	4.950
20	0.7	0.8	0.15	0.5	12.90	4.650
21	0.3	0.3	0.35	0.5	12.90	5.500
22	0.7	0.3	0.35	0.5	12.90	5.300
23	0.3	0.8	0.35	0.5	12.90	4.750
24	0.7	0.8	0.35	0.5	12.90	4.400
25	0.3	0.3	0.15	2.5	12.90	5.600
26	0.7	0.3	0.15	2.5	12.90	5.300
27	0.3	0.8	0.15	2.5	12.90	5.250
28	0.7	0.8	0.15	2.5	12.90	4.500
29	0.3	0.3	0.35	2.5	12.90	5.300
30	0.7	0.3	0.35	2.5	12.90	4.925
31	0.3	0.8	0.35	2.5	12.90	4.850
32	0.7	0.8	0.35	2.5	12.90	4.695
33	0.5	0.55	0.25	1.5	11.55	5.240
34	0.3	0.55	0.25	1.5	11.55	5.150
35	0.7	0.55	0.25	1.5	11.55	4.850
36	0.5	0.3	0.25	1.5	11.55	5.300
37	0.5	0.8	0.25	1.5	11.55	5.100
38	0.5	0.55	0.15	1.5	11.55	5.200
39	0.5	0.55	0.35	1.5	11.55	4.700
40	0.5	0.55	0.25	0.5	11.55	5.250
41	0.5	0.55	0.25	2.5	11.55	5.200
42	0.5	0.55	0.25	1.5	10.20	4.750
43	0.5	0.55	0.25	1.5	12.90	5.650

Table 8.27: Analysis of variance for *l*/D. Table 3 of Qian et al (2005).

Source	d.f.	Seq SS	Adj SS	Adj MS	F-ratio	P-value
Regression	20	20.8458	20.8458	1.04229	43.22	0.000
Linear	5	13.6016	13.6016	2.72032	112.8	0.000
Square	5	5.7557	5.7557	1.15114	47.73	0.000
Interaction	10	1.4884	1.4884	0.148844	6.17	0.000
Residual error	22	0.7958	0.7958	0.0362		
Total	42	21.6416				

8.28: Estimated regression coefficients for *l*/D (coded factors). Table 4 of Qian et al (2005).

Term	Coefficient	S.E. coeff	T-ratio	P-value
Constant	5.24721	0.03628	144.628	0.000
De/D	-0.22191	0.02663	-8.332	0.000
a/D	-0.24456	0.02663	-9.183	0.000
b/D	-0.17544	0.02663	-6.588	0.000
(h-S)/D	0.08779	0.02663	3.297	0.002
ln Re	0.50250	0.02663	18.868	0.000
De/D x De/D	-0.25803	0.09866	-2.615	0.013
a/D x a/D	-0.05803	0.09866	-0.588	0.560
b/D x b/D	-0.30803	0.09866	-3.120	0.004
(h-S)/D x (h-S)/D	-0.03303	0.09866	-0.335	0.740
ln Re x ln Re	-0.05803	0.09866	-0.588	0.560
De/D x a/D	-0.01453	0.02745	-0.529	0.600
De/D x b/D	0.08078	0.02745	2.943	0.006
De/D x (h-S)/D	0.07141	0.02745	2.601	0.014
De/D x ln Re	0.02922	0.02745	1.064	0.295
a/D x b/D	0.01734	0.02745	0.632	0.532
a/D x (h-S)/D	0.12047	0.02745	4.388	0.000
a/D x ln Re	-0.02797	0.02745	-1.019	0.316
b/D x (h-S)/D	-0.04672	0.02745	-1.702	0.098
b/D x ln Re	0.09859	0.02745	3.591	0.001
(h-S)/S x ln Re	-0.07953	0.02745	-2.897	0.007
S = 0.1553, R-sq = 96.3%, T-sq (Adj) = 94.1%				

The second-order regression model using the coefficients of Table 8.29 was given as Equation 6 of the paper. This equation is:

$$
\begin{aligned}
Y = \frac{l}{D} = &-3.59538 + 2.70570\, X_1 + 0.249231\, X_2 + 3.51131\, X_3 + 0.540555\, X_4 \\
&+ 1.00495\, X_5 - 6.45064\, X_1^2 - 0.928410\, X_2^2 - 30.8026\, X_3^2 - 0.0330256\, X_4^2 \\
&- 0.0318385\, X_5^2 - 0.290625\, X_1X_2 + 4.03906\, X_1X_3 + 0.357031\, X_1X_4 \\
&+ 0.108218\, X_1X_5 + 0.693750\, X_2X_3\; 0.481875\, X_2X_4 - 0.082870\, X_2X_5 \\
&- 0.0467188\, X_3X_4 + 0.730324\, X_3X_5 - 0.0589120\, X_4X_5
\end{aligned}
$$

Table 8.29: Estimated regression coefficients for *l*/*D* using data in uncoded units. Table 5 of Qian et al (2005).

Term	Coefficient
Constant	-3.595380
De/D	2.705700
a/D	0.249231
b/D	3.511310
(h-S)/D	0.540555
ln Re	1.004950
De/D x De/D	-6.450640
a/D x a/D	-0.928410
b/D x b/D	-30.802600
(h-S)/D x (h-S)/D	-0.033026
ln Re x ln Re	-0.031839
De/D x a/D	-0.290625
De/D x b/D	4.039060
De/D x (h-S)/D	0.357031
De/D x ln Re	0.108218
a/D x b/D	0.693750
a/D x (h-S)/D	0.481875
a/D x ln Re	-0.082870
b/D x (h-S)/D	-0.467188
b/D x ln Re	0.730324
(h-S)/S x ln Re	-0.058912

Attempts to reproduce the exact results obtained in the above tables were not successful using Minitab 18 or Design-Expert 12. The goodness-of-fit obtained using the given data is lower but the statistically significant terms in the quadratic model are similar to those reported by the authors. It seems likely that the analysis by the authors was done using a different set of data than those in Table 8.26.

Even if the results were correct, it was not clear why the authors included all terms in the model. Many of the terms were not statistically significant at the 5% level.

Case Study #8.8

Sin, H. N., S. Yusof, N. Sheikh Abdul Hamid, and R. Abd. Rahman (2006): Optimization of enzymatic clarification of sapodilla juice using response surface methodology. Journal of Food Engineering, 73, pp. 313-319.

This study investigated the effect of three factors – incubation time, temperature, and enzyme concentration on the enzymatic clarification of sapodilla juice using response surface methodology. Sapodilla, a tropical fruit normally eaten fresh, can be made into a juice but requires the use of enzymes to clarify the juice. The optimal conditions for producing clarified juice was the primary objective of the study.

A face-centered central composite design (FCD) with five center points was used. The factors and levels used in the experiment are summarized in Table 8.30. The materials and methods used in the experiment, including the preparation of the fruit, the extraction of the juice, and the treatment by enzymes, were explained in detail in the paper.

Table 8.30: Factors and levels used in the experiment.

		Levels		
Factors	Units	Low (-1)	Center (0)	High (+1)
Time, X_1	min	30	75	120
Temperature, X_2	°C	30	40	50
Enzyme concentration, X_3	%	0.03	0.065	0.10

The authors considered four responses as measures of quality of the extracted juice after treatment by the various combinations of the factors. They were the turbidity (NTU), clarity (abs), viscosity (cps), and the colour, based on L values.

The FCD had a total of 19 runs consisting of eight factorial points, six axial points, and five center points. The experiment was run in random order. The 19 run combinations in coded and actual factors, and the results for the four responses, were shown in Table 1 of the paper and are reproduced here as Table 8.31. ECHIP Software Version 6 (Echip Inc., Hockessin, Delaware, USA) was used for the design and analysis of the experiment.

Note that in Table 1 of the paper, all five center points were given the same treatment number of 15. However, it is more conventional to give each treatment combination its own run or treatment number. This was done in Table 8.31. Hence the five center points are labelled as treatment 15, 16, 17, 18, and 19.

The authors fitted a full second-order model (linear, two-factor interaction, and squared terms) to each of the responses. The estimated coefficients, different level of statistical significance, and goodness-of-fit statistics were shown in Table 2 of the paper and are reproduced here as Table 8.32. The coefficients were estimated using actual factors.

Table 8.31: Effects of incubation time, temperature and enzyme concentration on four dependent variables. Table 1 of Sin et al (2006).

	Indepdent variables						Dependent variables			
	Coded variables			Uncoded variables			Turbidity	Clarity	Viscosity	L
Treatment	X_1	X_2	X_3	Time (min)	Temp. (°C)	Enzyme conc. (%)	(NTU)	(abs)	(cps)	value
15	0	0	0	75	40	0.065	16.60	0.03	1.39	54.55
1	-1	0	0	30	40	0.065	34.40	0.03	1.40	52.15
9	1	-1	1	120	30	0.1	22.30	0.03	1.25	53.77
16	0	0	0	75	40	0.065	16.20	0.02	1.35	54.01
10	-1	-1	1	30	30	0.1	19.00	0.02	1.39	53.17
7	1	1	1	120	50	0.1	18.80	0.01	1.34	54.89
17	0	0	0	75	40	0.065	17.00	0.03	1.40	54.21
3	0	-1	0	75	30	0.065	18.10	0.03	1.42	54.01
8	-1	1	1	30	50	0.1	29.90	0.03	1.34	54.00
18	0	0	0	75	40	0.065	15.00	0.02	1.38	54.42
12	-1	1	-1	30	50	0.03	65.30	0.07	1.40	49.36
5	0	0	-1	75	40	0.03	49.70	0.05	1.45	51.73
19	0	0	0	75	40	0.065	14.60	0.03	1.40	54.38
11	1	1	-1	120	50	0.03	43.20	0.05	1.39	51.88
13	1	-1	-1	120	30	0.03	52.10	0.05	1.43	51.59
2	1	0	0	120	40	0.065	21.10	0.02	1.28	54.11
6	0	0	1	75	40	0.1	12.20	0.02	1.25	54.25
4	0	1	0	75	50	0.065	14.80	0.02	1.41	54.10
14	-1	-1	-1	30	30	0.03	68.90	0.08	1.46	48.10

8.32: Regression coefficients and R^2 value for four dependent variables for enzymatic clarified sapodilla juices. Table 2 of Sin et al (2006).

Regression coefficient	Turbidity (NTU)	Clarity (abs)	Viscosity (cps)	L value
b_0	16.449484	0.023128	1.377732	54.223608
b_1	-0.133333*	-0.000124**	-0.000667***	0.021022*
b_2	-0.084000	-0.000315	-0.000700	0.035900***
b_3	505.714303*	-0.534857*	-1.600000*	49.771423*
b_1^2	0.005229*	0.000002	-0.000015	-0.000484**
b_2^2	-0.007113	0.000031	0.000451	-0.000556
b_3^2	11256.049479*	10.132549**	-16.242336	-914.790594*
b_{12}	-0.005472**	-0.000004	0.000044	-0.000189
b_{13}	2.468254*	0.002548***	-0.007937	-0.358730**
b_{23}	7.107141**	0.000393	0.050000	0.142856
R^2	0.966	0.964	0.829	0.978
p	0.0000	0.0000	0.0138	0.0000

Subscripts: 1 = incubation time; 2 = temperature, 3 = enzyme concentration.

* Significant at 0.0001 level.

** Significant at 0.01 level.

***Significant at 0.05 level.

The full ANOVA results were not given in the paper. In addition, the adjusted and predicted R^2 values were not reported.

Reanalyses of data using Minitab 18 and Design-Expert 12 produced identical R^2 values as reported in the paper and the estimated coefficients were also identical for only the squared and two-factor interaction terms but the linear and intercept terms were quite different. Furthermore, for viscosity, the predicted R^2 = -0.5642 which indicates that the overall mean may be a better predictor than the second-order model.

For the optimization phase of the study, it was not clear whether the full second-order models were used or only the statistically significant terms were used in the models. The optimum combination of factors to give the lowest turbidity, absorbance value (clarity), and viscosity, while colour (L value) is as high as possible were: incubation time of 120 mins, temperature of 40°C, and 0.1% enzyme concentration. The optimum combination was obtained by a graphical approach.

Case Study #8.9

Tabaraki, Reza and Ashraf Nateghi (2011): Optimization of ultrasonic-assisted extraction of natural antioxidants from rice bran using response surface methodology. Ultrasonic Sonochemistry, 18, pp. 1279-1286.

This study used response surface methodology to optimize the conditions for the extraction of polyphenols and antioxidants from rice bran with ethanol as a food grade solvent. An ultrasonic-assisted extraction (UAE) method was used. Three factors – solvent percentage, temperature, and time were considered and four responses – total phenolic content (TPC), ferric reducing antioxidant power (FRAP), scavenging activity of 1,1-diphenyl-2-picrylhydrazyl (DPPH) radical, and the extraction yields were measured. The materials and methods used in the experiment were described in the paper. The factors and levels used were shown in Table 1 of the paper and are reproduced here in Table 8.33.

Table 8.33: Coded and uncoded levels of the independent variables. Table 1 of Tabaraki et al (2011).

Independent variables	Coded units	Coded levels		
		-1	0	1
Ethanol concentration, E (%)	X_1	50	70	90
Temperature, T (°C)	X_2	40	50	60
Time, t (min)	X_3	15	30	45

The experimental design was based on a face-centered central composite design (FCD) with a total of 16 runs consisting eight factorial points, six axial points, and two center points. Minitab 15 was used for experimental design and subsequent statistical analysis and modelling. The experimental design and responses measured were shown in Table 2 of the paper and are reproduced here as Table 8.34.

Table 8.34: Central composite design of three variables with their observed responses. Table 2 of Tabaraki et al (2011).

Exp No.	X_1	X_2	X_3	E%	T (°C)	t (min)	TPC (mg/GA/g dw)	FRAP (μmol Fe^{2+}/g dw)	DPPH (%)	Yield (%)
1	1	1	1	90	60	45	4.07	42.11	28.99	20.2
2	1	-1	1	90	40	45	2.37	31.74	16.88	18.4
3	-1	-1	-1	50	40	15	3.51	36.61	25.09	11.0
4	0	-1	0	70	40	30	4.72	46.33	34.81	15.6
5	0	0	0	70	50	30	6.29	56.76	55.61	17.5
6	-1	1	1	50	60	45	4.44	44.10	29.60	17.8
7	1	-1	-1	90	40	15	2.94	33.98	17.41	13.8
8	0	0	-1	70	50	15	5.07	50.98	43.66	16.2
9	0	0	1	70	50	45	5.21	51.70	45.42	18.2
10	-1	1	-1	50	60	15	3.78	39.42	27.09	15.1
11	-1	0	0	50	50	30	4.52	46.36	33.79	14.6
12	1	1	-1	90	60	15	3.66	38.10	26.20	19.0
13	1	0	0	90	50	30	3.24	35.68	22.71	18.6
14	-1	-1	1	50	40	45	4.79	47.36	36.04	14.0
15	0	0	0	70	50	30	6.35	57.23	49.25	17.4
16	0	1	0	70	60	30	5.11	48.20	40.29	18.8

A full second-order regression model consisting of linear, quadratic, and two-factor interaction terms was fitted to each of the responses. The estimated coefficients and their statistical significance were shown in Table 3 of the paper and are reproduced here as Table 8.35.

Table 8.35: Regression coefficients of predicted polynomial models for the investigated responses from rice bran extracts. Table 3 of Tabaraki et al (2011).

Coefficient	Response			
	TPC	FRAP	DPPH	Yield
β_0	-19.11	-156.45*	265.65*	-23.54*
β_1	0.43*	3.17	4.66**	0.40*
β_2	0.32	3.63	5.66	0.54
β_3	0.11	0.52	0.51	0.30*
β_{11}	-0.36×10^{-2}**	-0.26×10^{-1}**	-0.39×10^{-1}**	-0.21×10^{-2}**
β_{22}	-0.41×10^{-2}	-0.24	-0.64×10^{-1}	-0.22×10^{-2}
β_{33}	-0.08×10^{-2}	0.01×10^{-2}	0.03×10^{-1}	-0.10×10^{-2}
β_{12}	0.16×10^{-2}	0.09×10^{-1}	0.16×10^{-1}	-0.06×10^{-2}
β_{13}	-0.09×10^{-2}	-0.06×10^{-1}	-0.05×10^{-1}	0.01×10^{-3}
β_{23}	0.03×10^{-2}	0.01×10^{-1}	-0.04×10^{-1}	-0.31×10^{-2}
Model	*	*	*	***
Linear	*	*	*	*
Quadratic	**	**	**	*
Cross-product	ns	ns	ns	ns
Lack of fit	ns	ns	ns	ns
R^2	0.89	0.90	0.91	0.98

n.s., Not significant ($p>0.05$). * Significant at $p \le 0.05$, ** Significant at $p \le 0.01$, *** Significant at $p \le 0.001$.

Using the estimated coefficients Table 8.35, the following regression models (in actual factors) for TPC, FRAP, DPPH and Yield (Equations 3, 4, 5, and 6, respectively) were proposed for each of the responses, as shown:

$$\text{TPH (mg GA/g dw)} = -19.11 + 0.43E + 0.32T + 0.11t - 0.36 \times 10^{-2} E^2 - 0.41 \times 10^{-2} T^2 - 0.08 \times 10^{-2} t^2 + 0.16 \times 10^{-2} E.T - 0.09 \times 10^{-2} E.t + 0.3 \times 10^{-2} T.t$$

$$\text{FRAP } (\mu\text{mol } Fe^{2+} \text{/g dw}) = -156.45 + 3.17E + 3.63T + 0.52t - 0.03 E^2 - 0.04 T^2 - 0.1 \times 10^{-2} t^2 + 0.09 \times 10^{-1} E.T - 0.06 \times 10^{-1} E.t + 0.1 \times 10^{-2} T.t$$

$$\text{DPPH (\%)} = -265.65 - 4.66E + 5.66T + 0.51t - 0.04E^2 - 0.06T^2 + 0.03 \times 10^{-1} t^2 - 0.04 \times 10^{-1} T.t + 0.06 \times 10^{-1} E.T - 0.05 \times 10^{-1} E.t$$

$$\text{Yield (\%)} = -23.54 - 0.40E + 0.54T + 0.30t - 0.21 \times 10^{-2}E^2 - 0.22 \times 10^{-2} T^2 - 0.01 \times 10^{-1} t^2 - 0.31 \times 10^{-2} T.t - 0.06 \times 10^{-2} E.T + 0.01 \times 10^{-2} E.t$$

From Table 8.35, it can be seen that many of the estimated coefficients were not statistically significant at the 5% level yet they were included in the regression models. The full ANOVA results and full set of goodness-of-fit statistics were not given in the paper. On reanalysis of the data, the predicted R^2 values are actually quite low. The predicted R^2 for TPC, FRAP, DPPH, and Yield are 0.227, 0.146, 0.502, and 0.625, respectively.

The optimal ultrasonic-assisted extraction (UAE) conditions were identified as 65-67% ethanol, 51-54 °C, and 40-45 min. It was not mentioned how these values were obtained. The authors then compared the predicted values with actual values at the optimal condition for each of the responses. These comparisons were shown in Table 4 of the paper and are reproduced here as Table 8.36.

8.36: Estimated optimum conditions, predicted and experimental values of responses under these conditions. Table 4 of Tabaraki et al (2011).

Response variables	Optimum UAE conditions			Maximum values	
	Ethanol (%)	Temp (°C)	Time (min)	Predicted	Actual
TPC (mg GA/g dw)	67	54	40	6.05	6.21
FRAP (μmol Fe^{2+}/g dw)	65	51	45	54.14	54.94
DPPH (%)	67	51	45	52.83	52.65
Yield (%)	87	60	28	19.93	19.83

The condition for the simultaneous optimization of all four responses was not given in the paper.

Case Study #8.10

Wee Shin Ling, Tye Ching Thian, and Subhash Bhatia (2010): Process optimization studies for the dehydration of alcohol-water system by inorganic membrane based pervaporation separation using design of experiments (DOE). Separation and Purification Technology, 71, pp. 192-199.

This study investigated the effects of feed temperature, feed concentration, permeate pressure, and feed flow rate on membrane separation performance using response surface methodology based on a face-centered central composite design. A commercial ceramic membrane from Pervatech BV was used to study the dehydration of alcohol-water mixture by pervaporation. Isopropanol-water and ethanol-water mixtures were used in this study. Pervaporation apparently has many advantages over conventional membrane separation technologies. The theory behind pervaporation and the experimental setup were described in the paper.

The four factors used in the experiment and their levels were shown in Table 1 of the paper and are reproduced here as Table 8.37.

Table 8.37: Experimental independent variables. Table 1 of Wee et al (2010).

Variables	Factor code	Unit	Level and range (coded)		
			-1	0	+1
Temperature	A	°C	60	75	90
Isopropanol feed concentration	B	kg/kg	0.80	0.88	0.96
Permeate pressure	C	kPA	1	3	5
Feed flow rate	D	dm^3/h	40	70	90

Two responses were measured – permeation flux (kg/m^2 h) and selectivity. Their definitions and calculations were given in the paper.

Design-Expert 6.06 was used for the design and analysis of the experiment. The experimental design used was a face-centered central composite design (FCD) with a total of 30 runs consisting a full 2^4 factorial of 16 runs, eight axial points, and six center points. The authors added three additional replicated runs but these were not used in the statistical analysis. The experimental design and the measured responses were shown in Table 2 of the paper and are reproduced here as Table 8.38. The experimental runs were done in random order.

Full second-order regression models (consisting of linear, quadratic, and two-factor interaction terms) were fitted to each of the responses. The ANOVA results for permeation flux and selectivity were shown in Tables 3 and 4 of the paper, respectively. These tables are reproduced here as Tables 8.39 and 8.40, respectively.

Table 8.38: Experiment runs and responses for the pervaporation of isopropanol aqueous solutions. Table 2 of Wee et al (2010).

Run	Factor				Response 1	Response 2
	A: temperature (°C)	B: isopropanol feed concentration (kg/kg)	C: permeate pressure (kPa)	D: Feed flow rate (dm^3/h)	Permeation flux (kg/m^2 h)	Selectivity
1	90	0.88	3	70	6.61	177
2	90	0.80	1	100	9.55	145
3	60	0.96	5	40	0.62	607
4	60	0.96	1	40	0.44	1476
5	90	0.96	5	100	1.91	243
6	60	0.80	5	100	2.99	145
7	60	0.80	1	40	2.69	136
8	60	0.80	1	100	2.72	294
9	60	0.80	5	40	2.33	126
10	75	0.88	3	70	4.44	540
11	75	0.88	3	100	4.7	412
12	75	0.88	3	70	4.75	362
13	90	0.96	5	40	1.76	216
14	75	0.80	3	70	4.21	503
15	90	0.80	5	100	7.89	192
16	75	0.88	3	70	3.88	557
17	90	0.96	1	100	1.74	951
18	75	0.88	5	70	5.14	362
19	75	0.88	3	70	3.95	396
20	75	0.88	1	70	4.64	698
21	75	0.88	3	40	3.94	497
22	90	0.80	5	40	5.67	147
23	90	0.96	1	40	1.8	990
24	60	0.88	3	70	2.95	131
25	90	0.80	1	40	6.19	171
26	75	0.96	3	70	1.12	835
27	60	0.96	5	100	0.95	551
28	60	0.96	1	100	0.83	1346
29	75	0.88	3	70	4.7	393
30	75	0.88	3	70	3.8	296
Repeated runs						
31	60	0.88	3	70	3.21	130
32	60	0.88	3	70	3.15	138
33	60	0.88	3	70	3.42	129
Mean					3.27	132
Standard deviation					0.12	4.08

Table 8.39: Analysis of variance (ANOVA) for 2^4 full center composite design (CCD) for permeation flux. Table 3 of Wee et al (2010).

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	134.77	14	9.63	33.12	< 0.0001	significant
A-A	39.31	1	39.31	135.24	< 0.0001	
B-B	60.76	1	60.76	209.03	< 0.0001	
C-C	0.10	1	0.10	0.34	0.5667	
D-D	3.41	1	3.41	11.75	0.0037	
A^2	0.25	1	0.25	0.87	0.3665	
B^2	8.42	1	8.42	28.98	< 0.0001	
C^2	0.46	1	0.46	1.59	0.227	
D^2	0.06	1	0.06	0.20	0.6647	
AB	12.60	1	12.60	43.36	< 0.0001	
AC	0.32	1	0.32	1.10	0.3112	
AD	1.13	1	1.13	3.90	0.0669	
BC	0.46	1	0.46	1.57	0.2297	
BD	1.86	1	1.86	6.41	0.023	
CD	0.01	1	0.01	0.03	0.8697	
Residual	4.36	15	0.29			
Lack of Fit	3.44	10	0.34	1.87	0.2529	not significant
Pure Error	0.92	5	0.18			
Cor Total	139.13	29				

Table 8.40: Analysis of variance (ANOVA) for 2^4 full center composite design (CCD) for selectivity. Table 4 of Wee et al (2010).

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	3.49×10^6	14	2.49×10^5	26.50	< 0.0001	significant
A-A	1.39×10^5	1	1.39×10^5	14.73	0.0016	
B-B	1.59×10^6	1	1.59×10^6	169.32	< 0.0001	
C-C	7.27×10^5	1	7.27×10^5	77.26	< 0.0001	
D-D	420.50	1	420.50	0.04	0.8354	
A^2	2.10×10^5	1	2.1×10^5	22.30	0.0003	
B^2	1.38×10^5	1	1.38×10^5	14.61	0.0017	
C^2	21646.01	1	21646.01	2.30	0.1502	
D^2	655.30	1	655.30	0.07	0.7955	
AB	1.47×10^5	1	1.47×10^5	15.63	0.0013	
AC	8281.00	1	8281.00	0.88	0.3631	
AD	16.00	1	16.000	0.00	0.9677	
BC	5.66×10^5	1	5.66×10^5	60.16	< 0.0001	
BD	9702.25	1	9702.25	1.03	0.3261	
CD	324.00	1	324.00	0.03	0.8553	
Residual	1.41×10^5	15	9412.32			
Lack of Fit	88066.82	10	8806.68	0.83	0.6269	not significant
Pure Error	53118.00	5	10623.60			
Cor Total	3.63×10^6	29				

The authors then proposed the following prediction equations for the permeation flux and selectivity in terms of coded factors (Equations 4 and 5, respectively):

$$\text{Permeation flux} = 4.36 + 1.48\ A - 1.84\ B - 0.07\ C + 0.44\ D + 0.31193\ A^2 - 1.80\ B^2 + 0.42\ C^2 - 0.15\ D^2 - 0.89\ AB - 0.14\ AC + 0.27\ AD + 0.17\ BC - 0.34\ BD - 0.02\ CD$$

$$\text{Selectivity} = 431.3 - 87.78\ A + 297.56\ B - 201\ C - 4.83\ D - 284.60\ A^2 + 230.40\ B^2 + 91.40\ C^2 + 15.90\ D^2 - 95.88\ AB + 22.75\ AC + AC - 188.13\ BC - 24.63\ BD + 4.5\ CD$$

In the paper, only the R^2 values were given for the full model. For permeation flux, the R^2 was 0.9687, and for selectivity, the R^2 was 0.9611. Adjusted and predicted R^2 values were not given. As can be seen from Tables 8.39 and 8.40, many of the regression coefficients were not statistically significant at the 5% level but were included in the prediction models. Model performance could be improved by using reduced quadratic models with only statistically significant terms.

To obtain the optimal condition of the four factors, the authors used the numerical optimization tool available in Design-Expert 6.0.6 which is based on the desirability function approach. They considered three different sets of goals to obtain three optimal combination of the four factors. The full results were shown in Table 5 of the paper and are summarized here as Table 8.41.

Table 8.41: Optimum condition for three different set of goals from DOE for the pervaporation of isopropanol aqueous solution. Summarized from Table 5 of Wee et al (2010).

No.	Temperature (°C)	Isopropanol feed concentration (kg/kg)	Permeate pressure (kPa)	Feed flow rate (dm^3/h)	Permeation flux (kg/m^2 h)	Selectivity	Desirability
Goal 1: maximize selectivity and permeation flux ≥ 2.5 kg/m^2 h							
1*	75	0.94	1.00	84.00	2.50	1191	0.8883
Goal 2: maximize permeation flux							
1*	90	0.81	1.00	100.00	8.81	-	0.9188
Goal 3: maximize selectivity							
1*	69	0.96	1.02	41.05	-	1491	1.0000

* selected as optimium point as suggested by the software.

Additional experiments with isopropanol and ethanol aqueous solution were also carried out to check the accuracy of the optimum condition suggested by the software. The experimental results were given in Table 6 of the paper. The authors indicated that the error of the predicted results using the developed regression models were ±4% for permeation flux and ±5% for selectivity and concluded that the statistical analysis is reliable to optimize the pervaporation process. Other conclusions were given in the paper.

9. COMBINATION DESIGNS

This Chapter presents seven case studies of combination designs. These are designs that start with either a 2-level factorial or 2-level fractional factorial design, followed up with an RSM design. The RSM designs were either the rotatable CCD or face-centered CCD.

Case Study #9.1

Chen, X-C., Jian-Xin Bai, Jia-Ming Cao, Zhen-Jiang Li, Jian Xiong, Lei Zhang, Yuan Hong, and Han-Jie Ying (2009): Medium optimization for the production of cyclic adenosine 3', 5'-monophosphate by *Microbacterium* sp. no. 205 using response surface methodology. Bioresource Technology, 100, pp. 919-924.

This study employed a 2^{11-7} fractional factorial design followed by a rotatable central composite design to optimize the medium components that affect the production of cyclic adenosine 3', 5'-monophosphate (cAMP) with Microbacterium sp. no. 205. The importance of cAMP was explained in detail by the authors. Eleven factors affecting cAMP production were investigated first to evaluate the main effects only. The factors and levels investigated are given in Table 1 of the paper and are reproduced here as Table 9.1 in a slightly modified form.

Table 9.1: Factors and levels tested in the experiment. Modified from Table 1 in Chen et al (2009).

	Levels of factors		
Factor	-1	0	1
Glucose (X_1, g/L)	40	50	60
K_2HPO_4 (X_2, g/L)	5	10	15
KH_2PO_4 (X_3, g/L)	5	10	15
$MgSO_4$ (X_4, g/L)	5	10	15
Urea (X_5, g/L)	5	10	15
Biotin (X_6, g/L)	2	3	4
$CoCl_2$ (X_7, mg/L)	5	10	15
NaF (X_8, g/L)	0.05	0.10	0.15
Peptone (X_9, g/L)	3	4	5
Hypoxanthine (X_{10}, g/L)	2	3	4
Initial pH (X_{11})	7.0	7.5	8.0

In Table 1 of the paper, the levels were listed in order of +1, 0, and -1. In Table 9.1, they were listed from -1 to +1 which is more conventional. Design-Expert version 6.0 was used for the design and analysis of the experiments. The authors did not mention the defining relationship used for the fractional factorial design but a good guess is that they used the default in Design-Expert. The design was a resolution III design. Four center points were added to the design to give a total of 20 runs.

The run combinations and responses obtained from the fractional factorial experiment were shown in Table 2 of the paper and are reproduced in Table 9.2.

Table 9.2: Experimental design and results of the 2^{11-7} fractional factorial design. Table 2 of Chen et al (2009).

												cAMP (g/L)	
Std Order	X_1	X_2	X_3	X_4	X_5	X_6	X_7	X_8	X_9	X_{10}	X_{11}	Observed	Predicted
1	-1	-1	-1	-1	-1	-1	-1	-1	1	1	1	1.97	2.43
2	1	-1	-1	-1	1	-1	1	1	-1	-1	-1	4.15	4.77
3	-1	1	-1	-1	1	1	-1	1	-1	-1	1	5.61	5.30
4	1	1	-1	-1	-1	1	1	-1	1	1	-1	3.32	3.16
5	-1	-1	1	-1	1	1	1	-1	-1	1	-1	3.59	4.01
6	1	-1	1	-1	-1	1	-1	1	1	-1	1	2.68	3.25
7	-1	1	1	-1	-1	-1	1	1	1	-1	-1	7.96	7.88
8	1	1	1	-1	1	-1	-1	-1	-1	1	1	1.83	1.91
9	-1	-1	-1	1	-1	1	1	1	-1	1	1	4.87	4.95
10	1	-1	-1	1	1	1	-1	-1	1	-1	-1	0.97	0.89
11	-1	1	-1	1	1	-1	1	-1	1	-1	1	3.28	3.85
12	1	1	-1	1	-1	-1	-1	1	-1	1	-1	4.10	4.52
13	-1	-1	1	1	1	-1	-1	1	1	1	-1	6.18	6.02
14	1	-1	1	1	-1	-1	1	-1	-1	-1	1	1.46	1.15
15	-1	1	1	1	-1	1	-1	-1	-1	-1	-1	2.72	3.34
16	1	1	1	1	1	1	1	1	1	1	1	4.63	5.09
17	0	0	0	0	0	0	0	0	0	0	0	4.69	3.91
18	0	0	0	0	0	0	0	0	0	0	0	4.70	3.91
19	0	0	0	0	0	0	0	0	0	0	0	4.72	3.91
20	0	0	0	0	0	0	0	0	0	0	0	4.73	3.91

The authors fitted a first order model with only main effects and the results of the regression analysis were shown in Table 3 of the paper and are reproduced here as Table 9.3. The predicted values using only the main effect are also shown in Table 9.2.

The authors then selected only the three statistically significant effects at the 5% level for the next optimization stage of the experiment. The effects selected were: K_2HPO_4 (X_2), $MgSO_4$ (X_4), and NaF (X_8). There was no mention of any lack of fit test or checking for curvature in the paper. Upon reanalysis of the data, it seems that the coefficients, t-values, and corresponding p-values for X_1 and X_4 were incorrectly reported in the paper. The values have been interchanged. That is, the values for X_4 belong to X_1 and vice versa. This means that X_1 should have been chosen and not X_4. The correct factors should have been: X_1, X_2, and X_8.

Table 9.3: Regression results of the fractional factorial design. Table 3 of Chen et al (2009).

Factor	Coefficient	t-Value	P-value
Intercept	3.9080	22.0111	0.000000*
X_1	-0.1812	-0.9130	0.387887
X_2	0.4737	2.3866	0.044088*
X_3	0.1737	0.8753	0.406921
X_4	-0.8150	-4.1057	0.003411*
X_5	0.0725	0.3652	0.724405
X_6	-0.1587	-0.7997	0.446958
X_7	0.4500	2.2669	0.053141
X_8	1.3150	6.6245	0.000165*
X_9	0.1662	0.8375	0.426612
X_{10}	0.1037	0.5226	0.615371
X_{11}	-0.4162	-2.0969	0.069266

$R^2 = 0.9084$. * Statistically significant at 95% confidence level

For the optimization stage of the experiment, the authors performed a path of steepest ascent to determine the proper levels for the central composite design. This was given in Table 4 of the paper. The final levels used for the three chosen factors from the fractional factorial design were shown in Table 5 of the paper and are reproduced here in Table 9.4.

Table 9.4: Levels of the factors tested in the central composite design (modified from Table 5 of Chen et al, 2009).

Factor	Levels of factors				
	-1.68	-1	0	1	1.68
K_2HPO_4 (A, g/L)	9.64	11	13	15	16.36
$MgSO_4$ (B, g/L)	0.64	2	4	6	7.36
NaF (C, g/L)	0.032	0.1	0.2	0.3	0.368*

In Table 5 of the paper, the axial point of factor C was incorrectly listed as 0.268.

The central composite design with two center points has 16 runs. The design and the experimental results were shown in Table 6 of the paper and are reproduced here in Table 9.5. A full second-order model was then fitted to the data resulting in the following prediction equation:

$$Y = 8.44 - 0.15\,A - 0.29\,B - 0.17\,C - 0.60\,A^2 - 0.58\,B^2 - 0.48\,C^2 - 0.26\,AB + 0.20\,AC \\ + 0.035\,BC$$

The equation gave a R^2 of 0.9556. The regression results were given in Table 8 of the paper and are given here as Table 9.6. The predicted values are also shown in Table 9.5.

Table 9.5: Experimental design and results of the central composite design. Table 6 of Chen et al (2009).

				cAMP (g/L)	
Std Order	A (K_2HPO_4)	B ($MgSO_4$)	C (NaF)	Observed	Predicted
1	-1	-1	-1	7.41	7.37
2	1	-1	-1	6.61	6.56
3	-1	1	-1	7.54	7.24
4	1	1	-1	6.6	6.57
5	-1	-1	1	7.32	7.19
6	1	-1	1	7.05	7.18
7	-1	1	1	6.15	6.02
8	1	1	1	6.3	6.15
9	-1.68	0	0	6.84	6.99
10	1.68	0	0	6.43	6.49
11	0	-1.68	0	7.35	7.29
12	0	1.68	0	6.04	6.31
13	0	0	-1.68	7.12	7.37
14	0	0	1.68	6.84	6.8
15	0	0	0	8.49	8.44
16	0	0	0	8.43	8.44

Table 9.6: Regression results of the Central composite design. Table 8 of Chen et al (2009).

Factor	Coefficient	P-value
Intercept	8.4400	
A	-0.1500	0.0619
B	-0.2900	0.0040*
C	-0.1700	0.0391*
A^2	-0.6000	0.0003*
B^2	-0.5800	0.0003*
C^2	-0.4800	0.0009*
AB	-0.2600	0.0238*
AC	0.2000	0.0541
BC	0.0350	0.6941

Notice that several of the statistically insignificant terms were included in the prediction equation by the authors. It is also likely that the coefficients and associated p-values for AB and BC have been interchanged. Hence AB should have a coefficient of 0.035 and is not statistically significant, while BC should have a coefficient of -0.26 and is statistically significant.

The authors then used the prediction equation to maximize cAMP. They found that with A = -0.11, B = -0.24, and C = -0.21, all coded units gave a cAMP value of about 8.50 g/L which was about a 1.8 fold increase when compared to using the original medium.

Despite the wrong choice of factors from the fractional factorial design and wrong coefficients used, the final results seem to be still better than without using design of experiment methodologies!

Case Study #9.2

Chua, Y-T, Abdul Rahman Mohamed, and Subhash Bhatia (2007): Process optimization of oxidative coupling of methane for ethylene production using response surface methodology. Journal of Chemical Technology and Biotechnology, 82, pp. 81-91.

A combination of a half fractional factorial design with five factors followed up by a face-centered central composite design (FCD) with four factors was used to study the oxidative coupling of methane (OCM) over a Na-W-Mn/SiO_2 catalyst. The importance of studying this particular process was explained in the paper. The goal of the study was to optimize the yield of C_{2+}.

In the first part of the study, the authors considered five process factors: temperature, gas hourly space velocity (GHSV), pre-treatment time, dilution ratio, and the methane-oxygen (CH_4/O_2) ratio. The factors, units, and levels used were given in Table 1 of the paper and are reproduced here as Table 9.7.

Table 9.7: Factors and levels used in the experiment (after Table 1 of Chua et al, 2007).

Factors	Unit	Factor code	Low Level (-1)	High level (+1)
Temperature	C	A	750	850
GHSV	$cm^3/g/h$	B	18000	35000
Pre-treatment time	h	C	0	2
Dilution ratio		D	0.2	0.5
CH_4/O_2 ratio		E	3	7

Three responses were measured: methane conversion, selectivity of C2+ product, and ethylene/ethane ratio. Experimental design and analyses were conducted using Design-Expert version 6.0.6. A significance level of 5% was adopted for significance testing.

To save on experimental runs, the authors chose a half fractional factorial (2^{5-1}) design as a screening design. They replicated 2 runs for reproducibility giving a total of 18 runs. The fractional factorial design and the corresponding responses obtained were shown in Table 2 of the paper and are reproduced here as Table 9.8. The authors did not indicate what defining relationship was used to obtain the half fraction. It is also not clear what the design of the half factorial resolution was. It is clear that it was not the default design in Design-Expert.

Table 9.8: Experimental matrix of $2^{(5-1)}$ fractional factorial design. Table 2 of Chua et al (2007).

Factors						Responses		
Run	A	B	C	D	E	CH_4 Conversion (%)	C_{2+} Selectivity (%)	C_2H_4/C_2H_6 ratio
1	850	35000	2	0.5	7	40.43	62.09	1.64
2	850	35000	0	0.2	7	54.45	52.72	2.07
3	750	35000	2	0.5	7	19.96	34.38	0.44
4	750	18000	0	0.2	3	35.96	13.90	1.28
5	850	18000	0	0.2	3	31.93	8.31	1.20
6	750	35000	2	0.2	3	30.98	57.18	1.09
7	750	18000	0	0.5	7	34.77	55.11	0.86
8	850	18000	2	0.5	3	45.94	30.76	3.25
9	850	18000	0	0.5	7	23.34	47.68	1.76
10	750	35000	0	0.2	3	15.08	25.05	1.47
11	750	18000	2	0.5	3	41.83	2.79	1.76
12	850	35000	2	0.2	3	29.66	45.48	2.37
13	850	18000	2	0.5	7	47.46	29.70	2.12
14	750	18000	2	0.5	7	33.48	37.55	0.71
15	850	35000	0	0.2	3	45.86	13.87	1.20
16	750	35000	0	0.2	7	31.68	63.75	0.79
17	750	35000	0	0.2	7	32.61	65.75	0.79
18	750	35000	0	0.2	7	31.63	66.28	0.80

The last two runs were considered repeat tests for reproducibility checking. They were not used in the ANOVA or regression models.

For methane conversion, the authors fitted a linear model shown in Equation 4 of the paper, given by (in coded factors):

$$\begin{aligned} CH_4 \text{ conversion (\%)} = {} & 35.24 + 1.24\,A - 3.44\,B + 2.69\,C - 3.42\,D + 2.30\,E + 5.16\,AB - 0.96\,AC \\ & + 1.69\,AD + 0.23\,AE - 4.23\,BC + 2.53\,BE - 2.89\,ABE + 7.06\,ACD \end{aligned}$$

The authors stated that the selected effects in the equation were all statistically insignificant although AB, BC, and ACD have greater effects over the model. It is not clear why the authors included three-factor interaction terms in the model. These effects are usually ignored in practice. It is likely that the selected model is flawed.

For the selectivity of C_{2+} product, the ANOVA results are shown in Table 3 of the paper and are reproduced here in Table 9.9. The authors then suggested the following equation to represent C_{2+} selectivity (Equation 5 of the paper):

$$\begin{aligned} C_{2+} \text{ selectivity (\%)} = {} & 28.11 + 0.056\,A + 5.74\,B + 3.52\,C - 4.61\,D + 13.91\,E - 3.69\,AB + 9.04\,AD \\ & - 4.40\,AE + 12.41\,BC - 16.33\,BD + 5.48\,BE + 4.83\,ABE \end{aligned}$$

The ANOVA results show that only factor A (temperature) was not statistically significant at the 5% level.

Table 9.9: ANOVA table of C_{2+} selectivity. Table 3 of Chua et al (2007).

Source	Sum of Squares	d.f.	Mean square	F-value	Prob> F
Model	5805.62	12	483.80	418.79	0.0002
A	0.05	1	0.05	0.04	0.8476
B	263.70	1	263.70	228.26	0.0006
C	99.40	1	99.40	86.05	0.0027
D	84.92	1	84.92	73.50	0.0033
E	1547.07	1	1547.07	1339.16	<0.0001
AB	145.04	1	145.04	125.55	0.0015
AD	653.41	1	653.41	565.60	0.0002
AE	206.51	1	206.51	178.75	0.0009
BC	1232.06	1	1232.06	1066.49	<0.0001
BD	1066.35	1	1066.35	923.05	<0.0001
BE	240.35	1	240.35	208.05	0.0007
ABE	372.68	1	372.68	322.60	0.0004
Residual	3.47	3	1.16		
Corr. Total	5809.08	15			

The authors examined the two-factor interaction plots and concluded that interactions between factors AB, AE, and BE were not statistically significant in this study and hence the temperature factor was dropped from the next phase of experimentation. For the C_2H_4/C_2H_6 ratio, the authors did not provide any ANOVA results or model. However, some explanation regarding this response was given in the paper.

From the initial fractional factorial results, the authors concluded that the ANOVA was dominated by the C_{2+} selectivity response and that the temperature variable could be fixed at 850 °C for the next round of experimentation. For the optimization study, the authors chose a four factor face-centered central composite design (FCD) with six center points. The levels were the same ones used in the fractional factorial design. The FCD required 16 runs for the factorial points, 8 runs for the axial points, and 6 center-points. Hence a total of 30 experiments were conducted. An additional response, called the C_{2+} yield (%) was added. (This can be calculated from methane conversion and C_{2+} selectivity; details are given in the paper.)

The FCD and responses obtained are shown in Table 4 of the paper and are reproduced here in Table 9.10. The key goal was to maximize the C_{2+} yield hence the focus was on analysing this response. The authors did not show the ANOVA tables for any of the responses analysed. Only the prediction equation in coded factors for C_{2+} yield was given. This is Equation 6 of the paper, given by:

$$C_{2+} \text{ yield (\%)} = 20.63 + 2.34\,A + 2.28\,B - 0.53\,C + 4.30\,D - 1.75\,AB + 0.87\,AC - 0.30\,AD - 5.19\,BC - 0.70\,BD - 2.09\,CD.$$

No goodness of fit statistics were given and there was no indication of whether all the terms were statistically significant.

Table 9.10: Experimental matrix of 2^4 full factorial with central composite design. Table 4 of Chua et al (2007).

	Factors				Responses			
Run	GHSV ($cm^3/g/h$)	Pretreat time (h)	Dilution ratio	CH_4/O_2 ratio	CH_4 Conv (%)	C_{2+} select (%)	C_{2+} yield (%)	C_2H_4/C_2H_6 ratio
1	18000	0	0.2	3	32.78	9.02	2.96	1.20
2	35000	0	0.2	3	46.54	14.97	6.97	1.25
3	18000	2	0.2	3	44.63	51.44	22.96	2.72
4	35000	2	0.2	3	43.99	47.09	20.71	2.37
5	18000	0	0.5	3	43.62	25.85	11.28	2.76
6	35000	0	0.5	3	46.78	65.71	30.74	2.07
7	18000	2	0.5	3	36.12	32.72	11.82	3.25
8	35000	2	0.5	3	49.37	30.80	15.21	2.13
9	18000	0	0.2	7	25.61	57.13	14.63	1.15
10	35000	0	0.2	7	54.45	52.72	28.71	2.07
11	18000	2	0.2	7	42.01	77.12	32.40	1.53
12	35000	2	0.2	7	43.18	73.75	31.85	1.32
13	18000	0	0.5	7	42.49	62.15	26.41	1.76
14	35000	0	0.5	7	35.40	70.73	25.04	1.15
15	18000	2	0.5	7	48.11	31.61	15.21	2.12
16	35000	2	0.5	7	38.54	59.29	22.85	1.64
17	18000	1	0.35	5	37.42	65.42	24.48	1.93
18	35000	1	0.35	5	37.96	58.66	22.27	1.48
19	26500	0	0.35	5	20.74	60.58	12.56	1.20
20	26500	2	0.35	5	41.83	65.50	27.40	1.30
21	26500	1	0.2	5	41.78	61.76	25.80	1.49
22	26500	1	0.5	5	30.66	61.72	18.92	1.39
23	26500	1	0.35	3	46.84	51.10	23.94	1.63
24	26500	1	0.35	7	36.66	73.56	26.97	1.20
25	26500	1	0.35	5	25.60	67.98	17.40	1.99
26	26500	1	0.35	5	27.94	71.82	20.07	1.68
27	26500	1	0.35	5	33.10	56.92	18.84	1.85
28	26500	1	0.35	5	27.01	69.27	18.71	1.42
29	26500	1	0.35	5	25.61	67.42	17.27	1.75
30	26500	1	0.35	5	23.91	64.94	15.53	1.63

The authors then compared the results obtained by the DOE method with those obtained using an incipient wetness impregnation, and mixture slurry methods. They found that the results were almost identical.

Case Study #9.3

Fontes, G. C., Priscilla Filomena Fonseca Amaral, Marcio Nele, and Maria Alice Zarur Coelho (2010): Factorial design to optimize biosurfactant production by *Yarrowia lipolytica.* Journal of Biomedicine and Biotechnology, Volume 2010, Article 821306, 8 pages.

This study investigated the improvement of biosurfactant production by *Yarrowia lipolytica* IMUFRJ 50682 using a two-level factorial designs followed by a central composite design. Biosurfactants have advantages over synthetic ones because of their high specificity and biodegradability. They have applications in many industries, ranging from food to petrochemicals. However, biosurfactants are expensive to produce. Therefore any means to improve production at lower cost is welcomed.

This study evaluated two separate sources for biosurfactant production - a nitrogen source and a carbon source. For the nitrogen source study, four factors were investigated using a 2-level full factorial design with 4 factors and 3 center points. A total of 19 run combinations were required. STATISTICA version 7.0 software was used for regression and graphical analyses of the data. The details of the experiments were given in the paper.

The factors and levels used were shown in Table 1 of Fontes et al (2010) and are reproduced here as Table 9.11.

Table 9.11: Factors and levels used in the 2^4 full factorial design for the nitrogen source study. Table 1 of Fontes et al (2010).

		Level		
Factor	Units	-1	0	1
Peptone (x_1)	g/L	0	6.4	12.8
Yeast extract (x_2)	g/L	5	10	15
Ammonium sulfate (x_3)	g/L	0	5	10
Urea (x_4)	g/L	0	0.1	0.2

Two responses were measured for both studies. These were the maximum variation of surface tension (ST), in mN/m and the emulsification index (EI), in percentage. The exact definition of these responses were explained in the paper.

For the carbon source study, the same full factorial design was used except with factors and levels shown in Table 2 of the paper and are reproduced here as Table 9.12.

Table 9.12: Factors and levels used in the 2^4 full factorial design for the carbon source study (Table 2 of Fontes et al, 2010).

		Level		
Factor	Units	-1	0	1
Glycerol (z_1)	% w/v	0	1	2
Olive oil (z_2)	% w/v	0	2	4
Hexadecane (z_3)	%w /v	0	1	2
Glucose (z_4)	% w/v	0	2	4

The run combinations and results for the nitrogen source experiment were given in Table 3 of the paper and reproduced here as Table 9.13.

Table 9.13: Experimental design and results of the 2^4 full factorial design for nitrogen source evaluation (Table 3 of Fontes et al, 2010)

Std Order	x_1	x_2	x_3	x_4	ST	EI
1	-1	-1	-1	-1	6.5	37.3
2	1	-1	-1	-1	14.5	26.1
3	-1	1	-1	-1	5.1	27
4	1	1	-1	-1	4	1
5	-1	-1	1	-1	16.6	45.3
6	1	-1	1	-1	22	52.2
7	-1	1	1	-1	19.5	40.6
8	1	1	1	-1	13	50
9	-1	-1	-1	1	7.5	35.7
10	1	-1	-1	1	6	30.6
11	-1	1	-1	1	9.3	13
12	1	1	-1	1	4.6	6.2
13	-1	-1	1	1	15.2	40.3
14	1	-1	1	1	16.3	50.1
15	-1	1	1	1	21.1	60.4
16	1	1	1	1	9.4	43.2
17	0	0	0	0	13	26
18	0	0	0	0	11.4	24
19	0	0	0	0	11	25.4

For the nitrogen source study, the authors used a Pareto chart to display the ranked order of the effects and which effects were statistically significant. For the emulsification index (EI), the order of significant effects was x_3, x_2x_3, x_2, x_1x_3, x_1x_2, and x_1. Other effects were not statistically significant at the 5% level. The regression equation is thus:

$$EI = 33.4 - 2.5\, x_1 - 4.7\, x_2 + 12.8\, x_3 - 2.5\, x_1x_2 + 3.6\, x_1x_3 + 5.5\, x_2x_3$$

For the variation in surface tension (ST), the order of the significant effects were x_3, x_1x_2, x_1x_4, and x_2. Other effects were not statistically significant. The regression equation is given by:

$$ST = 11.9 - 1.16\, x_2 + 4.7\, x_3 - 2.3\, x_1x_2 - 1.4\, x_1x_4.$$

These equations are collectively known as Equation 1 in the paper.

Notice the equation for ST is not hierarchical as x_4 is missing. The R^2 values for EI and ST were reported as 0.87 and 0.86, respectively.

Table 9.14: Experimental design and results of the 2^4 full factorial design for carbon source evaluation. Table 3 of Fontes et al (2010).

Std Order	z_1	z_2	z_3	z_4	ST	EI
1	-1	-1	-1	-1	9	6.9
2	1	-1	-1	-1	19.5	62.3
3	-1	1	-1	-1	2	11.6
4	1	1	-1	-1	11	25.5
5	-1	-1	1	-1	2.6	12.8
6	1	-1	1	-1	10.7	70.2
7	-1	1	1	-1	2	27.5
8	1	1	1	-1	9.9	39.3
9	-1	-1	-1	1	27.8	56.2
10	1	-1	-1	1	20.2	82.9
11	-1	1	-1	1	14.4	48.8
12	1	1	-1	1	14.8	40
13	-1	-1	1	1	14.9	47.3
14	1	-1	1	1	12.6	76.8
15	-1	1	1	1	14.4	38.9
16	1	1	1	1	13	26
17	0	0	0	0	10.9	42.3
18	0	0	0	0	9.8	43.1
19	0	0	0	0	11.2	43.9

For the carbon source study, Pareto charts showed that for EI, the ordered significant effects were z_1, z_1z_2, z_4, z_2, z_1z_4, z_3z_4, and z_2z_3. Others were not statistically significant at the 5% level. For ST, the ordered significant effects were z_4, z_1z_4, z_3, z_2, z_2z_3, and z_1. Other effects were not statistically significant at the 5% level. According to the authors, although z_2 (Olive oil) and z_3 (Hexadecane) were statistically significant, they present a negative effect. Hence z_1 (glycerol) and x_4 (glucose) were considered the best substrates to increase biosurfactant production. The authors then performed another set of experiments using only z_1 and z_4 but in a rotatable CCD design with an additional response - the carbon to nitrogen (C/N) ratio. The factors and levels used in the CCD was shown in Table 5 of the paper and are reproduced here as Table 9.15.

Table 9.15: Coded and actual levels of the two variables in the CCD. Table 5 of Fontes et al, 2010).

		Level				
Factor	Units	-1.41	-1	0	1	1.41
Glycerol (z_1)	% w/v	0.59	1	2	3	3.41
Glucose (z_4)	% w/v	1.17	2	4	6	6.83

The CCD had eight runs plus three center points giving a total of 11 combinations. The design and results of the three responses are shown in Table 6 of the paper and are reproduced here as Table 9.16.

Table 9.16: Experimental design and results of the CCD. Table 6 of Fontes et al (2010).

Std Order	z_1	z_4	C/N	EI	ST
1	-1	-1	5.9	51.4	11.8
2	1	-1	10.4	66.2	13.6
3	-1	1	13.3	60.8	12.9
4	1	1	17.8	67.3	16
5	-1.41	0	8.7	54.1	12.1
6	1.41	0	15.1	57.8	15
7	0	-1.41	6.6	61.4	12.4
8	0	1.41	17.1	73.7	16.2
9	0	0	11.9	81.8	19.5
10	0	0	11.9	81.1	19
11	0	0	11.9	80.9	20.1

The authors fitted a second-order regression to the responses and obtained the following equations for EI and ST. This set of equations is Equation 2 in the paper, shown below:

$$EI = 81.3 + 4.8\, z_1 - 6.9\, z_1^2 + 1.9\, z_4 - 12.7\, z_4^2 - 2.0\, z_1 z_4$$

$$ST = 19.5 + 1.2\, z_1 - 2.6\, z_1^2 + 0.9\, z_4 - 3.0\, z_4^2$$

No ANOVA results were given but the R^2 for both equations were given as 0.99.

The above mathematical models were validated by using mean values at the central points. The authors stated that the experimental EI and ST values perfectly agreed with the predicted maximum.

Case Study #9.4

He, Z., Ya-Juan Han, Shuang Zhao, and Sung H. Park (2009): Product and process optimisation through Design of Experiments: A case study. Total Quality Management, Vol. 20, No. 1, pp 107-113.

This case study considered the application of DOE techniques to optimize the design of a fused biconical taper wavelength multiplexer that is used in communication devices. The major goal of the research was to increase isolation - a measure of crosstalk between communication channels; the larger the isolation, the higher the transmission quality, leading to higher profits. The authors first considered six factors in a 2^{6-2} resolution IV design to screen for significant factors. The factors and levels are shown in Table 9.17 which is adapted from Table 1 of the paper.

Table 9.17: Factors and their levels in the experiment. Adapted from Table 1 of He et al (2009).

Factor	Name	Low level	High level
A	Flame height	207.0	213.0
B	Hydrogen flux	52.0	69.0
C	Taper speed	1.0	1.3
D	Optic distribution ratio	74.5	78.0
E	Humidity	0.4	0.6
F	Temperatrure	19.0	25.0

The defining relationship used for this quarter fraction design was I=ABCE=BCDF=ADEF. Table 9.18 shows the run combinations with three center points. Hence total number of runs was 19. The response was the isolation, Y. No units were given for the factors or response.

The statistical analyses were carried out using Minitab (no version given). The initial analysis by the authors did not find any statistically significant factors when curvature was not taken into account in the ANOVA. However, when curvature was taken into account, the curvature term was highly statistically significant. This was shown in Table 5 of the paper and is reproduced here in Table 9.19.

Table 9:18: Experimental arrangement for the 2^{6-2}_{IV} (coded variables). Table 2 of He et al (2009)

Std order	Run order	A	B	C	D	E	F	Y
1	10	-1	-1	-1	-1	-1	-1	21.4
2	3	1	-1	-1	-1	1	-1	19.4
3	14	-1	1	-1	-1	1	1	22.2
4	2	1	1	-1	-1	-1	1	22.3
5	6	-1	-1	1	-1	1	1	24.5
6	7	1	-1	1	-1	-1	1	20.1
7	9	-1	1	1	-1	-1	-1	19.5
8	4	1	1	1	-1	1	-1	20.8
9	16	-1	-1	-1	1	-1	1	20.6
10	17	1	-1	-1	1	1	1	18.9
11	13	-1	1	-1	1	1	-1	23.4
12	18	1	1	-1	1	-1	-1	23.6
13	8	-1	-1	1	1	1	-1	22.6
14	5	1	-1	1	1	-1	-1	18.7
15	15	-1	1	1	1	-1	1	23.5
16	12	1	1	1	1	1	1	23.7
17	19	0	0	0	0	0	0	26.5
18	11	0	0	0	0	0	0	24.8
19	1	0	0	0	0	0	0	27.2

Table 9.19: Estimated effects and coefficients for Y (coded units). Table 5 of He et al (2009).

Term	Effect	Coeff	SE Coeff	T	P
Constant		21.575	0.2275	94.85	0.000*
A	-1.2750	-0.6375	0.2275	-2.8	0.049*
B	1.6000	0.8000	0.2275	3.52	0.025*
Constant	0.2000	0.1000	0.2275	0.44	0.683
D	0.6000	0.3000	0.2275	1.32	0.258
E	0.7250	0.3625	0.2275	1.59	0.186
F	0.8000	0.4000	0.2275	1.76	0.153
A*B	1.7250	0.8625	0.2275	3.79	0.019*
A*C	-0.4250	-0.2125	0.2275	-0.93	0.403
A*D	-0.0250	-0.0125	0.2275	-0.05	0.959
A*E	-1.2000	-0.6000	0.2275	-2.64	0.058
A*F	-1.7500	-0.8750	0.2275	-0.38	0.720
B*D	1.7500	0.8750	0.2275	3.85	0.018*
B*F	0.3000	0.1500	0.2275	0.66	0..546
Ct. Pt.		4.5917	0.5725	8.02	0.001*
S=0.909899, R-sq = 97.01%, R-sq (adj) = 86.54%					

Note: *means that the P-value is less than 0.05, and the corresponding term is significant.

The authors then considered factors A, B and D only for a follow up experiment using rotatable central composite design (CCD) with six center points to model the curvature. These individual factors were either statistically significant or were involved with two-factor interactions that were significant. The new set of experiments with A, B, and D are shown in Table 9.20.

A second-order model was then fitted to the new set of data from the rotatable CCD. The estimated regression coefficients for the full model are shown in Table 7 of the paper. After removing the non-significant two-factor interaction terms, and maintaining hierarchy, the final estimated regression coefficients for Y in coded units were calculated and shown in Table 9 of the paper and reproduced here as Table 9.20.

Table 9:20: Experimental arrangement for the CCD (coded units). Table 6 of He et al (2009).

Std order	Run order	A	B	D	Y
1	8	-1	-1	-1	21.2
2	7	1	-1	-1	21.4
3	5	-1	1	-1	21.7
4	6	1	1	-1	22.3
5	15	-1	-1	1	22.4
6	19	1	-1	1	21.9
7	1	-1	1	1	22.1
8	12	1	1	1	20.6
9	3	-1.68179	0	0	21.7
10	17	1.68179	0	0	21.3
11	2	0	-1.68179	0	19.2
12	18	0	1.68179	0	21.4
13	4	0	0	-1.68179	20.4
14	11	0	0	1.68179	19.8
15	16	0	0	0	23.6
16	9	0	0	0	25.3
17	20	0	0	0	25.7
18	10	0	0	0	26.4
19	14	0	0	0	24.8
20	13	0	0	0	24.1

Table 9.21: Final estimated regression coefficients for Y (coded units). Table 9 of He et al (2009).

Term	Coeff	SE Coeff	T	P
Constant	24.9404	0.4396	56.737	0.000*
A	-0.1371	0.2916	-0.470	0.646
B	0.2563	0.2916	0.879	0.396
D	-0.0446	0.2916	-0.153	0.881
A*A	-0.9508	0.2839	-3.349	0.005*
B*B	-1.3750	0.2839	-4.843	0.000*
D*D	-1.4457	0.2839	-5.092	0.000*
S=1.078, R-sq = 80.0%, R-sq (adj) = 70.8%				

Note: *means that the P-value is less than 0.05, and the corresponding term is significant.

The final prediction equation in coded units is (Equation 1 of the paper):

$$\hat{y} = 24.9404 - 0.1371\, x_1 + 0.2563\, x_2 - 0.0446\, x_3 - 0.9508\, x_1^2 - 1.375\, x_2^2 - 1.4457 x_3^2$$

where: x_1 is the flame height, x_2 is the hydrogen flux, and x_3 is the optic distribution ratio. The optimum conditions were near the center points with $x_1 = -0.072$, $x_2 = 0.093$ and $x_3 = -0.015$. The overall optimum condition for all factors were A = 210, B = 61, C = 1.00, D = 76, E = 0.4, and F = 19.

According to the authors, the isolation was 20 before optimization and 24.9404 after optimization with a 95% confidence interval of (23.9907, 25.8900), which was confirmed by confirmation runs.

Case Study #9.5

Raj, R. E., and B.S.S. Daniel (2011): Customization of closed-cell aluminum foam properties using design of experiments. Material Science and Engineering A, 528, pp. 2067-2075.

This study investigated factors that would influence the properties of closed-cell aluminum foam using experiments based on a fractional factorial design followed by a face-centered central composite design. According to the authors, aluminum foams are useful for various applications due to their high energy absorbing capacity, light weight, and high specific stiffness. The characteristic properties of interest are relative density, average pore diameter, and the average cell aspect ratio. The process of foaming and measurement of properties were described in the paper.

In the first stage of the experiment, the authors considered four major influencing factors - holding time, amount of TiH_2, amount of calcium, and the stirring time after calcium addition. A half fraction of a two-level factorial design (2^{4-1}) with the four factors was used. The factors and levels are summarized in Table 9.22.

Table 9.22: Factors and levels used in the fractional factorial design

Factor	Name	Units	Low level (-1)	High level (+1)
A	Holding time	s	80	120
B	Amount of TiH_2	wt %	0.6	1.0
C	Amount of Ca	wt %	0.8	1.2
D	Stirring time for Ca	min	8	12

Design-Expert 7.1.3 was used for the experimental design and subsequent statistical analysis and modeling. The defining relationship for the 2^{4-1} design was not mentioned in the paper. It is assumed that the default in Design-Expert was used.

Three responses were measured - relative density, the average pore diameter (mm), and the cell aspect ratio.

The experimental design and results are shown in Table 1 of the paper and are shown here in Table 9.23.

Table 9.23: Phase I experimental design conditions and their corresponding structural properties of the closed-cell aluminum foam. Table 1 of Raj et al, 2011).

	Process parameters				Structural properties		
Exp No.	Holding time (A)	Amount of TiH_2 (B)	Amount of Ca (C)	Stirring time (D)	Relative density	Average pore diameter (mm)	Cell aspect ratio
1	80	0.6	0.8	8	0.267	2.883	1.019
2	120	0.6	0.8	12	0.266	3.120	1.364
3	80	1.0	0.8	12	0.176	3.063	1.042
4	120	1.0	0.8	8	0.196	3.137	1.072
5	80	0.6	1.2	12	0.225	3.020	1.054
6	120	0.6	1.2	8	0.305	2.923	1.040
7	80	1.0	1.2	8	0.157	3.274	1.061
8	120	1.0	1.2	12	0.179	3.300	1.200

The ANOVA results for the three responses were summarized by the authors in Table 2 of the paper and are reproduced here as Table 9.24.

Table 9.24: ANOVA summary and the model statistic result of the phase-I half fractional factorial design of experiments. Table 2 of Raj et al (2011).

Response	Model type	F-vlaue	p-value	Adj R-squared	Pred R-squared	Signal-to Noise ratio
Relative density	Factorial (R2FI)	36.43	0.0269	0.9620	0.8262	15.356
Average pore diameter	Factorial (R2FI)	119.25	0.0083	0.9883	0.9465	34.883
Cell aspect ratio	Factorial (R2FI)	11.38	0.0827	0.8811	0.4566	9.644

The 2^{4-1} design is a resolution IV design, hence only the main effects can be cleanly estimated. The two-factor interactions are aliased with other two-factor interactions. The authors used Pareto charts to display the effects and determined that only factor A (holding time) and factor B (amount of TiH_2) were considered as significant factors for further investigation. The exact form of the reduced two-factor interaction models in Table 9.24 was not mentioned.

For the second phase of the investigation, the authors chose a face-centered central composite design with five center points. The range for factor A was changed to 60 - 140 (instead of 80 - 100), and for factor B it was changed to 0.5 - 1.5 (instead of 0.6 - 1.0). The face-centered CCD had 8 factorial points and 5 center points. The new design and experimental results were shown in Table 3 of the paper and are reproduced here in Table 9.25.

The ANOVA results were summarized by the authors in Table 4 of the paper and are reproduced here in Table 9.26.

Table 9.25: Phase II experimental design conditions and their corresponding structural properties of the closed-cell aluminum foam. Table 3 of Raj et al (2011).

	Process parameters		Structural properties		
Exp No.	Holding time (A)	Amount of TiH_2 (B)	Relative density	Average pore diameter (mm)	Cell aspect ratio
1	60	0.5	0.351	2.186	1.008
2	100	0.5	0.337	2.240	1.091
3	140	0.5	0.286	3.325	1.180
4	60	1.0	0.159	2.559	1.010
5	100	1.0	0.148	3.200	1.087
6	140	1.0	0.123	3.407	1.120
7	60	1.5	0.102	3.204	1.101
8	100	1.5	0.095	3.644	1.025
9	140	1.5	0.080	4.486	1.259
10	100	1.0	0.107	3.139	1.063
11	100	1.0	0.108	2.931	1.085
12	100	1.0	0.116	3.232	1.107
13	100	1.0	0.100	3.251	1.084

Table 9.26: ANOVA summary and the model statistic result of the phase-II CCD. Table 4 of Raj et al v(2011).

Response	Model type	F-vlaue	p-value	Adj R-squared	Pred R-squared	Signal-to Noise ratio
Relative density	Quadratic	62.49	0.0001	0.9624	0.9168	21.664
Average pore diameter	Linear	52.07	0.0001	0.8949	0.8185	24.505
Cell aspect ratio	Linear	7.78	0.0092	0.5306	0.1194	8.093

From Table 9.26, a quadratic model fitted the relative density response well while the linear model fitted the other two responses. However, it was not mentioned in the paper which terms in the model were statistically significant and which were not. The authors presented the regression models for each of the responses. These were Equations 2, 3, and 4 in the paper. They are reproduced below:

$$\text{Relative density} = 0.8343 - 0.0019846\,A - 0.9453\,B + 0.00053\,AB + 0.0000047166\,A^2 + 0.3302\,B^2$$

$$\text{Average pore diameter (mm)} = 0.5785 + 0.0136\,A + 1.1944\,B$$

$$\text{Cell aspect ratio} = 1.0217 + 0.0014307\,A - 0.2810\,B - 0.00017375\,AB + 0.0000028632\,A^2 + 0.1829\,B^2$$

It is not clear why the regression equation for the cell aspect ratio was a quadratic instead of a linear model as shown in Table 9.26. No validation was done for any of the models.

Case Study #9.6

Vicente, G., Mercedes Martinez, and Jose Aracil (2007): Optimization of integrated biodiesel production. Part I. A study of the biodiesel purity and yield. Bioresource Technology, 98, pp. 1724-1733.

This study investigated the development and optimization of potassium hydroxide catalyzed synthesis of fatty acid methyl esters (biodiesel) from sunflower oil. The authors first started the experiments with a full factorial design with three factors and four center points. Then the design was augmented by adding star or axial points to model non-linearity observed in the responses. The factors and levels investigated were temperature, initial catalyst concentration, and methanol:vegetable oil molar ratio. The factors and levels used in the factorial experiment are summarized in Table 9.27.

Table 9.27: Factors and levels used in the biodiesel experiment.

Factor (coded)	Name	Unit	Low level (-1)	Mid level (0)	High Level (+1)
T (X_T)	Temperature	°C	25	45	65
C (X_C)	Initial catalyst concentration	% wt	0.5	1.0	1.5
MR (X_{MR})	Vegetable oil molar ratio		4.5	6.0	7.5

The responses were the biodiesel purity (P in % weight) and the biodiesel yield (Y in % weight). The experimental design and results were given in Table 1 of the paper and are reproduced here in Table 9.28.

9.28: Experiment matrix and results: factorial and centre points. Table 1 of Vicente et al (2007).

Run	T (°C)	C (% wt)	MR	X_T	X_C	X_{MR}	P (%wt)	Y (% wt)
1	65	1.5	7.5	1	1	1	99.95	89.42
2	65	1.5	4.5	1	1	-1	98.14	90.43
3	25	0.5	7.5	-1	-1	1	84.08	99.28
4	25	1.5	4.5	1	1	-1	97.00	97.17
5	65	0.5	7.5	-1	-1	1	97.50	98.32
6	25	1.5	7.5	-1	1	1	99.95	96.88
7	65	0.5	4.5	1	-1	-1	87.00	98.70
8	25	0.5	4.5	-1	-1	-1	77.29	96.96
9	45	1	6	0	0	0	99.80	98.40
10	45	1	6	0	0	0	99.72	97.94
11	45	1	6	0	0	0	99.38	98.63
12	45	1	6	0	0	0	99.44	97.48

The software to carry out the statistical analyses was not mentioned in the paper. The statistical tests were performed at a 5% significance level. The statistical model obtained for P (purity), in terms of coded units was:

$$P = 92.6137 + 3.0337\ X_T + 6.1462\ X_C + 2.7562\ X_{MR} – 2.7487\ X_TX_C + 0.3221\ X_TX_{MR}$$

$$-1.5662\ X_CX_{MR}\ \ (r^2 = 0.994)$$

And for the yield Y was:

$$Y = 95.8950 – 1.6775\ X_T – 2.4200\ X_C + 0.0800\ X_{MR} – 1.8725\ X_TX_C – 0.4275\ X_TX_{MR}$$

$$-0.4050\ X_CX_{MR}\ \ (r^2 = 0.995)$$

These were Equations 1 and 2 in the paper. Equations in terms of actual factors were also given in the paper.

For response P, the authors identified the effects T, C, MR, T.C, T.MR and C.MR as statistically significant. Curvature was also statistically significant. For response Y, statistically significant effects were T, C, T.C, and T.MR. The curvature was also statistically significant.

To model the nonlinear effect, the authors augmented the two-level factorial design by adding star or axial points at (+α and –α) to the design making it a rotatable central composite design with 18 runs. The α used was 1.68. It was not mentioned whether these new runs were put in a separate block. The additional star points were shown in Table 3 of the paper and are reproduced here as Table 9.29.

9.29: Experimental matrix and results: star points. Table 3 of Vicente et al (2007).

Run	T (°C)	C (% wt)	MR	X_T	X_C	X_{MR}	P (%wt)	Y (% wt)
13	78.63	1	6	+α	0	0	99.82	94.28
14	11.36	1	6	-α	0	0	92.11	97.37
15	45	1.84	6	0	+α	0	99.80	92.45
16	45	0.16	6	0	-α	0	72.50	97.94
17	45	1	8.52	0	0	+α	99.83	97.77
18	45	1	3.47	0	0	-α	96.44	83.82

The authors then fitted a full second-order model to the responses and obtained the following equations (in coded units) for P and for Y (Equations 5 and 6 in the paper):

$$P = 99.4978 + 2.7266\,X_T + 6.9623\,X_C + 3.5861\,X_{MC} - 0.8896\,X_T^2 - 2.7487\,X_T X_C + 0.3212\,X_T X_{MR} - 4.3598\,X_C^2 - 1.5662\,X_C X_{MR} - 2.3534\,X_{MR}^2 \quad (r^2 = 0.969)$$

$$Y = 98.1063 - 1.4510\,X_T - 2.0058\,X_C + 0.2985\,X_{MC} - 0.7808\,X_T^2 - 2.0225\,X_T X_C - 0.5775\,X_T X_{MR} - 1.0036\,X_C^2 - 0.2550\,X_C X_{MR} - 0.3283\,X_{MR}^2 \quad (r^2 = 0.964)$$

Equations in terms of actual factors were also given in the paper. However there is no indication which of the regression coefficients were statistically significant.

Optimization of purity and yield are assumed to have used the quadratic equations developed. The optimal values of the variables were temperature at 25 °C, a 1.3% wt catalyst concentration, and a 6:1 methanol:sunflower oil molar ratio. Using these values the predicted purity was 100% wt and yield was 98.4 %wt.

Case Study #9.7

Zhou, J., Yong-Hong Wang, Ju Chu, Ling-Zhi Luo, Ying-Ping Zhuang, and Si-Liang Zhang (2009): Optimization of cellulose mixture for efficient hydrolysis of steam-exploded corn stover by statistically designed experiments. Bioresource Technology, 100, pp. 819-825.

In this study, statistically designed experiments were used to optimize the composition of cellulose mixture to maximize the amount of glucose produced from a steam-exploded corn stover - the purpose being to improve enzymatic hydrolytic efficiency and reduce production cost. A half fraction six-factor fractional factorial (2^{6-1}) design with 32 runs and four center points was first used as a screening experiment. This was followed up with a rotatable central composite design with only four factors identified from the screening experiment. The six factors and their levels used in the screening experiment were shown in Table 2 of the paper and are reproduced here with slight modifications as Table 9.30. The choice of factors and experimental methods used were described in the paper. Design-Expert Version 6.0.4 software was used for the design and analysis of the experiments.

Table 9.30: Factors and code values of fractional factorial design. Table 2 of Zhou et al (2009).

Factors	Code	Levels of factors (μmol/l)		
		-1	0	+1
Cel7A	x_1	1	3	5
Cel6A	x_2	2	6	10
Cel6B	x_3	0.5	1.0	1.5
Cel7B	x_4	0.5	2.5	4.5
Cel12A	x_5	1.5	2.25	3
Cel61A	x_6	0.3	0.5	0.7

The screening experiment was a resolution VI design meaning all main effects are aliased with five factor interactions and all two-factor interactions are aliased with four-factor interactions. This allows a full two-factor interaction model to be fitted without bias. The default defining relationship in Design-Expert was used to generate the 2^{6-1} fractional factorial design. The design and experimental results were shown in Table 1 of the paper and are reproduced here as Table 9.31. The response measured was the glucose production in mg/ml.

The authors fitted a two-factor interaction model to the experimental data and produced the following regression equation (Equation 3 in the paper). All terms were used in the model regardless of their significance.

$$\begin{aligned}\text{Glucose (mg/ml)} = {} & 4.875 + 0.665\ x_1 + 0.643\ x_2 + 0.291\ x_3 + 0.427\ x_4 + 0.427\ x_5 + 0.313\ x_6 \\ & + 0.119\ x_1x_2 + 0.153\ x_1x_3 - 0.0056\ x_1x_4 - 0.051\ x_1x_4 + 0.063\ x_2x_5 \\ & - 0.074\ x_2x_6 + 0.142\ x_3x_4 + 0.51\ x_3x_5 + 0.256\ x_3x_6 - 0.085\ x_4x_5 \\ & + 0.074\ x_4x_6 - 0.016\ x_5x_6\end{aligned}$$

The predicted values using the above equation were also given in Table 9.31. The estimated coefficients and their corresponding F and p-values were given in Table 3 in the paper and are reproduced here in Table 9.32. The full ANOVA results were not reported.

From Table 9.32, many of the effects were not statistically significant at the 5% level. No other goodness-of-fit statistics other than the R^2 value were given. A closer look at the coefficients showed that some if not all the coefficients might have been incorrectly estimated or that the data used was not the same as reported in Table 9.31. The estimated intercept term in the regression model was given as 4.875. This value should be equal to the average value of the responses which is 5.025 with the center points included in the analysis, and 4.92 without including the center points. Furthermore, upon reanalysis of the data, the R^2 value is much lower than reported and the predicted R^2 is in fact negative for the two-factor interaction model. The predicted results at the center points (when all factors are at the 0 value) in Table 9.31 were all 5.85. Using the prediction equation, the predicted values should all be 4.875.

The authors also indicated that there was statistically significant curvature and hence a follow up experiment to model the nonlinearity was required. This part of the ANOVA results was not shown in the paper.

Table 9.31: Experimental design and results of 2^{6-1} fractional factorial design. Table 1 of Zhou et al (2009).

	Experimental factors and code levels						Glucose production (mg/ml)	
Runs	x_1	x_2	x_3	x_4	x_5	x_6	Experiment	Predicted
1	-1	1	-1	-1	1	-1	5.39	5.20
2	-1	1	1	-1	1	1	5.13	4.95
3	1	1	1	1	-1	-1	6.03	6.11
4	-1	-1	1	1	1	1	5.74	5.32
5	-1	-1	1	1	-1	-1	3.29	3.26
6	-1	-1	-1	1	-1	1	3.69	3.36
7	1	-1	-1	1	-1	-1	3.75	3.79
8	-1	-1	1	-1	-1	1	2.83	3.26
9	1	-1	-1	1	1	1	4.85	5.00
10	-1	1	-1	-1	-1	1	3.92	3.42
11	-1	1	1	-1	-1	-1	3.62	3.20
12	1	1	-1	1	-1	1	6.42	6.34
13	1	1	1	-1	-1	1	5.68	6.07
14	-1	-1	-1	-1	1	1	3.82	3.54
15	1	1	-1	-1	-1	-1	4.64	4.95
16	1	1	1	-1	1	-1	5.70	5.93
17	-1	1	-1	1	1	1	4.31	5.32
18	1	-1	1	1	1	-1	4.73	5.13
19	1	1	-1	1	1	-1	8.87	8.50
20	-1	1	-1	1	-1	-1	4.83	4.83
21	-1	-1	-1	-1	-1	-1	2.66	2.97
22	1	1	-1	-1	1	1	6.24	6.16
23	-1	1	1	1	-1	1	5.50	5.32
24	-1	1	1	1	1	-1	5.46	5.63
25	-1	-1	-1	1	1	-1	4.48	3.95
26	1	-1	-1	-1	1	-1	3.96	4.15
27	-1	-1	1	-1	1	-1	3.74	3.67
28	1	1	1	1	1	1	6.82	6.63
29	1	-1	1	-1	1	1	6.04	6.09
30	1	-1	-1	-1	-1	1	4.18	4.02
31	1	-1	1	1	-1	1	6.49	6.45
32	1	-1	1	-1	-1	-1	4.63	3.61
33	0	0	0	0	0	0	6.09	5.85
34	0	0	0	0	0	0	5.64	5.85
35	0	0	0	0	0	0	5.80	5.85
36	0	0	0	0	0	0	5.94	5.85

x_1 = Cel7A, x_2 = Cel6A, x_3 = Cel6B, x_4 = Cel7B, x_5 = Cel12A, and x_6 = Cel61A.

Table 9.32: Regression analysis of the 2^{6-1} fractional factorial design. Table 3 of Zhou et al (2009).

Factors	Coefficient estimate	F-value	p-value
Intercept#	4.875	15.870	0.0044**
Cel7A (x_1)	0.666	73.080	0.0007**
Cel6A (x_2)	0.643	96.860	0.0002**
Cel6B (x_3)	0.291	0.163	0.3110
Cel7B (x_4)	0.427	13.590	0.0042**
Cel12A (x_5)	0.427	1.780	0.026*
Cel61A (x_6)	0.313	2.310	0.0222*
x_1x_2	0.119	6.070	0.0097**
x_1x_3	0.153	0.186	0.2139
x_1x_4	-0.0056	14.940	0.0036**
x_1x_5	-0.051	0.920	0.047*
x_1x_6	0.222	0.470	0.0842
x_2x_3	-0.097	0.142	0.4228
x_2x_4	0.108	8.430	0.0068**
x_2x_5	0.063	1.290	0.039*
x_2x_6	-0.074	0.131	0.5371
x_3x_4	0.142	0.166	0.2472
x_3x_5	0.051	0.124	0.6676
x_3x_6	0.256	0.880	0.0514
x_4x_5	-0.085	1.630	0.027*
x_4x_6	0.074	0.131	0.5371
x_5x_6	-0.016	0.073	0.8857

(p-value, * <0.05, ** <0.01, and R^2 = 0.9532)

Table 3 of Zhou et al (2009) reported this incorrectly as "Model"

Since the full ANOVA results were not given, it is not possible to check whether the authors used the wrong data in their analysis or the data reported in Table 9.31 was wrong.

Based on the results of Table 9.32, the authors identified only factors x_1, x_2, x_4 and x_5 and their interactions as significant. They then used a four-factor rotatable central composite design (CCD) with six center points in order to develop a second-order model for optimization of glucose production. The total number of runs required was 30 - 16 for the factorial points, eight for the axial points, and six center points. The axial points were at $\alpha = \pm 2.0$ for a rotatable design. The CCD and results were given in Table 4 in the paper and are reproduced here as Table 9.33. The other two factors, x_3 and x_6, were kept at their central values.

Table 9.33: Design and results of central composite design. Table 4 of Zhou et al (2009).

	Level of variabless				Glucose production (mg/ml)	
Run	x_1	x_2	x_4	x_5	Experiment	Predicted
1	-1	-1	-1	-1	5.00	5.12
2	1	-1	-1	-1	4.64	4.50
3	-1	1	-1	-1	5.26	5.19
4	1	1	-1	-1	6.83	7.23
5	-1	-1	1	-1	5.39	5.59
6	1	-1	1	-1	5.51	5.19
7	-1	1	1	-1	5.39	5.45
8	1	1	1	-1	7.31	7.12
9	-1	-1	-1	1	5.44	5.90
10	1	-1	-1	1	5.69	5.57
11	-1	1	-1	1	5.00	5.25
12	1	1	-1	1	7.12	7.10
13	-1	-1	1	1	6.38	5.92
14	1	-1	1	1	5.08	5.33
15	-1	1	1	1	5.68	5.53
16	1	1	1	1	7.50	7.23
17	-2	0	0	0	5.19	5.04
18	2	0	0	0	6.45	6.49
19	0	-2	0	0	6.17	6.00
20	0	2	0	0	7.33	7.39
21	0	0	-2	0	6.88	6.27
22	0	0	2	0	5.78	6.28
23	0	0	0	-2	4.98	4.83
24	0	0	0	2	4.99	5.03
25	0	0	0	0	6.70	6.67
26	0	0	0	0	6.75	6.67
27	0	0	0	0	6.67	6.67
28	0	0	0	0	6.59	6.67
29	0	0	0	0	6.56	6.67
30	0	0	0	0	6.75	6.67

The authors fitted a full second-order regression model to the experimental results and obtained the following equation (Equation 4 in the paper):

$$\begin{aligned} Glucose\ (mg\ per\ ml) \\ &= 7.141 + 0.361\, x_1 + 0.347\, x_2 + 0.005\, x_4 + 0.052\, x_5 - 0.344\, x_1^2 - 0.112\, x_2^2 \\ &- 0.216\, x_4^2 - 0.553\, x_5^2 + 0.545\, x_1 x_2 - 0.064\, x_1 x_4 - 0.047\, x_1 x_5 + 0.064\, x_2 x_4 \\ &- 0.062\, x_2 x_5 + 0.005\, x_4 x_5 \end{aligned}$$

No ANOVA results were given and no indication of the goodness-of-fit was given. The predicted values based on the equation were also given in Table 9.33. Again it can be observed that at the center point values, the predicted values did not match those from the equation. The results in Table 9.33 showed values of 6.67, but they should have been 7.141 if the regression equation was used. So either the data were incorrectly reported or the fitted equations were incorrect.

The authors then used the regression equation to obtain the combination of the factors to give maximum glucose production. They found that the optimal concentrations of Cel7A (x_1), Cel6A (x_2), Cel7B (x_4), and Cel 12A (x_5) were 4.2, 8.1, 3.7, and 3.2 μmol/l, respectively.

The results were validated by the authors using additional verification experiments and they found that the glucose produced using the optimal combinations was 15.5 mg/ml - a 2.1 fold increase when compared to a control group without optimization.

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Chapter 9: COMBINATION DESIGNS (7)

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Appendix A: Summary of factors and responses for each case study.

CS #2.1, pages 8-9.
Design: General factorial
3 Factors: Initial dye concentration, pH, Temperature
1 Response: Adsorption of Rhodomine 6G.

CS #2.2, pages 10-12.
Design: General factorial
3 Factors: Distance between cars, call duration, time of driving
1 Response: Driver's reaction time.

CS #2.3, pages 13-15.
Design: General factorial
4 Factors: SDAS, Titanium content, copper content, T7 heat treatment
5 Responses: Equivalent diameter, roundness, yield strength, ultimate tensile strength, elongation to fracture.

CS #2.4, pages 15-18.
Design: General factorial
4 Factors: Reaction time, persulfate concentration, initial MB concentration, process temperature
1 Response: Color removal efficiency (%).

CS #3.1, pages 19-22.
Design: 2-level factorial
3 Factors: pH, flow rate, magnetic field
3 responses: Induction time, total precipitation rate, homogenous precipitation rate of $CaCO_3$.

CS #3.2, pages 22-25.
Design: 2-level factorial
5 Factors: pH of initial solution, initial concentration of Zn or Cu, concentration of extractant, medium type of initial aqueous solution, stirring rate
2 responses: % removal of zinc (II), % removal of copper (II).

CS #3.3, pages 26-28.
Design: 2-level factorial
4 Factors: C, Q, A, T
2 responses: Fluoride removal efficiency, fluoride flux.

CS #3.4, pages 29-31.
Design: 2-level factorial
5 Factors: pH, particle size, Fe^{2+}, pulp density, leaching time
1 response: sulfur reduction (%).

CS #3.5, pages 31-34.
Design: 2-level factorial
4 Factors: Car weight, car speed, distance, surface inclination
1 response: time taken between bump point and stop point.

CS #3.6, pages 35-36.
Design: 2-level factorial
4 Factors: Dose AA, initial Cd (II) concentration, pH, temperature
1 response: % removal of cadmium.

CS #3.7, pages 37-38.
Design: 2-level factorial
4 Factors: Length of opening (or tube), radius of opening, temperature, initial RH
1 response: Diffusion time of moisture.

CS #3.8, pages 39-40.
Design: 2-level factorial
4 Factors: speed of rotation, cold finger temperature, experimental duration, inhibitor concentration
1 response: wax deposition.

CS #3.9, pages 41-42.
Design: 2-level factorial
4 Factors: Extraction time, microwave power, sample type
1 response: oil yield from fresh ginger (%).

CS #3.10, pages 43-44.
Design: 2-level factorial
3 Factors: Primary gas pressure, carrier gas pressure, powder feed rate
3 responses: microhardness, specific wear rate, surface roughness.

CS #4.1, pages 45-47.
Design: 2-level fractional factorial ($1/8^{th}$)
6 Factors: production interval between physical cleaning, duration of forward flush, duration of backwash, pressure during forward flush, type of water used, sequence of forward and backwash
2 responses: CWF recovery (%), wash water usage.

CS #4.2, pages 48-51.
Design: 2-level fractional factorial ($1/16^{th}$)
7 Factors: nitrogen concentration, phosphorus concentration, photon flux density, magnesium concentration, acetate concentration, ferrous concentration, NaCl concentration
2 responses: Weight content, cellular content.

CS #4.3, pages 52-55.
Design: 2-level fractional factorial (1/2)
5 Factors: Fibre, F/S, egg white, temperature, time
4 responses: Hardness, moisture content, water activity, colour.

CS #4.4, pages 55-59.
Design: 2-level fractional factorial (1/2)
6 Factors: temperature, oxygen, urea, inhibitor, sulfide, chloride
1 response: corrosion rate.

CS #4.5, pages 60-62.
Design: 2-level fractional factorial (1/16th)
7 Factors: Temperature, particle volume fraction, APPS, pH of nanofluids, elapsed time, sonication time, density of nanoparticles
1 response: Ratio of thermal conductivity of nanofluids to base fluid (K_{nf}/K_f).

CS #4.6, pages 63-65.
Design: 2-level fractional factorial (1/4)
5 Factors: Current, gas flow rate, powder feed rate, spray distance, carrier gas flow rate
3 responses: roughness, crystallinity, purity.

CS #4.7, pages 65-67.
Design: 2-level fractional factorial (1/16th)
8 Factors: Canny-Deriche filter, image amplification, edge low threshold, edge high threshold, contour closing, polygonal approximation, little chain threshold, slope threshold
1 response: covering rate

CS #4.8, pages 67-70.
Design: 2-level fractional factorial (1/16th)
7 Factors: pHc, T, Fe, N, Age, pHs, M
2 responses: percentage removal of As(V), percentage removal of As(III).

CS #4.9, pages 70-72.
Design: 2-level fractional factorial (1/8th)
7 Factors: temperature, pH agitation, water-to-substrate ratio, volume of inoculum, fermentation time, type of co-culture
1 response: Ferulic acid produced.

CS #5.1, pages 73-75.
Design: 3-level factorial
3 Factors: Cementitious materials content, water/cementitious materials ratio, fine/total aggregate ratio
2 responses: average compressive strength, standard deviation of compressive strength.

CS #5.2, pages 75-78.
Design: 3-level factorial
3 Factors: Temperature, time, pressure
5 responses: conversion rate, yield of total distillate fuels, yield of the gasoline fraction, yield of the kerosene fraction, yield of the diesel fraction.

CS #5.3, pages 78-81.
Design: 3-level factorial
4 Factors: Vibration amplitude, depth of cut, feed rate, cutting speed
1 response: Surface roughness.

CS #5.4, pages 82-83.
Design: 3-level factorial
2 Factors: Agitation speed, catalyst concentration
1 response: FAME concentration.

CS #5.5, pages 84-86.
Design: 3-level factorial
3 Factors: Basal medium, wastewater, cosubstrate type
2 responses: average compressive strength, standard deviation of compressive strength.

CS #5.6, pages 86-90.
Design: 3-level factorial
3 Factors: Cementitious materials content, water/cementitious materials ratio, fine/total aggregate ratio
1 response: Percentage of COD removed.

CS #6.1, pages 91-93.
Design: RSM - BBD
3 Factors: Ball diameter, Grinding time, bond work index
3 responses: coarse, middle, and fine size fractions of coal.

CS #6.2, pages 93-95.
Design: RSM - BBD
3 Factors: A/S, slag, sand ratio
1 response: Frost resistance coefficient.

CS #6.3, pages 96-97.
Design: RSM - BBD
3 Factors: Oxidant to sulfur molar ratio, formic acid to oxidant ratio, sonication time
1 response: Sulfur removal (%).

CS #6.4, pages 98-99.
Design: RSM - BBD
3 Factors: cutting speed, feed rate, point angle
1 response: Burr height.

CS #6.5, pages 100-101.
Design: RSM - BBD
3 Factors: Ultrasonic power, Irradiating time, pulse duty ratio
1 response: Ultrasound treatment efficiency.

CS #6.6, pages 102-105.
Design: RSM - BBD
6 Factors: Six grades of particle size (A to F)
1 response: Void content (%).

CS #6.7, pages105-107.
Design: RSM - BBD
3 Factors: Surfactant type, mass ratio of fibers/aniline, time of polymerization
1 response: Conductivity.

CS #6.8, pages 107-110.
Design: RSM - BBD
3 Factors: Ferrous iron dosage, hydrogen peroxide concentration, current density
2 responses: Percentage of color removal, COD removal.

CS #7.1, pages 111-114.
Design: RSM – rotatable CCD
3 Factors: Coagulant dosage, flocculant dosage, pH
2 responses: Turbidity, water recovery

CS #7.2, pages 115-117
Design: RSM – rotatable CCD
4 Factors: Treatment time, pH, Cr(VI) concentration, adsorbent dose
1 response: Removal of Cr(VI) (%)

CS #7.3, pages 118-120
Design: RSM – rotatable CCD
4 Factors: Temperature, sucrose concentration, salt, concentration, time.
4 responses: water loss, weight reduction, solid gain, water activity.

CS #7.4, pages 120-123.
Design: RSM – rotatable CCD
3 Factors: Rotational speed, plunge depth, dwell time
1 response: Tensile shear failure load.

CS #7.5, pages 123-125.
Design: RSM – rotatable CCD
3 Factors: Rotational speed, plunge depth, dwell time
1 response: Tensile shear failure load.

CS #7.6, pages 125-127.
Design: RSM – rotatable CCD
3 Factors: Ethanol concentration, temperature, liquid/solid ratio
2 responses: Total phenolic content, antioxidant capacity.

CS #7.7, pages 128-130.
Design: RSM – rotatable CCD
3 Factors: hydraulic retention time, up flow velocity, influent COD
1 response: COD removal (%), biogas rate.

CS #7.8, pages 131-132.
Design: RSM – rotatable CCD
3 Factors: Temperature, catalyst concentration, molar ratio
1 response: Biodiesel yield.

CS #7.9, pages 133-135.
Design: RSM – rotatable CCD
3 Factors: Coagulant dosage, flocculant dosage, pH
1 response: Turbidity, sludge volume index (SVI).

CS #7.10, pages 136-138.
Design: RSM – rotatable CCD
4 Factors: Methanol/oil molar ratio, catalyst concentration, reaction time, temperature
1 response: Biodiesel conversion (%).

CS #8.1, pages 139-142.
Design: RSM – FCD
5 Factors: Thickness of seismic mass, thickness of beams, area seismic mass, length of beams, width of beams
1 response: Natural frequency.

CS #8.2, pages 142-146.
Design: RSM – FCD
4 Factors: Fin height, pin diameter, longitudinal pitch, transverse pitch
1 response: Thermal resistance, pressure drop.

CS #8.3, pages 146-148.
Design: RSM – FCD
3 Factors: Feed rate, cutting speed, depth of cut
1 response: Delamination factor.

CS #8.4, pages 148-150.
Design: RSM – FCD
3 Factors: Radius of inner magnet, thickness of yoke, thickness of top plate
2 responses: Mean and variance of electromagnetic force.

CS #8.5, pages 150-152.
Design: RSM – FCD
4 Factors: Oil, biomass, nitrogen, phosphorus
1 response: Weathered crude oil removal (%).

CS #8.6, pages 153-155.
Design: RSM – FCD
3 Factors: Cutting speed, feed, SCEA
2 responses: Surface roughness, tangential force.

CS #8.7, pages 156-159.
Design: RSM – FCD
5 Factors: *De/D, a/D, b/D, (h-s)/D, ln Re*
1 response: *l*/D.

CS #8.8, pages 160-162.
Design: RSM – FCD
3 Factors: Time, temperature, enzyme concentration
4 responses: Turbidity, clarity, viscosity, colour.

CS #8.9, pages 162-164.
Design: RSM – FCD
3 Factors: Ethanol concentration, temperature, time
4 responses: TPC, FRAP, DPPH, Yield.

CS #8.10, pages 165-168.
Design: RSM – FCD
4 Factors: Temperature, isopropanol feed concentration, permeate pressure, feed flow rate
2 responses: Permeation flux, selectivity.

CS #9.1, pages 169-173.
Design: FFD + CCD
11 Factors: Glucose, K_2HPO_4, KH_2PO_4, $MgSO_4$, Urea, Biotin, $CoCl_2$, NaF, Peptone, Hypoxanthine, Initial pH
1 response: cAMP

CS #9.2, pages 173-176.
Design: FFD + FCD
5 Factors: Temperature, GHSV, pre-treatment time, dilution ratio, CH_4/O_2 ratio
3 responses: CH_4 conversion, C_{2+} selectivity, C_2H_4/C_2H_6 ratio.

CS #9.3, pages 177-180.
Design: FD + CCD
4 Factors (Nitrogen source): Peptone, yeast extract, ammonium sulfate, urea
4 Factors (Carbon source): Glycerol, olive oil, hexadecane, glucose
2 responses: Surface tension, emulsification index (%).

CS #9.4, pages 181-184.
Design: FFD + FCD
6 Factors: Flame height, hydrogen flux, taper speed, optic distribution ratio, humidity, temperature
1 response: Isolation.

CS #9.5, pages 184-187.
Design: FFD + FCD
4 Factors: Holding time, amount of TiH_2, amount of Ca, stirring time for Ca
3 responses: Relative density, average pore diameter, cell aspect ratio.

CS #9.6, pages 187-189.
Design: FD + CCD
3 Factors: Temperature, initial catalyst concentration, vegetable oil molar ratio
2 responses: Biodiesel purity, biodiesel yield.

CS #9.7, pages 189-194.
Design: FFD + CCD
6 Factors: Cel7A, Cel6A, Cel6B, Cel7B, Cel12A, Cel61A
1 response: Glucose production.

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Brief Biography

Dr. Leonard Lye is a happily retired professor in the Faculty of Engineering and Applied Science. Dr. Lye is the winner of numerous awards for teaching, community service, and service to the engineering profession. He is also an inventor and entrepreneur. He is the inventor of the DOE-Golfer™, and the DOE-SIM and DOE-SIM Pro IOS apps for teaching design of experiments principles which are used worldwide by corporations, universities, and six-sigma trainers.

About the book

This book provides a selection of 2^6 case studies that cover a wide range of DOE applications in engineering and science. Only papers where there is a complete set of data available for reanalysis were chosen. The selection is not exhaustive and does not cover every discipline of engineering or science. However, readers of the book should get a good sense of the wide application of DOE methods, and will try their hand at reanalyzing the published data. The methods most commonly used in the papers deal mainly with factorial designs, fractional factorial designs, and response surface methodologies, particularly the use of the central composite and Box-Behnken designs.

The book is ideal for students who have taken or is taking a course in DOE. It is also useful for those who want to learn more about the power of DOE methods or who are looking for research ideas. Each dataset is available in print form in the book and available as an Excel file (.xls) and as a Design-Expert® file (.dxpx). Hence this collection of case studies is also be a good resource for instructors of DOE.