CASE STUDIES OF USE OF DESIGN OF EXPERIMENTS IN MATERIAL RESEARCH

Salil Kumar Roy
Postgraduate Program in Civil Engineering, Petra Christian University

I Nyoman Sutapa
Department of Industrial Engineering, Petra Christian University

ABSTRACT

The paper describes principles of factorial and fractional factorial design of experiments. The various ways of analysing data obtained by these procedures are shown via four case studies. Yates method was followed in case 1 where the effect of anode type, carbon content of steel, temperature, and agitation on cathodic protection of steel in seawater, on current density, was studied. In case 2, a glass was formulated within 10 constituante melted, quantity water and tested for flow caracteristics, from the result the factor effect was calculated. In case 3, analysis of results is done in a very simple way. In this case, the effect of carbon content, surface condition, temperature, and agitation on the corrosion of steel in seawater was studied. In case 4, the effect of eleven constituents on acid resistance of a cast iron enamel has been studied through sixteen experimental compositions. This case gives a method to find out which of the sixteen experimental compositions is nearest to a target value.

Keywords : statistical design of experiment, material research.

1. INTRODUCTION

Design of experiments is an advanced statistical tool to study efficiently the effect of a large number of variables with a minimum effort in data collection. The general framework of the design is shown below in Table 1. The inputs and outputs are described as factors and responses and the experimental settings of the factors are designed with orthogonal arrays; statistical means are available for analysis of the response data. This method can give maximum amount of information with a given amount of experimental data, in other words, a certain amount of information can be obtained through a number of experiments.

2. BASIC THEORY

The simplest method of experimental design is the one dimensional search i.e. one parameter fixed at a time. This method, which is time consuming and not very efficient, is now gradually being replaced by factorial design methodology introduced by Fiscer (1960). A factorial experiment is one in which the effects of a number of different factors are investigated simultaneously, rather than conducting a series of single factor experiments. The theory and application of factorial design methodology and also some other design approaches can be found in books and articles (Cochran, et al., 1957, Box, et al., 1978, Kempthorn, 1979). One of the well known fractional factorial design approaches is the orthogonal array design. Taguchi, a Japanese engineer who has been active in the quality improvement of Japan's industrial products and processes since the late 1940s, has developed both a philosophy and methodology based on orthogonal arrays, essentially highly fractionlised factorial design (Sons, 1988).

A $L_{16}(2^4)$ orthogonal array indicates a total of 16 experiments designed with four factors each at two level of settings. The experimental conditions for this array is illustrated in Table 1. The '-' and '+' indicate the 'low' and 'high' settings of each factor i.e. A, B, C, D. The treatment combination indicates the main effect or interactions among the four factors. Thus, for:

- Null effect, having all n factor at low levels.
- Main effect, having only one factor at high level all others low i.e. A only high; B only high; etc.
- 2-factor interactions; having two factors at a time high, all others low, i.e. A and B high; A and C high; B and C high; etc.
- 3-factor interactions; having three factors at a time high, all others low, i.e. A, B and C high; A, C and D high; A, B and D high; etc… and so on, up to,
- n-factor interactions where all n factors are at high level.

In case study 1, the four factor for cathodic protection of steel evaluated, are listed in Table 2. In order to measure all the interactions among these four factors for a two level factorial experiment, the $L_{16}(2^4)$ array was chosen. In this case the response measured was current density (mA/m^2).

The response can be analysed for statistical significance by various methods (Davis, 1978). The *F-test* is very common and widely used. A convenient technique to find the *F-values* was formulated by Yates (1937). The step-by-step numerical procedure is schematically indicated in Table 3, in which *P* and *Q* are the two sets of replicate test values; *R* is their sum; *S*, *T* and *U* are intermediate steps in the computation of the total effects *V*; and *W* is the sum of squares, from which the *F-values* are calculated as:

$$F_i = \frac{W_i}{EMS} \tag{1}$$

In equation (1), EMS is the *error mean square*, obtained as follows:

$$C = \frac{(\sum R_i)^2}{32} \tag{2}$$

$$SST = \sum P_i^2 + \sum Q_i^2 - C \tag{3}$$

$$SSTR = 0.5(\sum R_i)^2 - C \tag{4}$$

$$SSR = \frac{(\sum P_i)^2 + (\sum Q_i)^2}{16} - C \tag{5}$$

$$SSE = SST - SSTR - SSR \tag{6}$$

$$EMS = \frac{SSE}{15} \tag{7}$$

The *F-values* are then checked againts *F-Distribution* Tables for various levels of significance for different combinations of the degrees of freedom of the two-variables concerned at a time. For the 2^4 factorial design, the degree of freedom V_1 and V_2 are 1 and 15 respectively, and levels of significance are known to be as follows:

If F-values is less than:	The effect is significant for:
1.43	Upper 25%
3.07	Upper 10%
4.54	Upper 5%
8.68	Upper 1%

The larger the *F-value*, the smaller is the level and hence stronger is the significance. Usually the upper 5% and 1% levels are considered sufficiently strong in significance in scientific investigations.

3. CASE STUDIES OF USE OF DESIGN OF EXPERIMENT

3.1 Case Study 1

Factorial experiments were done to study the effects of four factors (anode type, carbon content of steel, temperature, and agitation) and all the interactions among these four factors for each factor at two levels (*Zn/Al* for anode type, *0.06%/0.43%* for carbon content of steel, $20^0C/32^0C$ for temperature, and *no/yes* for agitation) (Ho, 1987). The response measured was current density (mA/m^2). The data for the 16 duplicate specimens for the current density measured, the results and the computed F-values are listed in Table 4.

The factors and interactions that are significant at various levels are grouped and shown in Table 5.

3.2 Case Study 2

Sixteen samples were obtained with factor combinations set according to Table 6. After melting and quenching in water they were tested for flow. Measured of flow is analysed with reference to every single additive (factor). The average of the first eight measurements reflects the response level when factor TiO_2 is at higher level, $\bar{y}_1$,

$$\bar{y}_1 = (1546 + 1545 + \cdots + 1600)/8 = 1576.5$$

and the average response when factor TiO_2 is at lower, $\bar{y}_2$,

$$\bar{y}_2 = (1558 + 1550 + \cdots + 1498/8 = 1489.9 .$$

Thus the effect of raising the level of factor TiO_2 from lower to higher is an increase in the response amounting to $\bar{y}_1 - \bar{y}_2 = 91.6$. Similar calculations are carried out for each of the other factors B_2O_3, Na_2O, …, Na_2SiF_6, and the result are shown in Table 7.

3.3 Case Study 3

This case shows how analysis of results can be done in a much simpler way (Ho and Roy, 1994). The corrosion of steel in tropical sea water was studied in this case. The effects of carbon content, surface condition, temperature, and agitation on the corrosion of steel in sea water were studied under laboratory conditions. Each of the four factors was studied at two levels: carbon content (0.06/0.43 wt-%), surface preparation method (dry blasted/wet blasted), temperature ($20/30^0$C), and agitation (with/without). The corrosion rates of the 16 pairs of samples studied in the laboratory are given in Table 8. The effect of agitation on corrosion based on the differences in the average corrosion rates obtained from these experiments in which the agitation changes and other condition are the same, is given in Table 9. It can be seen from Table 9 that agitation increased the rate usually by 70-100%, the effect being greater for the dry blasted specimens and particularly small for wet blasted specimens at the higher temperature.

3.4 Case Study 4

In this case, a method has been sugested to find out which of the sixteen samples is nearest to a target (Roy, 1985). Acid resistence of sixteen cast iron enamel each having Al_2O_3, CaF_2, BaO, ZrO_2, PbO, B_2O_3, TiO_2, Li_2O, CaO, Na_2O, and SiO_2 have been studied using a special design of composition. Following the design sixteen oxide compositions were calculated. The oxide compositions were then converted into batch composition. The sixteen batches were weighed and each melted in a fireclay crucible in an electric furnace. The molten mass was poured in cold water. The frit was then ground in a ball mill sieved and the powdered enamel was used to determine its acid resistance. The sixteen experimental compositions are shown in Table 10 and the results of acid resistance are also shown in that table. The target value was set as 30 for weight loss. The deviation from target was calculated and is given in the same table. A scheme was set to calculate a penalty value from each value of deviation from target. For deviation of 30, 70, 100, and 1000 the penalties were set as 1, 2, 3, and 4. The calculated penalties for each of the sixteen compositions can be seen in Table 10. Sample no. 7 has the lowest penalty value and hence is nearest to the target.

4. CONCLUDING REMARKS

- The statistical design of experiments is found very useful in material research.
- The data obtained from statistical design of experiments can be analysed by Yates method (case 1).
- The statistical design of experiments offer means to find out the effect of factors in such a way that even non-statistician can be use it (case 2 and 3).
- Development of materials with target properties can be done effectively by use of statistical design of experiments (case 4).

REFERENCES

Fisher, R.A., 1960. *The Design of Experiments*, 7th Ed., Oliver & Boyd, Edinburgh.

Cochran, W.G., and G.M. Cox, 1957. *Experimental Design*, John Wiley & Sons, New York.

Box, G.E.P., W.G. Muster, and J.S. Hunter, 1978. *Statistics for Experiments*, John Wiley & Sons, New York.

Kempthorn, O., 1979. *The Design and Analysis of Experiments,* R.E. Krieger Pub. Co., Huntington, New York.

Sons, R.J., 1988. *Taguchi Techniques for Quality Engineering*, Mc Graw-Hill.

Taguchi, G. *System of Experimental Design*, Kraus Int. Pub., New York.

Davis, O.L., 1978. *Design and Analysis of Industrial Experiments,* 2nd Ed., Longman.

Yates, F., 1937. *Design and Analysis of Factorial Experiments*, Imperial Bureau of Soil Sciences, Harpenden, England.

Ho, K.H., 1987. Study of Corrosion of Steel in Sea Water", *M.Sc Thesis*, Dept. of Mechanical Engg., National University of Singapore.

Ho, K.H., and S.K. Roy, 1994. "Corrosion of Steel in Tropical Sea Water", *British Corrosion Journal*, Vol. 29 No. 3.

Roy, S.K., 1985. "Experimental Design Approach to Enamel Formulation", *Journal of Indian Ceramics*, India.

APPENDIX

Table 1. Experimental Condition for 2^4 Factorial Design

No.	Treatment Combination	Experimental Conditions			
		A	B	C	D
1	1	+	-	-	-
2	a	-	-	-	-
3	b	+	+	-	-
4	ab	-	+	-	-
5	c	+	-	+	-
6	ac	-	-	+	-
7	bc	+	+	+	-
8	abc	-	+	+	-
9	d	+	-	-	+
10	ad	-	-	-	+
11	bd	+	+	-	+
12	abd	-	+	-	+
13	cd	+	-	+	+
14	acd	-	-	+	+
15	bcd	+	+	+	+
16	abcd	-	+	+	+

Table 2. Factors and Levels in The Laboratory Investigation on Cathodic Protection of Steel in Seawater by Sacrificial Anodes

Designation	Factor	- Level ("Low" or "1")	+ Level ("High" or "2")
A	Anode type	Zn	Al
B	Carbon content	0.06%	0.43%
C	Temperature	20^0C	32^0C
D	Agitation	No	Yes

Table 3. Determination of F-Values for 2^4 Factorial Design of Experiment

No	Condition	2 Replications		Sum	Intermediate Steps			Total Effect V_i	$W_i=V_i^2/32$	F-Val
		P_i	Q_i	$R_i=P_i+Q_i$	S_i	T_i	U_i			$F_i=W_i/EMS$
1	1	P01	Q01	R01	S01=R01+R02	T01=S01+S02	U01=T01+T02	V01=U01+U02	W01	F01
2	a	P02	Q02	R02	S02=R03+R04	T02=S03+S04	U02=T03+T04	V02=U03+U04	W02	F02
3	b	P03	Q03	R03	S03=R05+R06	T03=S05+S06	U03=T05+T06	V03=U05+U06	W03	F03
4	ab	P04	Q04	R04	S04=R07+R08	T04=S07+S08	U04=T07+T08	V04=U07+U08	W04	F04
5	c	P05	Q05	R05	S05=R09+R10	T05=S09+S10	U05=T09+T10	V05=U09+U10	W05	F05
6	ac	P06	Q06	R06	S06=R11+R12	T06=S11+S12	U06=T11+T12	V06=U11+U12	W06	F06
7	bc	P07	Q07	R07	S07=R13+R14	T07=S13+S14	U07=T13+T14	V07=U13+U14	W07	F07
8	abc	P08	Q08	R08	S08=R15+R16	T08=S15+S16	U08=T15+T16	V08=U15+U16	W08	F08
9	d	P09	Q09	R09	S09=R02-R01	T09=S02-S01	U09=T02-T01	V09=U02-U01	W09	F09
10	ad	P10	Q10	R10	S10=R04-R03	T10=S04-S03	U10=T04-T03	V10=U04-U03	W10	F10
11	bd	P11	Q11	R11	S11=R06-R05	T11=S06-S05	U11=T06-T05	V11=U06-U05	W11	F11
12	abd	P12	Q12	R12	S12=R08-R07	T12=S08-S07	U12=T08-T07	V12=U08-U07	W12	F12
13	cd	P13	Q13	R13	S13=R10-R09	T13=S10-S09	U13=T10-T09	V13=U10-U09	W13	F13
14	acd	P14	Q14	R14	S14=R12-R11	T14=S12-S11	U14=T12-T11	V14=U12-U11	W14	F14
15	bcd	P15	Q15	R15	S15=R14-R13	T15=S14-S13	U15=T14-T13	V15=U14-U13	W15	F15
16	abcd	P16	Q16	R16	S16=R16-R15	T16=S16-S15	U16=T16-T15	V16=U16-U15	W16	F16

Table 4. Result and F-values

Sample	Treatment Combination	Anode Type	Steel Type (%)	Seawater Temperature (^0C)	Agitation	Current Density (mA/m^2)		F-Value
						Rep. 1	Rep. 2	
1	1	Zn	0.06	20	No	258.0	230.8	-
2	a	Al	0.06	20	No	779.2	818.4	3694.1
3	b	Zn	0.43	20	No	199.0	198.8	269.7
4	ab	Al	0.43	20	No	681.8	658.1	73.4
5	c	Zn	0.06	32	No	419.3	396.0	586.6
6	ac	Al	0.06	32	No	1060.4	1122.0	15.9
7	bc	Zn	0.43	32	No	289.6	306.9	0.40
8	abc	Al	0.43	32	No	878.4	880.5	13.2
9	d	Zn	0.06	20	Yes	148.3	138.4	0.76
10	ad	Al	0.06	20	Yes	937.7	917.0	3.05
11	bd	Zn	0.43	20	Yes	132.2	106.3	13.3
12	abd	Al	0.43	20	Yes	541.3	485.0	14.3
13	cd	Zn	0.06	32	Yes	431.4	479.3	20.4
14	acd	Al	0.06	32	Yes	1172.8	1074.7	4.77
15	bcd	Zn	0.43	32	Yes	330.2	329.2	9.9
16	abcd	Al	0.43	32	Yes	902.3	915.6	17.0

Table 5. Levels of Significance for Various Factors and Interactions

Response	Significant at 1% Level (F >8.68)	Significant at 5% Level (F=4.54 to 8.68)	Significant at 10% Level (F=3.07 to 4.54)
Current Density	A, B, C, AB, AC, BD, CD, ABC, ABD, BCD, ABCD	ACD	AD

Table 6. Composition of Sixteen Glasses and Their Properties

	TiO_2	B_2O_3	Na_2O	ZnO	Li_2O	Al_2O_3	PbO	CaO	CaF_2	Na_2SiF_6	Opacity (%)
1	12.4	3.7	11.3	0.8	1.6	5.7	11.6	0.8	5.5	10.2	99.5
2	12.4	3.7	11.3	0.8	1.6	5.7	9.6	0.0	4.5	7.0	100.0
3	12.4	3.7	11.3	0.0	0.7	4.7	11.6	0.8	5.5	7.0	91.0
4	12.4	3.7	11.3	0.0	0.7	4.7	9.6	0.0	4.5	10.2	82.5
5	12.4	2.7	9.3	0.8	1.6	4.7	11.6	0.8	4.5	10.2	97.0
6	12.4	2.7	9.3	0.8	1.6	4.7	9.6	0.0	5.5	7.0	96.0
7	12.4	2.7	9.3	0.0	0.7	5.7	11.6	0.8	4.5	7.0	100.0
8	12.4	2.7	9.3	0.0	0.7	5.7	9.6	0.0	5.5	10.2	97.0
9	8.4	3.7	9.3	0.8	0.7	5.7	11.6	0.0	5.5	10.2	92.5
10	8.4	3.7	9.3	0.8	0.7	5.7	9.6	0.8	4.5	7.0	84.0
11	8.4	3.7	9.3	0.0	1.6	4.7	11.6	0.0	5.5	7.0	80.5
12	8.4	3.7	9.3	0.0	1.6	4.7	9.6	0.8	4.5	10.2	84.0
13	8.4	2.7	11.3	0.8	0.7	4.7	11.6	0.0	4.5	10.2	89.0
14	8.4	2.7	11.3	0.8	0.7	4.7	9.6	0.8	5.5	7.0	71.5
15	8.4	2.7	11.3	0.0	1.6	5.7	11.6	0.0	4.5	7.0	77.5
16	8.4	2.7	11.3	0.0	1.6	5.7	9.6	0.8	5.5	10.2	88.0

Table 7. Summary of Factor Effects on Flow

Additive	TiO_2	B_2O_3	Na_2O	ZnO	Li_2O	Al_2O_3	PbO	CaO	CaF_2	Na_2SiF_6
Flow	91.6	19.9	-66.9	-15.1	7.6	33.4	3.4	12.6	6.4	0.1

Table 8. Results of Corrosion of Steel in Sea Water (Laboratory Test)

Specimen No.	Carbon Content (wt-%)	Surface Preparation method	Temperature (^{0}C)	Agitation	Corrosion rate (mm/year)		
					Replica 1	Replica 2	Average
1	0.06	Dry blasted	20	No	0.131	0.120	0.126
2	0.43	Dry blasted	20	No	0.095	0.098	0.097
3	0.06	Wet blasted	20	No	0.126	0.111	0.119
4	0.43	Wet blasted	20	No	0.127	0.092	0.110
5	0.06	Dry blasted	32	No	0.255	0.261	0.258
6	0.43	Dry blasted	32	No	0.181	0.196	0.189
7	0.06	Wet blasted	32	No	0.225	0.231	0.228
8	0.43	Wet blasted	32	No	0.170	0.174	0.172
9	0.06	Dry blasted	20	Yes	0.217	0.212	0.215
10	0.43	Dry blasted	20	Yes	0.225	0.198	0.212
11	0.06	Wet blasted	20	Yes	0.137	0.164	0.151
12	0.43	Wet blasted	20	Yes	0.200	0.177	0.189
13	0.06	Dry blasted	32	Yes	0.378	0.524	0.451
14	0.43	Dry blasted	32	Yes	0.290	0.291	0.291
15	0.06	Wet blasted	32	Yes	0.283	0.243	0.263
16	0.43	Wet blasted	32	Yes	0.197	0.181	0.189

Table 9. The Effect of Agitation on Corrosion Rate

Experiment	Condition	Change in Corrosion Rate (%)
At 20^{0}C		
1-9	Dry blasted, 0.06%C	+70.6
3-11	Wet blasted, 0.06%C	+26.9
2-10	Dry blasted, 0.43%C	+118.6
4-12	Wet blasted, 0.43%C	+71.8
At 32^{0}C		
5-13	Dry blasted, 0.06%C	+74.8
7-15	Wet blasted, 0.06%C	+15.4
6-14	Dry blasted, 0.43%C	+54.0
8-16	Wet blasted, 0.43%C	+10.0

Table 10. Composition of the Sixteen Enamels, Weight Loss Due to Acid, Deviation from Target, and Penalty for Deviation

Sample	Al_2O_3	CaF_2	BaO	ZrO_2	PbO	B_2O_3	TiO_2	Li_2O	CaO	SiO_2	Na_2O	Weight Loss Due to Acid	Deviation from Target	Penalty for Deviation
1	7.06	4.53	6.8	18.22	2.6	10.15	11.24	4.4	1.24	20.78	12.98	415	385	3.32
2	6.0	4.53	6.0	18.22	2.6	10.15	7.24	4.4	1.0	26.88	12.98	215	185	3.09
3	7.06	3.83	6.0	18.22	2.6	8.15	11.24	1.0	1.24	27.68	12.98	52	22	0.73
4	6.0	3.83	6.8	18.22	2.6	8.15	7.24	1.0	1.0	32.18	12.98	56	26	0.87
5	7.06	3.83	6.8	18.22	1.0	10.15	11.24	4.4	1.0	25.3	11.0	85	55	1.63
6	6.0	3.83	6.0	18.22	1.0	10.15	7.24	4.4	1.24	30.92	11.0	98	68	1.95
7	7.06	4.53	6.0	18.22	1.0	8.15	11.24	1.0	1.0	30.8	11.0	47	17	0.57
8	6.0	4.53	6.8	18.22	1.0	8.15	7.24	1.0	1.24	34.82	11.0	49	19	0.63
9	6.0	4.53	6.8	16.0	1.0	10.15	11.24	4.4	1.24	29.06	12.98	47	17	0.58
10	7.06	4.53	6.0	16.0	1.0	10.15	7.24	4.4	1.0	33.04	12.98	56	26	0.87
11	6.0	3.83	6.0	16.0	1.0	8.15	11.24	1.0	1.24	29.16	12.98	111	81	2.32
12	7.06	3.83	6.8	16.0	1.0	8.15	7.24	1.0	1.0	31.54	12.98	152	122	3.02
13	6.0	3.83	6.8	16.0	2.6	10.15	11.24	4.4	1.0	30.38	11.0	48	18	0.60
14	7.06	3.83	6.0	16.0	2.6	10.15	7.24	4.4	1.24	33.88	11.0	46	16	0.53
15	6.0	4.53	6.0	16.0	2.6	8.15	11.24	1.0	1.0	29.08	11.0	101	71	2.03
16	7.06	4.53	6.8	16.0	2.6	8.15	7.24	1.0	1.24	30.98	11.0	113	83	2.43