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Statistical approaches in surface finishing. Part 3. Design-of-experiments

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Abstract
This paper is the third and final part of a short series of articles aimed towards describing some of the various statistical methods and approaches that have been used in surface finishing. Here, various types of Design-of-Experiments (DOE) techniques are described, such as full and fractional factorial design, central composite design, response surface methodology and Taguchi methods. Their application in the design of surface finishing related experiments are then reviewed, ending with a worked example of a two-level 2^4 full factorial DOE.

Keywords: Statistics; Design-of-Experiments; DOE; factorial design; central composite design; response surface methodology; Taguchi methods; electrodeposition; electroplating; coatings; surfaces.

Introduction

In the two previous parts of this short series of articles describing statistical approaches in surface finishing research, parametric statistics testing of data[1] and non-parametric methods for data analysis[2] with hypothesis testing were discussed. In Part 1,[1] an introduction to statistical design-of-experiments (DOE) and some examples of the use of this in various surface finishing research studies were given. In the current article, the final part of this series, statistical DOE and its application in surface finishing research are covered in more detail, and its value to researchers in the finishing field will hopefully become clear.

Since an earlier publication,[3] dealing with the use of statistics as an aid in improving production quality control in electroplating plants more than 20 years ago, it was noted in Part 1 that there has been a marked increase in the use of quite sophisticated statistical tools in design of metal finishing experimentation and analysis of data obtained, but no data were given to show this trend.

In Fig. 1, data are provided from Google Scholar for the number of articles published (hits) in six 3-year periods going back to 1996 and accessed from two sets of keywords: (i) experimental design statistics electrodeposition, and (ii) electrodeposition, in order to determine within these time periods how many of the total articles published reported the use of statistical design in their experimentation. Changing the keywords slightly, *e.g.*, to statistical experimental design electrodeposition, gave slight changes in numbers of published articles, but the trends remained the same. It seems clear from Fig. 1, in which total numbers of articles published *via* keyword "electrodeposition" are plotted at 10% actual numbers to facilitate comparison with those from "experimental design" samples, that in recent years a marked increase in studies in metal finishing using statistical experimental design has occurred. In the sample shown (Fig. 1), only just over 7% of electrodeposition 1996-1999 studies reported by Google Scholar used DOE, whereas this had increased to about 19% in those reported for 2016-2019. The latter period is incomplete (accessed on 25th August 2019) and thus the precise ratio is uncertain, but the trend seems clear; indeed the data (Fig. 1) implies a notable surge in uptake of DOE methodologies in this latest 3-year period, a finding yet to be validated.

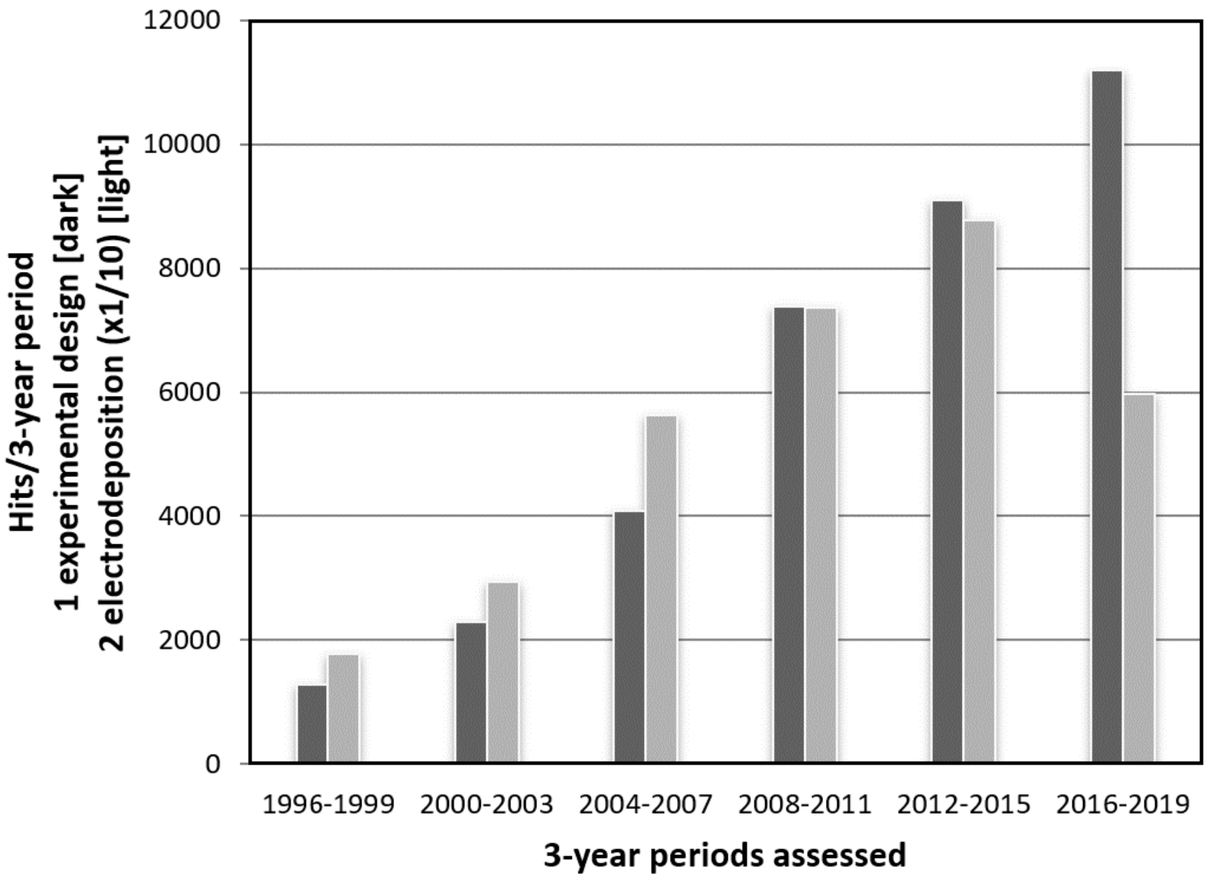

Figure. 1. Number of articles published (hits) in six 3-year periods from 1996 to 2019 accessed from two sets of keywords: (i) experimental design statistics electrodeposition, and (ii) electrodeposition, to determine within these time periods how many of the total articles published reported the use of statistical design in their experimentation. Source of data: Google Scholar.

As noted in the short review of DOE in the first part of this series,[1] examples of the different DOE statistical tools used in metal finishing are widespread. The use of some of these, both singly and in combination with other statistical tools, now follows after a brief description of some of the techniques. A number of the examples using DOE given before are revisited along with more recently cited studies.

Fundamentals of DOE

In the DOE method,[1] when large numbers of variables (called factors) are involved and the experiment requires them to be controlled, a methodology is used to minimise the number of component experiments, often with each variable being allocated a high and low value.[4-6] It is then possible to statistically work out the significance and effect size of each variable (or combination of variables). Careful implementation (design) at the beginning of the set of measurements is required, as this cannot be introduced after the experimentation has begun. In its basic form, DOE can allow the saving of experimentation, resources and costs, but yield more in results; a well-designed experiment can give statistical analysis of data to a high degree of confidence, and with more certainty about the conclusions. It also leads to ease of replication and validation by other researchers. Not every experimental combination is performed in a DOE, which is a disadvantage, and thus selecting appropriate levels for the upper and lower limits is crucial.

Full and fractional factorial design (FFD)

Typically, a full factorial design (FFD) DOE is noted as an L^k design, where k denotes the number of factors (experimental variables) and L is the number of levels (experimental settings) of each factor. Often, L = 2 (a two-level full factorial design), with the 2 levels being realistic high and low values (typically denoted +1 and -1, respectively) of each factor. Thus, a 2^4 design will have 4 factors (say, *A*, *B*, *C* and *D*), each with a high and low value. Therefore, 16 experiments will be required, each with the 4 factors being set as high and low. The first step in such a DOE is to identify the factors involved; this is usually achieved from carrying out initial (non-DOE) experiments, theoretical considerations or brain storming. A worked example of a two-level full factorial design DOE is presented at the end of this article.

A fractional factorial design (with the same FFD acronym) is similar to the full factorial design, except that the full range of experiments, *e.g.,* 16 in a 2^4 full factorial design, are considered not worth carrying out. For example, some experiments might be thought to be redundant, giving little or no new information. Thus, the L^k design notation is reduced to L^{k-p}, where *p* describes the size of the fraction of the full factorial used. For example, a 2^{5-2} design is ¼ of a 2^5 full factorial design, using 8 runs (2^3) rather than 32 runs (2^5). This reduction is achieved by considering 3 factors (say *A*, *B* and *C*) but then choosing to 'confound' the remaining (*D* and *E*) factors, such that $D = A \times B$ and $E = A \times C$.[7]

Factorial design and fractional factorial design (FFD) are still used, often in conjunction with other methods detailed below.

Central composite design

A central composite design (CCD) is an experimental design, with application in response surface methodology, for creating a second order (quadratic) model for the response variable.[8] This obviates the requirement to use a complete three-level factorial experiment, for example, when a non-linear relationship between factors and response are found and an intermediate (between high and low) level is required. On completion of the designed experiment, the data are analysed by linear regression, sometimes iteratively.

Response surface methodology

Response surface methodology (RSM) explores the relationships between several explanatory variables and one or more response variables.[9] Essentially, RSM uses a sequence of designed experiments to obtain an optimal response. Using a second-degree polynomial model to do this often gets results, but firstly estimating a first-degree polynomial model can be easily done using a factorial experiment or an FFD. This is enough to show which explanatory variables affect the relevant response variable(s). Once the researcher believes that only significant explanatory variables are left, then a more complicated design, such as a CCD can be implemented to estimate a second-degree polynomial model. The Box–Behnken design (BBD)[10] array is often opted for as it gives optimised results for more than three parameters in less number of experiments. Four control variables and three levels involve 81 experiments to determine optimum deposition condition, whereas BBD can achieve more specific optimum conditions in only 29 experiments.

Taguchi methods

Another statistical tool finding favour is the Taguchi experimental design approach;[11] Taguchi proposed extending each experiment with an "outer array" (possibly an orthogonal array); the "outer array" (often used in product quality assessment and control) should simulate the random environment in which the product, or research interaction, would function. This is an example of judgmental sampling. Many of the orthogonal arrays that Taguchi has advocated are saturated arrays, allowing no scope for estimation of interactions. However, interactions are part of the real world. In Taguchi's arrays, interactions are confounded and not always easy to resolve. However, this is only true for "control factors" or factors in the "inner array". By combining an inner array of control factors with an outer array of "noise factors", Taguchi's approach provides "full information" on control-by-noise interactions, it is claimed. Taguchi argues that such interactions do much in achieving a design that is robust to noise factor variation. The Taguchi approach provides more complete interaction information than typical FFDs, its adherents claim.

Taguchi users believe that the designs offer rapid results and that interactions can be eliminated by proper choice of quality characteristics. However, sensibly, a "confirmation experiment" carried out would offer protection against any residual interactions.

There is an argument to be made that an eight run array is the most practical and universally applicable array that can be chosen for the use of a DOE. There are several forms of these and the various types of these eight run arrays (e.g., 2^3 Full Factorial, Taguchi L8, 2^{4-1} Half Fraction, Plackett-Burman 8-run etc.) have various names, but in essence they are all considered to be quite similar.

Applications of DOE in metal finishing research

Factorial factorial design

A 2-level FFD was employed with Pareto charts and normal probability plots of standardized effects to assess factors affecting %Ni deposited in statistical studies of Zn–Ni alloy electrodeposition using non-cyanide alkaline baths containing polyethyleneimine complexing agents.[4]

Two partially superimposed factorial designs with two factors at two levels were employed in a study on pulsed electrodeposition of cobalt nanoparticles on copper,[12] the influence of the operating parameters on size distribution and morphology. The effect of current density (i = 10 and 50 mA cm^{-2}) and discharged cobalt ($Q = 2.5 \times 10^{-3}$ and 1.0×10^{-2} C) was investigated by the first factorial design investigated; the second factorial design studied, for the same two levels of Q, the effect of cobalt concentration (Co = 0.01 and 0.1 M).

Oliveira *et al.* investigated the optimal current density and bath temperature and their effect on the cathodic current efficiency for Ni–W–Fe electrodeposition, using a factorial design 2^2 with 2 centre points.[13]

Casciano *et al.*[14] used a two-level and centre-point factorial design in the electrodeposition of Co-Mo coatings and their evaluations for hydrogen evolution reaction. Optimisation of CuW alloy electrodeposition towards high-tungsten content was carried out by Bacal *et al.*[15] employing FFD.

The tribological properties of bolts depending on different screw coatings and lubrications was studied by Croccolo *et al.*[16] A full factorial design of 2^5 with 10 replications would have required a number of 320 experiments to be carried out. Therefore, this was reduced, by fractionating the experimental design and using the Taguchi L8 magic square, which made it possible to easily manage a 2^{5-2} experiment. Zinc-coated screws showed a lower friction coefficient in comparison to black oxidised ones, and ceramic paste lubrication, even when performed only at the underhead, was seen to be more effective than oil lubrication.

Huang *et al.* using FFD and CCD coupled with RSM in an investigation of the optimal electroplating parameters for a pulsed-current co-electrodeposition system of Au-Sn deposits in a non-cyanide electrolyte.[17]

In the non-anomalous plating of Co-Ni and Fe–Co,[18] and Fe–Ni[19] alloys using pulse-reverse electroplating was achieved by employing experimental strategies including FFD, path of steepest ascent and CCD coupled with RSM.

Response surface methodology and central composite design-based studies

The optimisation of hardness and hydrogen content of chromium coating under pulse reverse electroplating was carried out by Addach *et al.* who used experimental strategies including a factorial portion of CCD and optimum paths coupled with the desirability function.[20] CCD and response surface methodology for statistical modelling and multi-response optimisation of a nickel electroplating process was used by Poroch-Seritan and colleagues.[21] Electrodeposition concentrations for hydroxyapatite coatings on CoCrMo biomedical alloys were optimised by Coşkuna *et al.* by employment of RSM and CCD, and the results obtained from RSM were treated by analysis of variance (ANOVA) and using a second order polynomial equation.[22]

Yaghoubinezhad and Afshar[23] used CCD in an experimental design for optimising the corrosion resistance of pulse reverse electrodeposited graphene oxide thin film by measuring linear polarisation resistance (LPR); the best prediction model proposed a definitive linear relation without any significant interaction of the LPR by analysis of variance.

The influence of deposition parameters on the electrodeposition of Cu–Zn[24] and Cu–Co[25] alloys in citrate medium has been carried out with the use of RSM and optimisation techniques. Experimental investigations on redefining the surface quality of bevel gears by pulsed electrochemical honing[26] have also been carried out with the employment of RSM design methodology.

Patel and Gohel[27] studied optimisation of sol–gel spin-coated Cu_2ZnSnS_4 (CZTS) thin-film control parameters by the RSM method to enhance the solar cell performance, using the Box–Behnken design (BBD) array for the optimisation. Using RSM, Haider *et al.*[28] were able to evaluate of the effects of copper electroplating parameters on the adhesion of the deposit to the austenitic stainless steel substrate.

Raghavendra *et al.*[29] studied the optimisation of coating parameters in the electrodeposition of Ni–Al_2O_3 nanocomposite coating on Al6061 substrate, using RSM based Grey relation analysis to get maximum coating thickness, adhesive strength, microhardness, and minimum wear rate.

Process optimisation for window material CdS thin films grown by a successive ionic layer adsorption and reaction method was achieved by Yucel *et al.*[30] using RSM based on CCD. An RSM approach using CCD was applied to the modelling and optimisation of PPy-ND nanocomposite deposition using sonoelectrochemical synthesis by Ashassi-Sorkhabi *et al.*;[31] they determined the concentration of nanoparticles, applied current density and synthesis time to achieve the most protective films.

Bahadormanesh *et al.*[32] studied electrodeposition of nanocrystalline Zn/Ni multilayer coatings from a single bath with emphasis on influences of deposition current densities and number of layers on characteristics of deposits. The optimisation model based on RSM gave results demonstrating that improvement in corrosion resistance due to increase in difference of deposition current densities was more effective with a higher number of layers. Modelling and optimisation of an electrode modified with poly(3,4-phylenedioxythiophene)/graphene oxide composite by an RSM/Box-Behnken design approach was carried out by Talib *et al.*[33] Jagadish *et al.*[34] carried out a process optimisation for n-type Bi_2Te_3 films electrodeposited on flexible recycled carbon fibre using RSM.

Taguchi methods

The electrodeposition of copper on titanium wires,[35] has been studied by Rosa *et al.* employing the Taguchi method as has the optimisation of electroplating conditions of chromium from hexavalent (CrVI) baths, by Jadid and colleagues.[36] Hui Jin *et al.*[37] have reported on the synthesis and properties of electrodeposited Ni–CeO_2 nano-composite coatings, based on an orthogonal experimental design and analysis method. Jeyarag *et al.*[38] carried out investigations on the effect of process parameters of electrodeposited Ni-Al_2O_3 composite coating, and also[39] studied electrodeposited Ni-Cr composite coating using a Taguchi L27 orthogonal array approach and mathematical modelling, using robust design approach. Pech-Rodríguez *et al.*[40] studied the electrophoretic deposition of copper-carbon nanotubes composite coatings in deep eutectic solvent using a Taguchi experimental design approach. The optimisation of microhardness of electrodeposited Co–Ag coatings using Taguchi design and ANOVA methods was carried out by Kirati *et al.*[41]

Worked example of a full factorial design DOE experiment

Consider a required experiment to investigate the effect of electroplating bath variables on the thickness of a nickel deposit. A two-level full factorial design DOE approach could be used. This would reduce the number of experiments to be performed (and hence costs), allow identification of combinations of variables that would produce a required (not necessarily maximum) coating thickness, and permit an evaluation of the interaction of variables.

In this hypothetical example, let's suppose the main factors were identified as being nickel concentration (*A*), the concentration of a plating additive of interest (*B*), current density (*C*) and temperature (*D*). Thus, a full factorial 2^4 design approach (4 factors, 2 realistic high and low settings for each factor; 16 experiments, Table 1), could be performed.

Table 1. Model matrix for 2^4 full factorial experimental design. *A* to *D* are the factors; *Z* is the mean nickel coating thickness in this hypothetical example.

Run order	*A*	*B*	*C*	*D*	*Z* / µm
1	-1	-1	-1	-1	5.65
2	-1	-1	-1	+1	8.00
3	-1	-1	+1	-1	5.27
4	-1	-1	+1	+1	9.70
5	-1	+1	-1	-1	8.28
6	-1	+1	-1	+1	11.50
7	-1	+1	+1	-1	6.50
8	-1	+1	+1	+1	8.60
9	+1	-1	-1	-1	6.70
10	+1	-1	-1	+1	15.40
11	+1	-1	+1	-1	9.90
12	+1	-1	+1	+1	10.70
13	+1	+1	-1	-1	14.30
14	+1	+1	-1	+1	19.30
15	+1	+1	+1	-1	11.20
16	+1	+1	+1	+1	21.30

Table 2. Factors and levels for the full factorial experiments.

Factor	Factor code letter	Low level (-1)	High level (+1)
Nickel concentration / g dm^{-3}	*A*	1	5
Additive concentration / g dm^{-3}	*B*	0.01	1.0
Current density, j / mA cm^{-2}	*C*	10	50
Temperature / °C	*D*	22	35

Let's assume these experiments have been carried out in duplicate (or triplicate) and means for the coating thickness (*Z*) are provided (Table 1). Realistic high and low values for each of the variables (*A-D*) should be selected (Table 2). The order in which the runs (combination of high and low variables) are sequenced (performed experimentally) should ideally be random, to reduce experimental error and bias, although not strictly necessary. Factorial design allows an investigation of the interactions of factors on the outcome value (coating thickness) to take place; *e.g.*, the combination of nickel concentration (*A*) and additive concentration (*B*), denoted *AB* (a two-way interaction), may have a greater influence than *A* or *B* singularly. For 4 factors, there will be 11 such combinations: *AB*, *AC*, *AD*, *BC*, *BD*, *CD*, *ABC* (a 3-way interaction), *ABD*, *ACD*, *BCD* and *ABCD* (a 4-way interaction). Thus, it is possible that such combinations might have a greater effect on coating thickness than each factor individually (*A*, *B*, *C* or *D*).

A convenient software package to use for DOE analyses is Mintab (a commercially available statistics package first developed by Pennsylvania State University in 1972; Minitab 17 used here). Calculations could be carried out using Microsoft Excel, although for brevity, the example here is demonstrated using Minitab. A step-by-step guide for entering the factors and performing the software operations in this example is documented in the Supplementary materials (Fig. S1 – S4).

An equation relating the output variable (*Z*, coating thickness) to the factors is calculated by Minitab (Eq. 1):

$$Z = 6.402 - 7.756A - 5.093B - 0.228C - 0.03230D + 5.248AB + 0.1300AC + 0.1718AD + 0.3054BC + 0.2972BD + 0.008939CD - 0.2040ABC - 0.1674ABD - 0.004877ACD - 0.01408BCD + 0.007867ABCD \quad \text{(Eq. 1)}$$

One of the main outputs from performing a full factorial DOE is a Pareto chart (Fig. 2). This details the effects of the factors, and combination of factors, on the outcome parameter (coating thickness, in this example). Horizontal bars that exceed the calculated significant threshold (vertically dotted line) correspond to factors that are statistically significant at the $\alpha = 0.05$ level);[1] any factors below this line do not significantly affect the outcome parameter. Thus, nickel ion concentration (*A*), temperature (*D*) and additive concentration (*B*) significantly affect the plating thickness (*Z*). Longer bars exceeding the threshold line also represent greater significance, *i.e.*, *A* has the greatest effect in this hypothetical example, with *D* having a lower effect and *B* having the least effect. Again, in this hypothetical example, *C* has no significant effect on plating thickness, nor do any of the factor combinations (*AB*, *CD*, *ABCD* etc). In a real-life example, *C* would be expected to have a significant effect, from Faraday's Laws, although maybe higher current densities favour a side-reaction, such as hydrogen evolution rather than metal deposition. The effect sizes are also output numerically in Minitab (Table 3) and are also shown as a normal plot, where deviations from linearity show the significant factors (Fig. 3).

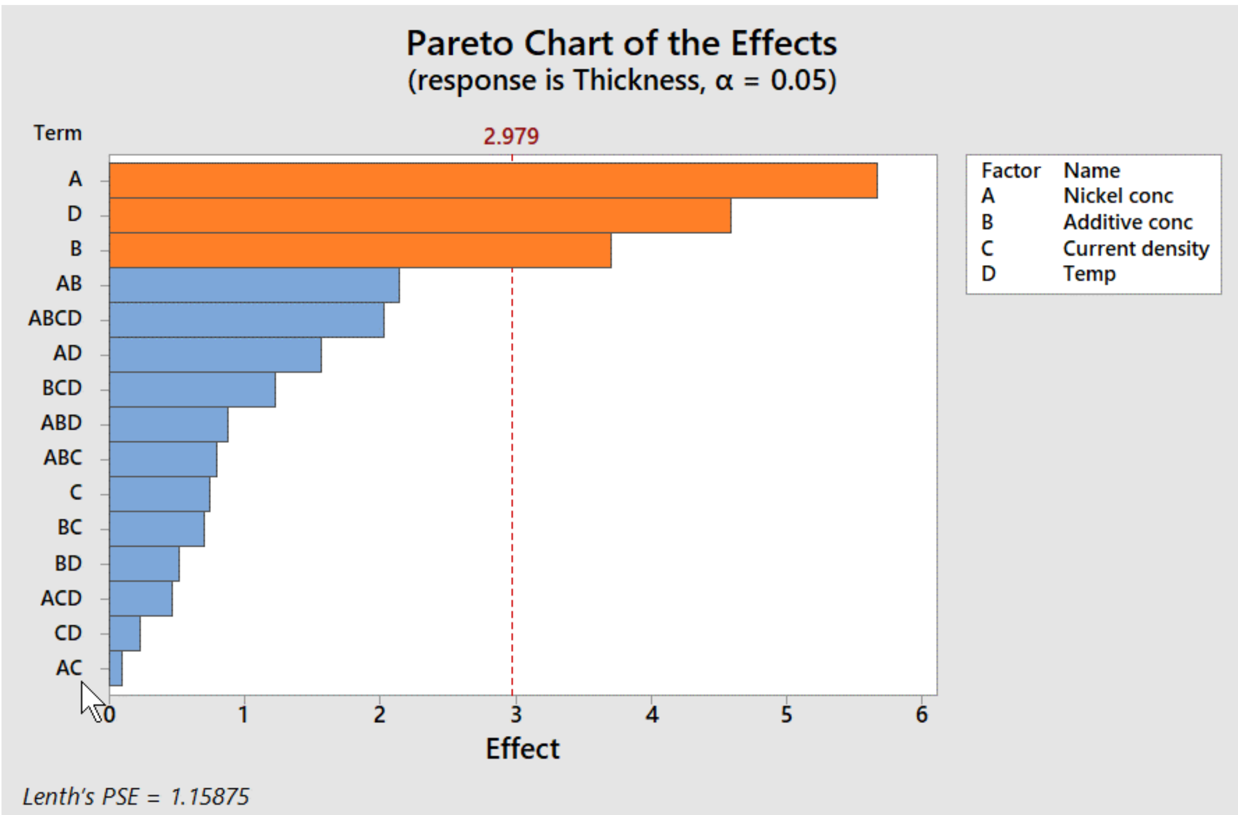

Figure 2. Pareto plot showing effect size and significance (effect size > threshold, here 2.979) of factors and combination of factors on the output variable (*Z*). Here, *A*, *B* and *D* are significant factors.

Having established which factors are significant in affecting coating thickness, the next question might be which combination of variables can achieve a specific coating thickness (*Z*). For example, what values of *A* – *D* are required to produce a coating thickness of 10 μm. Minitab allows answers to such questions using the 'Response Optimizer' function (see Supplementary materials, Figs. S3 and S4). Typical output is shown in Fig. 4, where optimal values of *A*, *B*, *C* and *D* are output (3.0000 g dm^{-3}, 0.5050 g dm^{-3}, 30 mA cm^{-2} and 26.3 °C, respectively).

Table 3. Effect sizes output by Minitab in the hypothetical example. Numbers above the threshold (2.979, *) have a significant ($p < 0.05$) effect on Z (A has the greatest effect); numbers below the threshold (2.979) have an insignificant (NS, $p > 0.05$) effect on Z.

Factor code and interactions	Effect size	Significance
A	5.662	*
B	3.707	*
C	-0.7450	NS
D	4.588	*
AB	2.143	NS
AC	0.09500	NS
AD	1.5625	NS
BC	-0.7000	NS
BD	0.5175	NS
CD	-0.2300	NS
ABC	0.8000	NS
ABD	0.8825	NS
ACD	-0.4700	NS
BCD	1.2250	NS
ABCD	2.025	NS

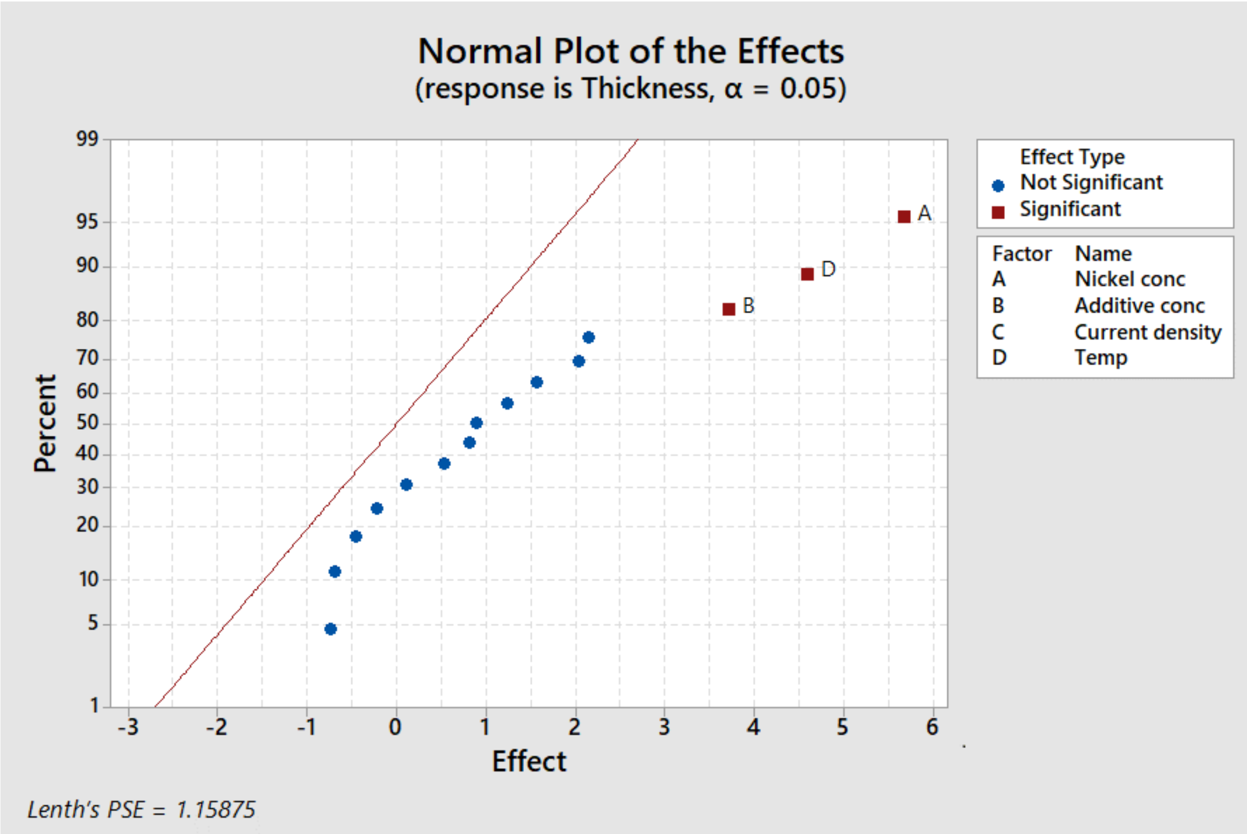

Figure 3. Normal plot showing effect of factors and combination of factors on the output variable (Z). Plot should be a straight line with significant factors (e.g., *A*, *B* and *D*) deviating away from linearity.

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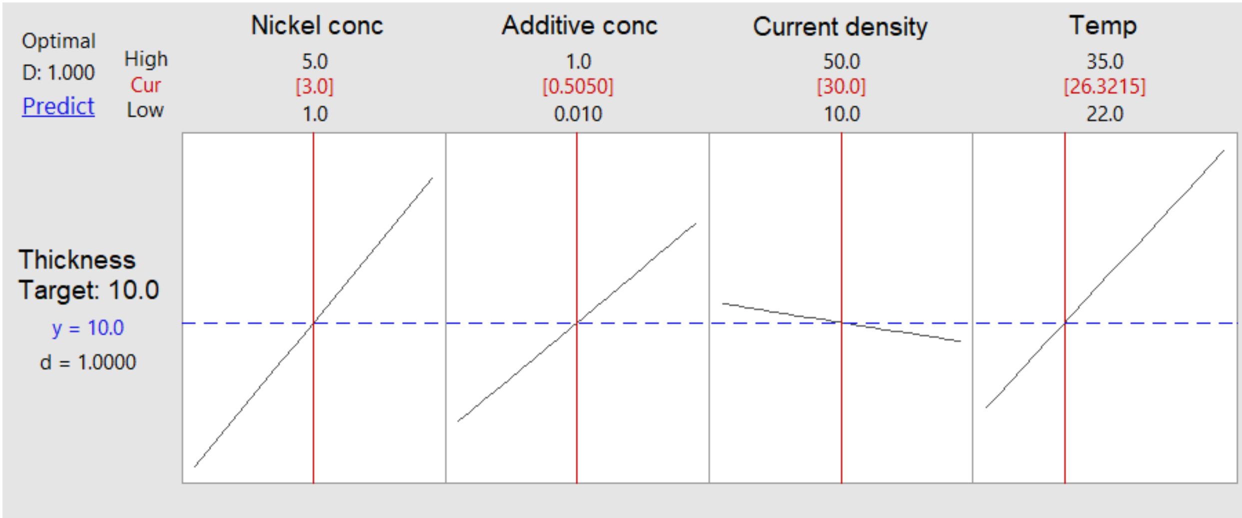

Figure 4. Optimisation plot showing optimal values of *A*, *B*, *C* and *D* (3.0000 g dm^{-3}, 0.5050 g dm^{-3}, 30 mA cm^{-2} and 26.3 °C, respectively) to achieve a specific coating thickness (*Z*) of 10 µm, in the hypothetical example.

It might be the case that a reduction in some factors would be operationally more favourable than others to achieve the same desired output variable. For example, minimising *C* and *D* to reduce costs might well be considered important, even at the expense of an increase in *A* and *B*. Again, this can be done using the software by adjusting the horizontal slider on each of the factors (Fig. 5). Alternative values of 3.7949 g dm^{-3}, 0.5438 g dm^{-3}, 11.72 mA cm^{-2} and 22.8 °C, for *A*, *B*, *C* and *D*, respectively, are now obtained to produce the same 10 µm value for *Z*.

Note, on these optimisation plots (of *Z vs*. factor), the gradient of the line is proportional to the effect size (Table 3); negative gradients show inverse relations between Z and the factor, as is the case with *C* (although not a significant factor), in this hypothetical example.

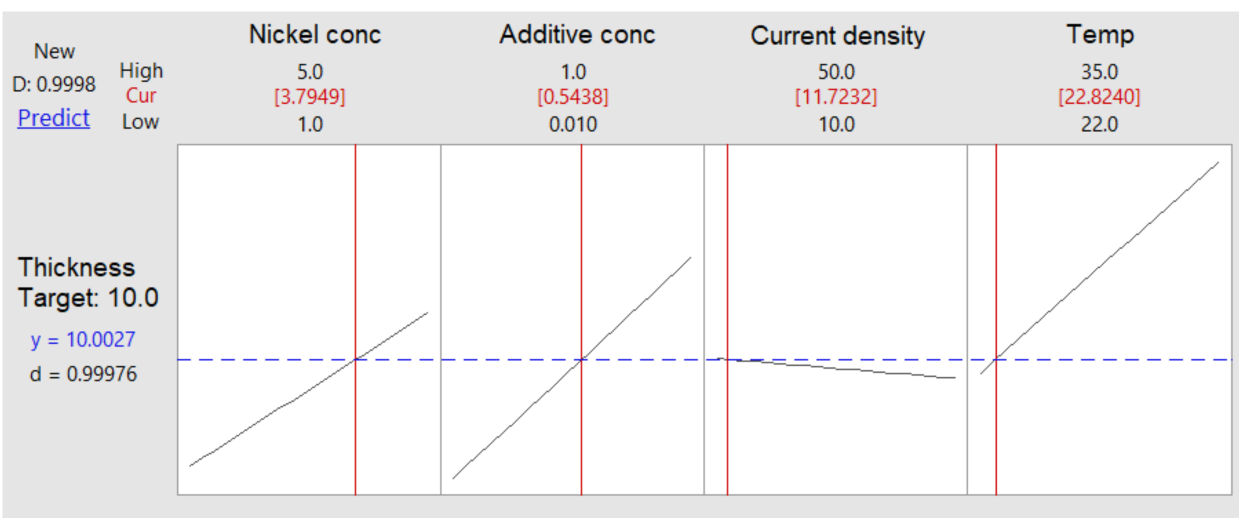

Figure 5. Optimisation plot showing slightly sub-optimal values of *A* – *D* (3.7949 g dm^{-3}, 0.5438 g dm^{-3}, 11.72 mA cm^{-2} and 22.8 °C, respectively) to achieve a specific coating thickness (*Z*) of 10 µm but with low values of *C* and *D*, considered to be expensive, in the hypothetical example.

Minitab also allows many of the other DOE methods described in this article to be performed, such as partial factorial design and Taguchi methods, which the reader may want to explore. A worked example of the latter method is shown in ref.[42]

Summary

This paper is the third and final part of a short series of articles that report the use of statistical methods in surface finishing. The focus in this paper has been on the use of DOE relevant to surface finishing investigations. There is evidence to suggest that increasing numbers of metal finishing studies are taking advantage of the benefits afforded by using DOE and statistical analysis of resulting data.

Disclosure statement

No potential conflict of interest was reported by the authors.

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Supplementary material
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Minitab - Nickel3.MPJ - [Worksheet 1 ***]

File Edit Data Calc Stat Graph Editor Tools Window Help Assistant

↓	C1	C2	C3	C4	C5	C6	C7
	Nickel conc	Additive conc	Current density	Temp	Thickness		
1	1	0.01	10	22	5.65		
2	1	0.01	10	35	8.00		
3	1	0.01	50	22	5.27		
4	1	0.01	50	35	9.70		
5	1	1.00	10	22	8.28		
6	1	1.00	10	35	11.50		
7	1	1.00	50	22	6.50		
8	1	1.00	50	35	8.60		
9	5	0.01	10	22	6.70		
10	5	0.01	10	35	15.40		
11	5	0.01	50	22	9.90		
12	5	0.01	50	35	10.70		
13	5	1.00	10	22	14.30		
14	5	1.00	10	35	19.30		
15	5	1.00	50	22	11.20		
16	5	1.00	50	35	21.30		
17							
18							

Figure S1. Data entry.

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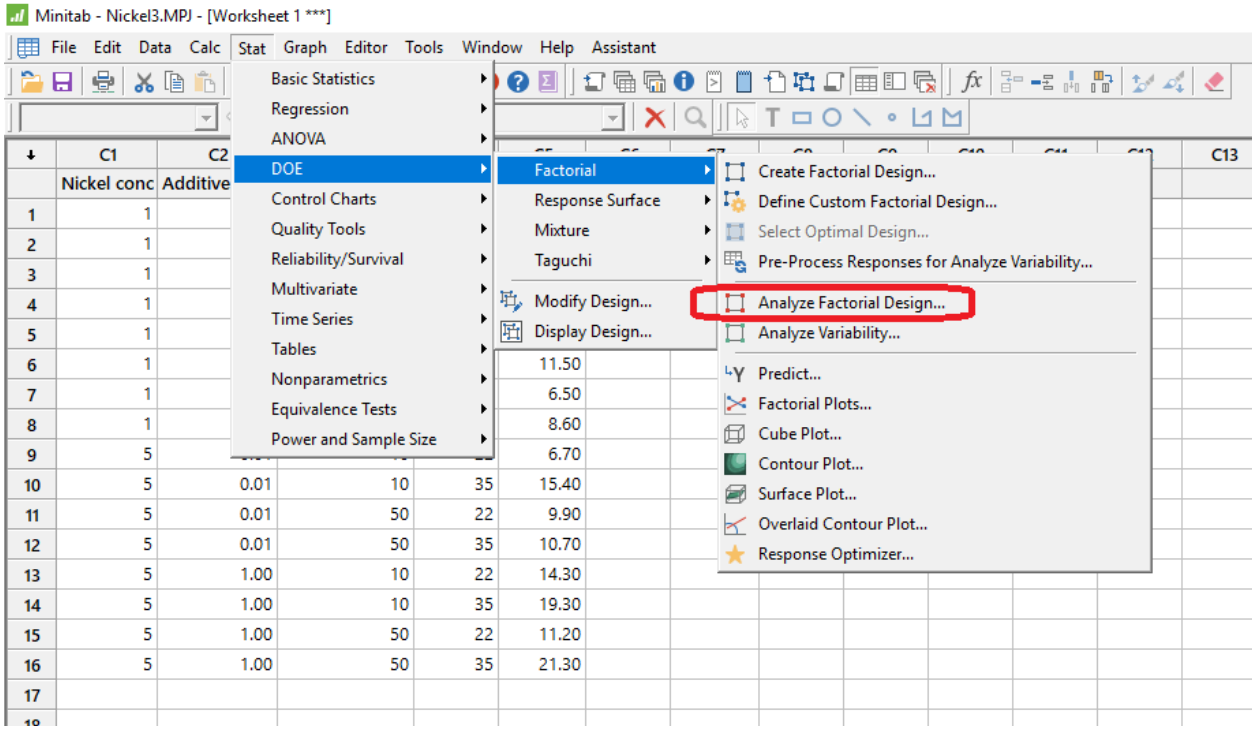

Figure S2. Analyse factorial design.

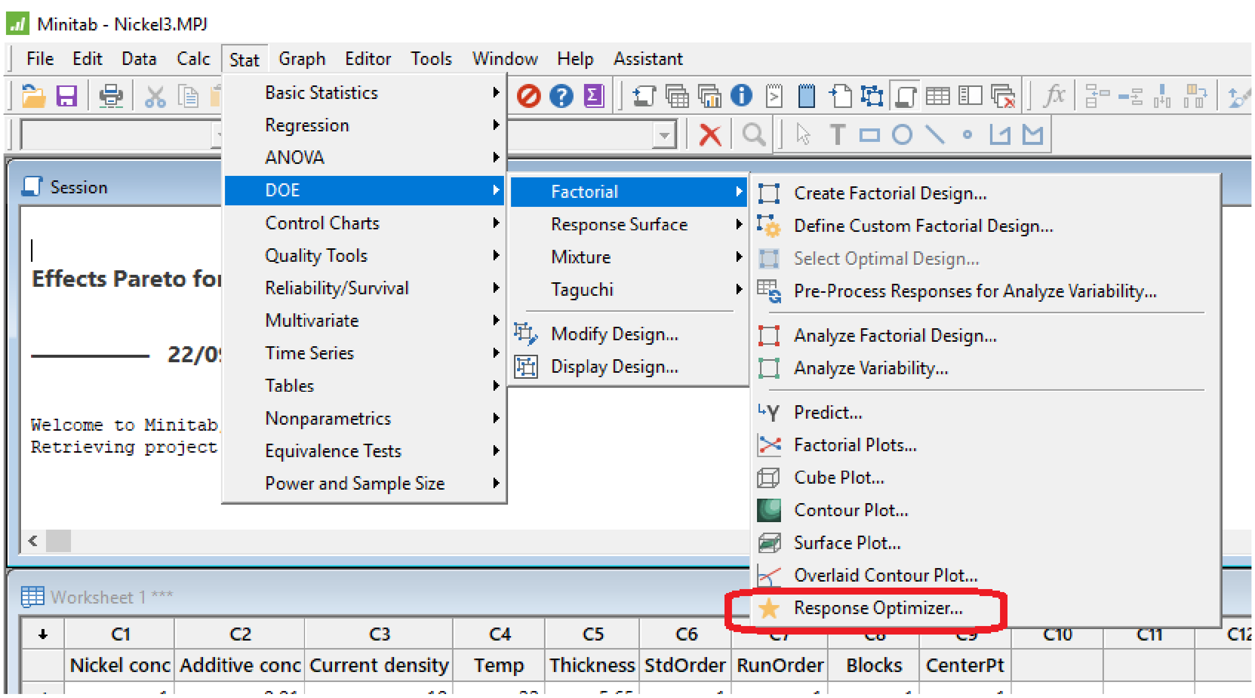

Figure S3. Response analyser.

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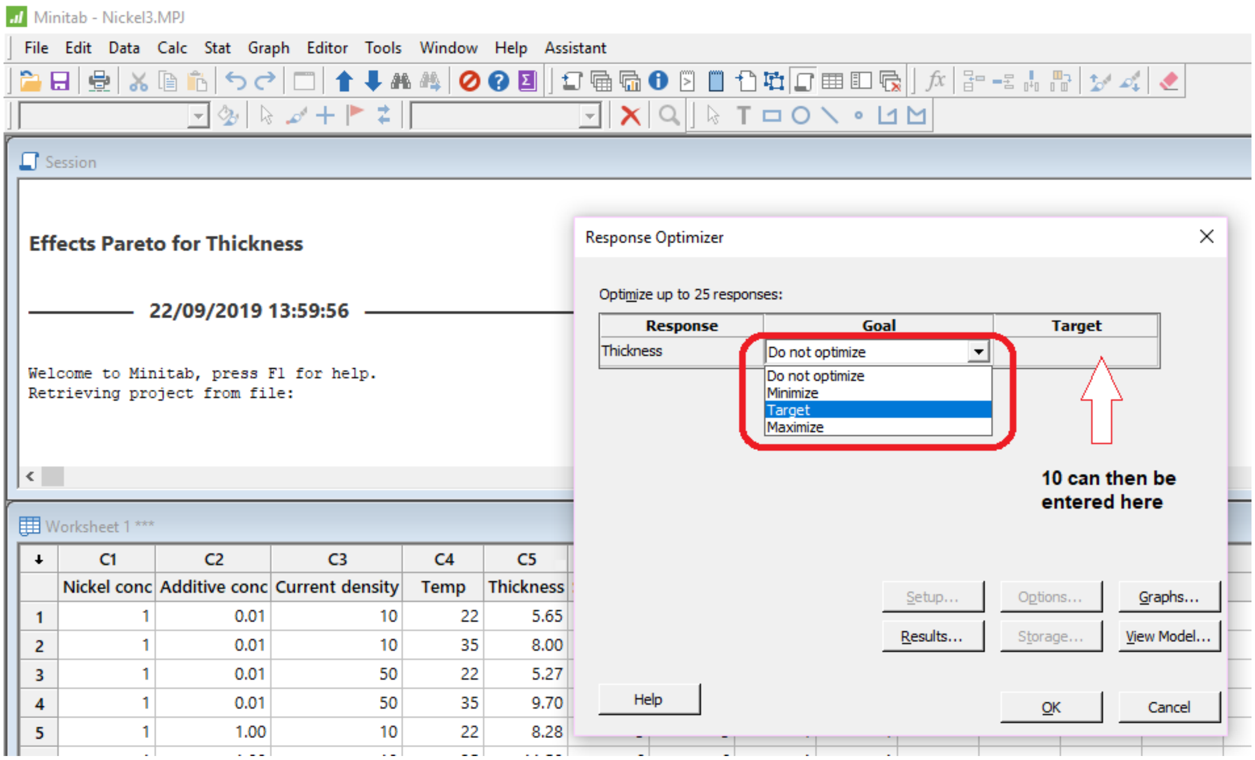

Figure S4. Set response target.