Published in final edited form as:

Int J Nanomanuf. 2009 July 1; 3(4): 351–367. doi:10.1504/IJNM.2009.027501.

Yield improvement for lost mould rapid infiltration forming process by a multistage fractional factorial split plot design

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Abstract

Statistical design of experiments is widely used among scientists and engineers to understand influential factors in a laboratory or manufacturing process. One of the underlying principles of using the statistical design of experiments method is randomisation, each run of experimental settings will be determined completely unsystematically. In practice, especially in a complicated process that consists of multiple stages, randomisation may pose too high a burden on time and cost.

In this study, the multistage fraction factorial split plot design is proposed for green yield improvement in a lost mould rapid infiltration process that has been developed to fabricate zirconia ceramic parts. This design allows a relaxation of the randomisation principle so that certain experimental runs can be carried out in convenient groups. The results indicate that the type of immersion chemical and mould coating play a role in improving process yield. Additionally, the results suggest that a mould infiltration machine should be used to improve the reproducibility of the process.

Keywords

split plot design; multistage experimentation; restricted randomisation; colloidal processing; zirconia

1 Introduction

Due to the complexity of many nanomanufacturing processes that consist of several stages, experimenters do not readily employ traditional design of experiment (DOE) techniques. The underlying assumption behind traditional DOE techniques is based on the randomisation principle. The randomisation principle refers to the concept that both the allocation of the experimental material and the order in which the individual runs or trials of the experiment are performed are randomly determined.

However, as with many nanomanufacturing applications it is impossible to execute a fully randomised design across the whole system. This is because the system is actually a multiple-stage process where factors across stages are not independent. It is difficult to change the levels of some of the factors and there are physical restrictions on the process and instrument. In such cases, the design can be treated as having a split-plot structure.

Our objective is to develop a multistage fractional factorial (FF) split plot (MSFFSP) design that is primarily used for factor screening and for process optimisation. The design is applied to improve the manufacturability of the lost mould rapid infiltration forming (LM-RIF) process that was developed by Antolino et al. (2009a, 2009b).

The organisation of this paper is as follows. Section 2 describes the LM-RIF process. Then, the topics related to split-plot design and its variants as well as the procedure to MSFFSP design are reviewed and presented in Section 3, followed by the results and analysis in Section 4. Finally, the conclusion and future research is given in Section 5.

2 LM-RIF process

The LM-RIF process, developed by Antolino et al. (2009a, 2009b) is a lithography-based lost mould approach composed of five sub processes: colloid preparation, gel-casting preparation, mould fabrication, colloid deposition and sintering. The process map is in Figure 1. For more detail see Antolino et al. (2009a, 2009b).

- Colloid preparation: Well dispersed, high solids loading slurries are needed to obtain high strength, dense ceramic parts via gel-casting. In these slurries, yttria stabilised tetragonal zirconia (Tosoh Corp. 3Y-TZP) is dispersed and concentrated by chemically-aided attrition milling (CAAM). During CAAM, the spray-dried commercial powder is added to deionised water containing ammonium polyacrylate (RT Vanderbuilt, Darvan 821A) as the dispersant, and milled using zirconia milling media. After particle size reduction is complete, the gel-casting precursor chemicals are added, along with the binder and plasticiser.
- Gel-casting preparation: Methacrylamide (Sigma-Aldrich) and N,N'-methylenebisacrylamide (Sigma-Aldrich) are used as the monomer and cross-linking agent for gel-casting in a 6:1 mass ratio. The total monomer content is 20% by mass of the water in the system. Ammonium peroxydisulfate (Sigma-Aldrich) and N',N',N',N'-tetramethylethylenediamine (Sigma-Aldrich) are used to initiate and catalyse the monomers.
- Mould fabrication: Polycrystalline alumina substrates (Kyocera Corp.) are used in order to avoid handling individual parts between processing steps. SU8 (Microchem Corp.) photoresist moulds are fabricated on top of the substrates using a modified UV lithography process. Initially, an antireflective coating of Barli-II90 is spin coated to eliminate light from scattering off of the substrate surface. Secondly, a 25 µm under layer of SU8 photoresist is spin coated to form the bottom of the mould. Finally, an

SU8 layer is spin coated on and patterned in the desired mould dimensions using a UV-photolithography approach.

- Colloid deposition: First, the gelation reaction is initiated leaving a working time of approximately 25 min. Following initiation, slurry is cast into the moulds via a screen printing squeegee at a rate of 10 cm/s. Multiple passes with the squeegee are needed to ensure complete mould filling with no entrapped air bubbles. Gelation is then allowed to complete in 100% relative humidity nitrogen environment, to both minimise drying and complete the gelation reaction.
- Sintering: Substrates are placed into a standard box furnace in ambient atmosphere where both mould removal and sintering take place. Sintered instruments can be picked and placed for further characterisation or testing, using a micromanipulator.

This process was originally intended to fabricate micro surgical instruments that are used for minimally invasive surgery (MIS). However, unsuccessful fabrication trials made it clear that the initial process could not reliably fabricate components suitable for surgical applications. Functional surgical instruments require larger lengths and cross-sections than reported by Antolino et al. (2009a, 2009b) in order to withstand the forces needed to perform surgical actions. Aspect ratios of 17:1 are standard for ASTM (2002), three point bend bars, however, surgical instrument designs require larger aspect ratios to withstand the deformation and force used in surgery. Aguirre et al. (2008) show that 400 micron thick parts with an aspect ratio of 35:1 are feasible dimensions for surgical instruments. Therefore, it is necessary to improve the process to fabricate such parts for surgical instruments by increasing the dimensions.

Specific process changes and additions were incorporated to fabricate the desired parts. First, a binder and plasticiser system was added to the slurry formulation to improve strength of the green ceramic body so that it will survive the stresses that arise during drying. Second, a solvent exchange drying technique was instituted. The water in the gelled wet parts is displaced by ethanol to minimise drying stresses due to solvent surface tension and reduce capillary forces in the pore structure of the large cross sections. Lastly, a silicone mould release layer was applied to the mould prior to infiltration to minimise part to mould wall adhesion. These new modifications were the main focus of this DOE study.

3 Experimental design methodology

The approach most commonly used to create the foundation for science and development of new manufacturing processes is overwhelmingly that of changing one factor at a time (OFAT) while attempting to maintain other variables constant. While methodical, this approach is not efficient and will miss an opportunity to recognise the interaction among variables (Giesbrecht and Gumpertz, 2004; Montgomery, 2005; Ryan, 2007). Surprisingly, this approach is still widely used among scientists and engineers (Yuangyai and Nembhard, 2009). Other traditional techniques are factorial design, FF design and Taguchi's method.

The major underlying assumption for these designs is that the randomisation principle has been applied. This means that each experimental run has been carried out completely unsystematically. In other words, there is no grouping such as or running all experiments at one process factor level followed by running all experiments at another process factor level. If the standard analysis that accompanies the traditional designs is carried out when the experiment has been conducted with such compromises, the results will be erroneous and completely wrong conclusions may be made. However, in practice that is impractical to execute or prohibitive due to cost, time and/or physical constraints. In such cases, restriction is imposed and the experiment is forced to resort to a split-plot (SP) design or some of its variants. Over the past years, much research has been focused on employing SP designs and their variants. In

this paper, a multistage FF split plot (MSFFSP) DOE is developed, presented and applied to the nanomanufacturing process, resulting in a three-stage design associated with five factors.

The name SP comes from agricultural experiments in which the whole plots are large areas of land and the subplots are small areas of land within the large area. The standard split plot design is a design which has a two-factor factorial arrangement. For example A with a levels, is designed as a completely randomised (CR) design and is called a whole plot experimental unit. Each experimental unit is divided into b split plot experimental units of the second factor named B .

In addition, there is another type of design named 'split-block', also known as strip block, SP, two-way whole plot, subtreatments in strips across blocks and criss-cross design (Federer and King, 2007). This design is bit different from the SP design. This design has two factors, A with a levels and B with b levels. The levels of factor A are randomly assigned to the whole plot experimental unit. Then, perpendicular to the a experimental units, the b experimental units are formed and the b levels of factor B are randomly allocated to the second set of b whole plot units in each of the complete blocks. Box and Jones (2000–2001) provide a description about the recognition of the completely randomised design, SP design and split-block design.

The simplest way to reduce the number of settings is to conduct the experiment under the FF design. However, in doing so, we face the inability to randomise all of the treatment combinations. The idea of fractionating the SP design was investigated by Bingham and Sitter (2001). On the other hand, Archaya and Nemhard (2008), Bisgaard and Vivacqua (2004) as well as Piniagua-Quinones and Box (2008) suggest applying the SP and split-block structure to multistage experiments. The multistage split plot (MSSP) design will significantly decrease the number of settings. Furthermore, the combination of SP and split-block structure with fractionating, the MSFFSP design, will therefore be the most efficient and effective design because it provides the smallest number of settings.

3.1 Design setup and choices of design for the LM-RIF process

There are many types of designs that can be used for this experimentation. To understand the implications in choosing these designs, we first demonstrate the different arrangements of the CR design, SP design and split-block design. Let us consider only two sub processes – gel-casting and immersing, as in Figure 2. We will assume that in the gel-casting process there are two factors of interest – percent of binder volume (A) and solid volume (B) – and that each factor has two levels. In the colloid deposition process (only the immersing stage considered), there is one factor of interest – the type of immersing chemical (E) – and this factor has two levels. The response (y) is the green state yield.

If a CR design is used, eight samples are prepared at different times and each sample must be placed into a furnace at different times (see Figure 3a). If the SP design is used, there are only four sample preparations required. Each sample is split into two sub-samples. Then each sub-sample is placed into the immersing bath at a different time. Therefore, there are four sample preparations and eight immersing settings [see Figure 3(b)].

In the split-block design, only four samples are prepared. Each is split into two sub-samples, similar to those in SP design. However, these sub-samples are then regrouped and placed into the bath at the low level or the high level 'together'. This reduces the sintering settings from eight to only two [Figure 3(c)].

3.2 MSFFSP design with the three-stage experimentation

The objective of this study is to understand how each of the five factors identified in Table 1 affects the process yield. The factors and levels were chosen in consultation with the scientists

who developed the process. Note that we alternate between the use of capital letters and small letters to distinguish the factors among stages. Also note that the Stage 2 and Stage 3 are both in the colloid deposition process.

Let us reconsider the three-stage problem with five factors and similarly analyse our position. Table 2 shows that the CR design requires a total of $2^5 = 32$ runs, which means 32 settings for all factors in each stage, are required. When the MSSP design, both SP and split-block arrangement, is used, the number of settings is reduced. Particularly in the case of the split-block arrangement for all stages, this provides the lowest number settings that are equal to the number of treatment combinations applied to experimental units at each stage. Note, however, that the number of runs remains at 32.

The simplest way to reduce the number of runs is to conduct the experiment under the FF design. Assuming, for example, a 1/4 fraction, the number of runs would be decreased from 32 to $2^{5-2} = 8$, as shown in Table 2. However, this design still requires the ability to completely randomise all the treatment combinations.

In this research, it was recognised that in some situations it is not practical to directly employ SP or split-block structure. For example, it is practical to perform experiments from Stage 1 through Stage 2 (particle preparation through mould surface coating) and then regroup experimental units in the immersing process at different immersing solutions. If regrouping is done after Stage 1 (particle preparation), all experiment units after this stage have to be filled in the same substrates which is physically impossible. The combination of SP and split-block structure with the MSFFSP design will be the most efficient and effective design under such constraints. In our application, it is preferable that experimentation under Stages 1 and 2 is performed with SP structure but in Stage 3 split-block structure is performed.

Based upon an experimental ‘budget’ of eight runs, since this research focuses on fractional design, it is important to review some recent methods of factor confounding in multistage experiments suggested by Bisgaard (2000). Based on the existing literature, we show, in Table 3, the factor confounding using the two-stage FFSP designs for Stages 1 and 2 which involve factors *A*, *B*, *C* and *d*. This design can be represented by a $2^{3-1} \times 2^1$ FFSP design.

This fractionation in the MSSP design can be classified into two types – confounding ‘within stages’ and confounding ‘between stages’. The generator for the within stage design is $I = ABC$. This design provides overall and partial resolution III, which indicates that there is no confounding between stages. However, the main effects and interaction effects within a stage are confounded. If the objective of this experiment is to determine the main effects, this design will not be very helpful. Table 3 shows the design tables for the two-stage experiment with different confounding strategies. The factor settings for each run follow the typical rules of DOE.

On the other hand, in order to maintain the highest resolution of the design, the fractionation in each stage could be confounded between stages with the highest interaction effect of other stage factors. Its design generator is $I = ABC$. This design provides an overall resolution IV which indicates the main effects are not confounded with any second order interaction.

However, in this experimentation, if we employ the between stage confounding factor, for example $I = Abd = BCE = ACdE$, the split plot structure of the experimentation will be destroyed. This destruction means eight particle preparations are required, which after discussing with the manufacturing scientists was deemed experimentally impractical. Therefore, we decided to use $I = ABC = BdE = ACdE$. This generator provides a resolution III design as shown in Table 4. The design format is shown in Table 5.

The experimental procedure is described in Figure 4. In Stage 1, four units of particles are applied with four treatment combinations (yellow-shaded) of factors *A*, *B* and *C*. Then, each unit is split into two groups for two treatment combinations (green-shaded) of Stage 2 factors (*d*). After that, in Stage 3, each group is regrouped and then immersed in the ethanol or toluene (blue-shaded). The number labelled in each block refers to the experiment unit number, for example label 112 indicates that this experimental unit is performed with treatment combination no. 1 from Stage 1, with treatment combination no. 2 from Stage 2, with treatment combination no. 2 from Stage 3).

With the combination of SP and split-block structure, the numbers of settings for each type of design are shown in Table 6. Note that the lowest number of settings in each stage is obtained when only the split-block structure is employed.

3.3 Linear model

The linear model is useful to describe the observational data from an experiment. In order to construct a linear model for the MSFFSP design, an understanding of the linear models in the SP and split-block design is required, found in Federer and King (2007). For the experimentation, we employ a combination of SP structure in the first two stages and split-block structure in the last stage. Assuming that there are *a* levels of a Stage 1 factor, *b* level of a Stage 2 factor, *c* levels of Stage 3 and *n* replicates, the linear model for this design structure is,

$$y_{ghij} = \mu + \rho_g + \alpha_h + \varepsilon_{gh}^{s_1} + \beta_i + \alpha\beta_{hi} + \varepsilon_{ghi}^{s_2} + \gamma_j + \varepsilon_{gj}^{s_3} + \alpha\gamma_{hj} + \varepsilon_{ghj}^{s_1 \& s_3} + \beta\gamma_j + \varepsilon_{gij}^{s_2 \& s_3} + \alpha\beta\gamma_{hij} + \varepsilon_{ghij}^{s_1 \& s_2 \& s_3} \quad (1)$$

where

y_{ghij} the $ghij$ th response of the experiment

μ a general overall mean effect

ρ_g the g th replicate effect $\sim \text{iid } N(0, \sigma_\rho^2)$

α_h the effect of h th level of Stage 1 factor

$\varepsilon_{gh}^{s_1}$ the gh th random error effect $\sim \text{iid } N(0, \sigma_{\varepsilon^{s_1}}^2)$

β_i the effect of i th level of Stage 2 factor

$\alpha\beta_{hi}$ the interaction effect of h th combination of Stages 1 and 2 factor

$\varepsilon_{ghi}^{s_2}$ the gh th random error effect $\sim \text{iid } N(0, \sigma_{\varepsilon^{s_2}}^2)$

γ_j the effect of j th level of Stage 3 factor

$\varepsilon_{gj}^{s_3}$ the g th random error effect $\sim \text{iid } N(0, \sigma_{\varepsilon^{s_3}}^2)$

$\alpha\gamma_{hj}$ the interaction effect of h th combination of Stages 1 and 3 factor

$\varepsilon_{ghj}^{s_1 \& s_3}$ the ghj th random error effect $\sim \text{iid } N(0, \sigma_{\varepsilon^{s_1 \& s_3}}^2)$

$\beta\gamma_{ij}$ the interaction effect of ij th combination of Stages 2 and 3 factor

$\varepsilon_{gij}^{s_2 \& s_3}$ the gij th random error effect $\sim \text{iid } N(0, \sigma_{\varepsilon^{s_2 \& s_3}}^2)$

$\alpha\beta\gamma_{hij}$ the interaction effect of hij th combination of Stages 1, 2 and 3 factor

$\varepsilon_{ghij}^{s_1 \& s_2 \& s_3}$ the $ghij$ th random error effect $\sim \text{iid } N(0, \sigma_{\varepsilon^{s_1 \& s_2 \& s_3}}^2)$

All errors are mutually independent.

Note that the superscript of each error term indicates the stage number and its interaction with factors from other stages, for example, $\varepsilon_{ghij}^{s_1 \& s_2 \& s_3}$ is the error term due to the interaction among factors from Stage 1, Stage 2 and Stage 3.

The linear model indicates that there are six error terms in total, the first three error terms correspond to effects of the first three stage factors, respectively. While the other three error terms correspond to interaction effects among Stage 1 and Stage 2, Stage 2 and Stage 3 and Stage 1, Stage 2 and Stage 3. From the linear model displayed in equation 1, there are six error terms in total. It is important to know which contrasts will be tested against which error term in order to test for significance. In addition, this linear model is generalised and can also be used for the MSSP design and the MSFFSP design with or without replication.

3.4 MSFFSP design implementation for the LMRIF process

In our experimentation, if the design without replications is used, Table 7 shows the contrasts having the same error. However, since this is a fractionated design, some contrasts are confounded with others, based on the generator, the effects must be properly assigned to appropriate errors, as summarised in Table 7. Note that only main effects and the second-order interactions are considered. Normality probability plots are used to analyse the results. There are four plots required to test the significance of effects A, B, C, d, Ad, E and AE . However, we do not have enough effects to construct the plots because the maximum effect for each error term is three.

In order to overcome this situation, there are two possible methods: increase the level of fractionation or perform another replication. For the first method, another eight runs with different treatment combinations is required with a new generator. In the second method, we need to replicate the design. After discussion among the design members and manufacturing scientists, we decided to replicate the design because this replication permits estimation of the experimental errors as well as lending itself to easier analyses.

4 Results and discussion

The results of the experiment, shown in Table 8, were analysed using Minitab software (general linear model). All mean square errors have been calculated with selected effects regrouped and reorganised with the associated error terms based on Table 7.

Table 9 shows the reorganised ANOVA table. The effects of A, B, C are tested with $\varepsilon_{gh}^{s_1}$ (Error 1) derived from the summation of sum square errors of interaction of factor A and replication, sum square errors of the interaction of factor B and replication, as well as the sum square of interaction of factor C and replication. Due to the nature of fractional design, the interaction of A, B, C and replication is not estimable. However, in general, if the full factorial design is used, the effects of A, B and C will be tested with the interaction of A, B, C and replication.

The effects of factor d , E and AE are tested against $\epsilon_{ghi}^{s_2}$ (interaction of d and replication), $\epsilon_{gj}^{s_3}$ (interaction of E and replication) and $\epsilon_{ghj}^{s_1 \& s_3}$ (interaction of A , E and replicate), respectively, and the replication is tested with $\epsilon_{gh}^{s_1}$. Note that we are not able to estimate $\epsilon_{gij}^{s_2 \& s_3}$ and $\epsilon_{ghij}^{s_1 \& s_2 \& s_3}$ because we do not have enough degrees of freedom.

From the ANOVA table, there is a significant difference between replications as we can observe from the large ratio of $MS_{Rep}/MS_{Error} = 6.90$. Note that, in this case due to the restriction on randomisation of replications, the F test is not valid so the ratio is used instead of F test [for more details, see Montgomery, (2005), p.123]. The reason behind this significance is that the both replicates were done by different operators who have different skill levels in mould filling. The operator no. 2 (Replication 2) outperforms the operator no. 1 (Replication 1). In order to improve the reproducibility of the process, the scientists agree to investigate an automatic filling machine, eliminating human error from the process.

In addition, the type of immersing chemical (p-value = 0.225), as well as the mould surface coating factor is significant (p-value = 0.274). Although these p-values are slightly high compared to the general acceptance level (0.05 or 0.10), this level should not be automatically used as a decision criteria without experimenters' judgement [Box et al., (2005), p.188]. In our results, we believe that this information combined with our scientific knowledge of the LM-RIF process is still valid for further improvement.

The immersing solution is used in the process because a solvent exchange drying process is employed to minimise drying stress and improve yield. Water saturated, gelled, green parts are placed into an ethanol bath for four hours, during which solvent exchange takes place. After four hours, the parts are dried in the ambient atmosphere. Ethanol was chosen because of its miscibility with water as well as a low surface tension. The lower surface tension reduces the capillary drying stresses in the porous parts and minimises cracking.

The silicone mould surface coating was selected for this process to ensure a non-wetting surface for our aqueous based slurries, as well as provide lubrication between the green parts and the mould walls. As the parts dry, they shrink away from the mould walls. If green parts adhere to the mould walls, the part experiences increased stress during drying and cracking can occur. The main effect plots of the immersing solution, coating effect and block factors are shown in Figure 5. In addition, from the experiment analysis, we know which factors are significant. This knowledge will be used to further optimise the process.

If we use the ANOVA technique based on the CR design as shown in Table 10, the incorrect interpretation is obtained. That means only factor B and Ad seems to be insignificant (p-value = 0.956 and 0.743, respectively). This is because the estimated sum square of the error is very low.

5 Conclusions and future research

The concepts presented in this study address the challenges of multistage FFSP experiments in nanomanufacturing. The LM-RIF forming process conducted over three stages was studied some properties and characteristics of MFFSP structure are presented. Through these designs, we illustrated how DOE techniques can be used in nanomanufacturing design and engineering by aiding manufacturing development scientists understand process dynamics.

These multistage designs overcome the severe limitations of the commonly used SP designs by providing greater flexibility in the choice of confounding patterns. In addition, multistage

designs require fewer runs than SP designs, while yielding greater information about the factors of interest.

In this study, only the processes in series are considered; however, the mould fabrication process takes place in parallel with the four processes. Therefore, adding this process in parallel to the MSFFSP design will be studied in future work. In addition, we will continue extending the MSFFSP design to more general cases.

In summary, using these designs and integrating them into the modelling and production for manufacturing research will yield strategic advantages by speeding the research and development cycle, stretching the experimental budget and helping to create more reliable, robust and better performing products. It is also believed that as advanced nanotechnology applications are explored with experimental design, there will be new questions that call for modifications or perhaps completely new constructs of experimental designs that will simultaneously advance the field of DOE.

Acknowledgments

The research was supported by grant number CMMI 0800122 from the National Science Foundation and by grant number R21EB006488 from the National Institute of Biomedical Imaging and Bioengineering. The content is solely the responsibility of the authors and does not necessarily represent the official views of the National Institute of Biomedical Imaging and Bioengineering or the National Institutes of Health. This publication was also supported by the Pennsylvania State University Materials Research Institute NanoFabrication Network and the National Science Foundation Cooperative Agreement No. 0335765, National Nanotechnology Infrastructure Network, with Cornell University. Chumpol Yuangyai gratefully acknowledges a fellowship from the Royal Thai Government.

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Biographies

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Figure 1.

The lost mould rapid infiltration process developed by Antolino et al. (2009a, 2009b) for the nanomanufacturing of mesoscale ceramic components

Figure 2.
Two-stage process

Figure 3.
(a) CR (b) SP (c) split block design arrangements (see online version for colours)

Figure 4.
Illustration of three-stage experimentation (see online version for colours)

Figure 5.
Main effects plots

Table 1

Factors of interest

Process	Stage	Factors of interest	Levels	
			Low (−1)	High (+1)
Powder preparation	1	A Powder preparation	8	10
		B Solid volume (%)	35	40
Colloid deposition	2	C Binder ratio (PEG:PVA)	1:1	1:1.5
	3	d Surface coating	No	Yes
		E Immersion solution	Ethanol	Toluene

Table 2

Number of settings and number of runs in CR, FF and MSSP design

Design	Number of settings in			Number of runs
	Stage 1: powder preparation	Stage 2: gel-casting	Stage 3: immersing	
CR	32	32	32	32
MSSP ($2^3 \times 2^1 \times 2^1$)	8	16	32	32
FF (2^{5-2})	8	2	2	32
	8	8	8	8

Table 3

Design for a two stage experiment

(a) Confounding within stages					(b) Confounding between stages				
Run	Factor				Run	Factor			
	Stage 1		Stage 2			Stage 1		Stage 2	
	A	B	C = AB	d		A	B	C	d = ABC
1	-1	-1	1	-1	1	-1	-1	-1	
2	-1	-1	1	1	2	-1	-1	1	
3	-1	1	-1	-1	3	1	-1	1	
4	-1	1	-1	1	4	-1	1	-1	
5	1	-1	-1	-1	5	1	-1	1	
6	1	-1	-1	1	6	-1	1	-1	
7	1	1	1	-1	7	1	1	-1	
8	1	1	1	1	8	1	1	1	

Table 4

Factor confounding

I = ABC = BdE = ACdE
A + BC + CdE + ABdE
B + AC + dE + ABCdE
C + AB + AdE + BCdE
d + BE + ACE + ABCd
E + Bd + ACd + ABCE
Ad + CE + ABE + BCd
AE + Cd + ABd + BCE

Table 5

Design table

Run	Factors				
	A	B	C=AB	d=BE	E
1	-1	-1	1	1	-1
2	-1	-1	1	-1	1
3	-1	1	-1	-1	-1
4	-1	1	-1	1	1
5	1	-1	-1	1	-1
6	1	-1	-1	-1	1
7	1	1	1	-1	-1
8	1	1	1	1	1

Table 6

Number of settings in the MSFFSP design

Design	Number of settings in			Number of runs
	Stage 1: particle preparation	Stage 2: gel-casting	Stage 3: immersing	
MSFFSP ($2^{3-1} \times 2^1 \times 1$)SP	4	8	8	8
Split-block	4	2	2	8
Combination of SP and split-block	4	8	2	8

Table 7

Contrast effects and their error structure: MSSP design, only main and second-order

No	Error term	Contrast
1	s_1 ε_{gh}	A, B, C
2	s_2 ε_{ghi}	d, Ad
3	s_3 ε_{gj}	E
4	$s_1 \& s_3$ ε_{ghj}	AE
5	$s_2 \& s_3$ ε_{gij}	None
6	$s_1 \& s_2 \& s_3$ ε_{ghij}	None

Table 8

Experimentation results

Run no.	Stage 1		Stage 2		Stage 3		% Yield	
	Binder volume	Solid volume	Binder ratio	Surface coating	Immersion chemical		Rep1	Rep2
1	8	35%	1:1.5	No	Toluene		16.7	72.2
2	8	35%	1:1.5	Yes	Ethanol		87.5	100
3	8	40%	1:1	No	Ethanol		37.5	98.6
4	8	40%	1:1	Yes	Toluene		16.7	80.0
5	10	35%	1:1	No	Toluene		0.0	59.7
6	10	35%	1:1	Yes	Ethanol		75.0	94.4
7	10	40%	1:1.5	No	Ethanol		52.8	97.2
8	10	40%	1:1.5	Yes	Toluene		31.9	76.4

Table 9

ANOVA table

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Rep	1	8,831.3	8,831.3	8,831.3	6.902149*	
A	1	87	87	87	0.344464	0.599
B	1	0.1	0.1	0.1	0.000396	0.985
C	1	205.2	205.2	205.2	0.812459	0.434
Error 1 (A * Rep + B * Rep+ C * Rep)	3	757.7	757.7	352.8		
D	1	1,272.7	1,272.7	1,272.7	4.747109	0.274
A * d	1	2.8	2.8	2.8	3.684136	0.306
Error 2 (d * Rep)	1	268.1	268.1	268.1		
E	1	4,688.8	4,688.8	4,688.8	7.340013	0.225
Error3 (E * Rep)	1	638.8	638.8	638.8		
A * E	1	52.2	52.2	52.2	0.189655	0.851
Error	2	39.6	39.6	19.8		
Total	15	16,844.3				

* Note: It is the ratio of MSRep/MSError

Table 10

ANOVA in the case of CR design

Source	DF	Seq SS	Adj SS	Adj MS	F	P
A	1	87	87	87	4.39	0.171
B	1	0.1	0.1	0.1	0	0.956
C	1	205.2	205.2	205.2	10.36	0.085
d	1	1,272.7	1,272.7	1,272.7	64.23	0.015
E	1	4,688.8	4,688.8	4,688.8	236.62	0.004
Rep	1	8,831.3	8,831.3	8,831.3	445.67	0.002
A*d	1	2.8	2.8	2.8	0.14	0.743
A*e	1	52.2	52.2	52.2	2.63	0.246
A*Rep	1	99.5	99.5	99.5	5.02	0.154
B*Rep	1	417.2	417.2	417.2	21.05	0.044
C*Rep	1	241	241	241	12.16	0.073
d*Rep	1	268.1	268.1	268.1	13.53	0.067
E*Rep	1	638.8	638.8	638.8	32.24	0.03
Error	2	39.6				
Total	15	16,844.4		19.8		