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The Design and Analysis for the Icing Wind Tunnel Experiment of a New Deicing Coating

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ABSTRACT
A new kind of deicing coating is developed to provide aircraft with efficient and durable protection from icing-induced dangers. The icing wind tunnel experiment is indispensable in confirming the usefulness of a deicing coating. Due to the high cost of each batch relative to the available budget, an efficient design of the icing wind tunnel experiment is crucial. The challenges in designing this experiment are multi-fold. It involves between-block factors and within-block factors, incomplete blocking with random effects, related factors, hard-to-change factors, and nuisance factors. Traditional designs and theories cannot be directly applied. To overcome these challenges, we propose using a step-by-step design strategy that includes applying a cross array structure for between-block factors and within-block factors, a group of balanced conditions for optimizing incomplete blocking, a run order method to achieve the minimum number of level changes for hard-to-change factors, and a zero aliased matrix for the nuisance factors. New (theoretical) results for D-optimal design of incomplete blocking experiments with random block effects and minimum number of level changes are obtained. Results of the experiments show that this novel deicing coating is promising in offering both high efficiency of ice reduction and a long service lifetime. The methodology proposed here is generalizable to other applications that involve nonstandard design problems. Supplementary materials for this article are available online.

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Cross array; D-optimal design; Incomplete block design; Minimum number of level changes; Random block effect

1. Introduction

Aircraft icing occurs when water droplets in the atmosphere freeze on the airframe. It may increase drag, decrease lift, and cause control problems. As a result, ice protection systems are of great importance to ensure safe flights. Among many kinds of deicing methods, deicing coatings prove to be convenient and effective in preventing ice accumulation. Existing deicing coatings such as electro-thermal coatings (Huneault, Langheit, and Caron 2005; Liao et al. 2007), opto-thermal coatings (Miles 2000), super-hydrophobic coatings (Saito, Takai, and Yamauchi 1997; Menini and Farzaneh 2009; Kulinich and Farzaneh 2009; Mishchenko et al. 2010), and coatings containing slowly released freezing point depressants or lubricating oils (Simendinger 2004; Ayres, Simendinger, and Balik 2007) suffer from problems such as low icephobicity (the ability of a solid surface to repel ice or prevent ice formation), flashover (abnormal electrical discharge) of the polymer matrices, and severe aging.

1.1. Icing Wind Tunnel: Experiment and Materials

A new kind of coating has been developed to apply to the aircraft surface in providing efficient and durable solution to the aircraft icing problem. The deicing coating is made of silicone elastomer and alkanes (Guan et al. 2010). It takes advantage of the phase transition of alkanes in silicone elastomer with changing temperature to achieve the deicing property. When the temperature drops below the freezing point of the alkane, the alkane molecules migrate to the surface of the coating and form a thin layer on the surface. On the other hand, when the temperature is raised above the melting point of the alkane, the alkane is redissolved into the coating. Since the alkane layer on the surface reduces ice accumulation on the coating, it is postulated to be icephobic. In addition, lab centrifuge experiments have shown that the adhesive force between the accumulated ice and the coating is largely reduced when compared to the force between ice and pure aircraft surfaces such as aluminum. Therefore, accumulated ice tends to break and fall off under natural vibrations during flight, which further enhances the deicing property of the coating.

After repeated use, it is likely that some alkanes that have diffused to the coating surface have been removed with ice that fell off of the coating. Therefore, it is desirable to incorporate a mechanism that ensures slow release of alkanes to maintain the coating's long-term capabilities. Lab experimental data from repetitive scrape tests show that an addition of montmorillonoid nanoparticles into the coating may slow the speed of migration for alkane molecules and therefore may have the potential to extend the service lifetime of the coating.

However, aircraft icing is a very complex process that depends on atmospheric conditions such as temperature, amount, and droplet size of water in the air as well as the moving speed of aircraft. As a result, in addition to lab experiments,

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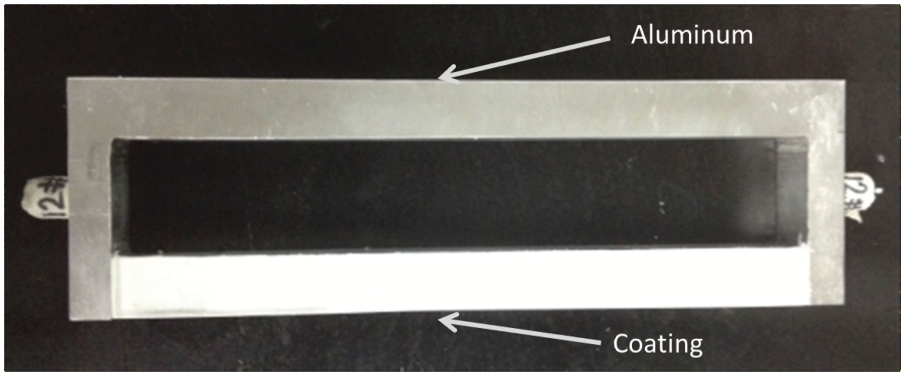

Figure 1. Experiment unit used in the icing wind tunnel. In the icing wind tunnel, the unit is installed in an upright position. To save space, the figure shows the unit horizontally.

icing wind tunnel experiments are required to confirm the usefulness of the coating under real atmospheric conditions. In an icing wind tunnel, supercooled water (i.e., liquid water below 0 degree centigrade, the cause for aircraft icing) is sprayed with certain speed and temperature and travel through the tunnel. An experimental unit (hereafter referred to as a "unit") is placed in the center of the tunnel with two cylinder-shaped fixtures attached to the top and bottom of the tunnel. Figure 1 shows such a unit in a horizontal position. The unit has a concentric rectangular shape on the windward face, with the left and/or right side applied with coatings. On the leeward face, one vibrator is attached to each side and the two vibrators may be simultaneously turned on or off. Each time supercooled water travels through the tunnel for a specified time period to allow ice formation on the unit, and the weight of the accumulated ice on each side of the unit is measured, which is treated as the response variable in this study. Measurements are thus obtained in pairs, one from each side of the unit. A pair of measurements is referred to as a "batch" hereafter, and the individual measurements on each side of the unit are referred to as "trials." As a result, each batch consists of two trials.

1.2. Objectives

The primary goal is to evaluate the usefulness of the coating in reducing ice accumulation and to investigate whether the addition of nanoparticles affects (either increases or decreases) ice accumulation. The secondary goal is to assess the effects of temperature, wind speed, vibration, repeated usage of the coating, and their interactions if possible. The cost for each batch of the icing wind tunnel experiment is high ($\approx$1600 USD per batch). Four preliminary screening batches have been collected to explore the possible composition for the coating. The current study has a budget of no more than 20 batches and, as a result, an efficient design is indispensable.

1.3. Relevant Literature

Two trials are conducted simultaneously within one batch of the icing wind tunnel experiment. A random block effect may exist, and as such, an efficient design is needed to address this concern. Throughout the article, "block" and "batch" are used interchangeably to denote the group of two trials conducted during one icing wind tunnel experiment.

Optimal block designs have been extensively studied for treatment comparison, for example, in Shah and Sinha (1989), Cheng (1995), and Atkins and Cheng (1999). However, such designs are not applicable to cases with both between-block factors (e.g., temperature, wind speed, and vibration) and multiple within-block factors (e.g., whether and which coating is applied, whether nanoparticle is added), as in the icing wind tunnel experiment.

Balanced incomplete block designs (BIBD) and partially balanced incomplete block designs (PBIBD; Bose 1939; Bose and Nair 1939; Cochran and Cox 1957) have been adopted for incomplete blocking. However, the conditions required by BIBDs cannot be satisfied in the icing wind tunnel experiment, and the treatments of this experiment do not have multiple classes of associates as the treatments in PBIBD. Alternatively, D-optimal designs for split plot experiments can deal with correlated estimates. Iterative algorithms can be used to obtain these designs. See, for example, Goos and Vandebroek (2003), Goos (2006), Johns and Goos (2009), Macharia and Goos (2010), and Johns and Goos (2012). However, these algorithms cannot be directly applied to the icing wind tunnel experiment due to the complicated restrictions. As a result, new methods for constructing an efficient block design are required.

Moreover, a few other features add to the complexity of the icing wind tunnel experiment. As will be further explained in Section 2, we need to imbed preliminary screening batches (prior to the whole design) into the whole design, take care of related factors, adjust the run order to facilitate a minimum number of level changes for hard-to-change factors (Ju and Lucas 2002), and retain a parsimonious model under the presence of nuisance factors. To the best of our knowledge, there is no standard design that is able to address all of these aspects.

1.4. The Proposed Design

In this article, we propose a framework for constructing an efficient design for the icing wind tunnel experiment. The framework can be generalized to other experiments that have similar challenges.

The design involves three between-block factors, two within-block factors, and incomplete blocks. We adopt a cross array, where the outer array is used for the between-block factors, and the inner array is used for the within-block factors. To deal with the incomplete blocks, we introduce the notion of "group balance," which is weaker than the condition from BIBD. Together with the cross array structure, it guarantees uncorrelated estimates of the fixed effects with the presence of random effects. This, in turn, facilitates a high D-efficiency of our design. Additionally, the run order of our design facilitates the minimum number of level changes for two hard-to-change factors; and the nuisance factors are designed to be orthogonal to the factors of interest.

The rest of the article is as follows. Section 2 introduces the proposed design. The details to construct this design are provided in Section 3. In Section 4, an analysis of the experimental results is provided. The results show that the deicing coating offers both high efficiency in ice reduction and a long service lifetime. In Section 5, theoretical properties of the design and general applications are discussed. In Section 6, we conclude the article with some final remarks and discussions. The general methodology proposed here provides a standard approach to nonstandard design problem.

2. The Design of Experiments

In this section, we provide our design for the icing wind tunnel experiment. Factors of the icing wind tunnel experiment are shown in Table 1.

F_1–F_3 are environmental factors of our secondary consideration. F_1 is the average temperature we try to hold; F_2 is the wind speed in the icing wind tunnel; F_3 is whether the vibrators are turned off or turned on. For two trials in the same batch, the vibrators are either both on or off. Each unit is tested in two batches, which differ with respect to at least one between-block factor (e.g., wind speed, temperature, vibration). F_4 denotes whether it is the initial batch or the repeated batch and is used to evaluate the degradation of the coating under repeated usage.

The three levels for F_5 are: B_0 (Aluminum only), B_1 (Elastomer silicone coating with 10% methylsilicone oil, 10% hexadecane, and 10% octadecane), and B_2 (Elastomer silicone coating with 10% methylsilicone oil and 20% hexadecane). When compared to B_0, both B_1 and B_2 showed superior properties in lab centrifuge test. For F_6, the montmorillonoid nanoparticles are either added or not added into the coating. Addition of the montmorillonoid nanoparticles can lengthen the lifetime for the coating and enhance its mechanical properties. However, it is also possible that nanoparticles slow the speed of alkane migration such that they affect the icephobic property of the coating. It is hoped that addition of nanoparticles does not significantly increase ice accumulation. Note that F_6 is only applicable when F_5 is B_1 or B_2. We call F_5 and F_6 related factors because they take value dependently. There are five possible combinations for F_5 and F_6, namely, $\{B_0, B_1^-, B_1^+, B_2^-, B_2^+\}$, in which the subscript denotes the level of F_5 and the superscript denotes the level of F_6 when applicable.

The factors labeled F_7 to F_9 take into consideration the direction when installing the unit (which side is facing the wind, left or right) and information on the process of measurement (which side of the unit is measured first). While the weight of the ice is of primary interest, the experimenters also recorded the shape of the accumulated ice for each trial; although this information is not analyzed below, the process of recording the shape may affect the final recorded ice weight, and therefore this should be accounted for in the design. Factors F_7 to F_9 are nuisance factors ancillary to our interest.

Due to constraints of the experiment, it is apparent that the levels of F_1 to F_4 must be exactly the same and the levels of F_7 to F_9 must be the opposite for the two trials under the same batch. Other challenges and considerations for the design of this study are listed below.

- Four preliminary batches as shown in the upper part of Table 2 should be imbedded into the whole design for cost saving purposes.
- For the two trials within the same batch, within-block factors F_5 and F_6 can be varied. Each trial can be installed with one of the five possible combinations of F_5 and F_6: $\{B_0, B_1^-, B_1^+, B_2^-, B_2^+\}$. Hence, it is an incomplete block design problem of block size of two and treatment level of five.
- The ease with changing the levels of different factors is not the same. In particular, temperature is the hardest to change, wind speed comes second, and other factors are relatively easy to change. Thus, it makes sense to design an experiment such that the level changes for temperature and speed are as infrequent as possible.
- There are three nuisance factors F_7 to F_9. The inference about factors F_1 to F_6 should be robust to the impact of F_7 to F_9.

With all of the above challenges in mind, it is apparent that the design should not be constructed with currently available software. Particularly, we handle the related factors F_5 and F_6 by introducing four orthogonal contrasts in Section 4 and Table 6. We design the icing wind tunnel experiment by applying the following steps. Details of our design will be discussed in Section 3.

Step 1. Plan a cross array for factors F_1–F_3 and F_5–F_6, where a $2 \times 2 \times 2$ array is the design for F_1–F_3, called the outer array. The inner array for F_5 and F_6, which is combined with each row of the outer array, is explained in Section 3.1.

Step 2. Group trials into blocks (batches in this case). The grouping scheme for the batches should also coincide with the design of the preliminary batches.

Table 1. The factors in the study.

Factor	Level	Classification
F_1 (Temperature)	−11°C (−), −7°C (+)	Between-block, hard to change
F_2 (Wind speed)	20 m/s (−), 30 m/s (+)	Between-block, hard to change
F_3 (Vibration)	off (−), on (+)	Between-block, easy to change
F_4 (Repetition)	initial (−), repeated (+)	Between block, easy to change
F_5 (Icing surface)	B_0*, B_1**, B_2***	Within-block, easy to change
F_6 (Nanoparticles)	not added (−), added (+)	Within-block, easy to change
F_7 (Side of the unit)	right (−), left (+)	Nuisance factor, easy to change
F_8 (Measuring weight)	first (−), second (+)	Nuisance factor, easy to change
F_9 (Measuring shape)	first (−), second (+)	Nuisance factor, easy to change
Batch	{1, 2, . . . , 24}	Random effect

*B_0: Aluminum only.
**B_1: Elastomer silicone coating with 10% methylsilicone oil, 10% hexadecane, and 10% octadecane.
***B_2: Elastomer silicone coating with 10% methylsilicone oil and 20% hexadecane.

Table 2. The 24 batches of the proposed design.

Unit	Batch	F_1	F_2	F_3	F_4	F_5 & F_6	$F_7(+)$	$F_8(+)$	$F_9(+)$
U1	P1	+	−	−	−	B_0, B_1^-	B_0	B_0	B_1^-
U2	P2	+	+	−	−	B_0, B_1^-	B_0	B_1^-	B_0
U1	P3	−	−	−	+	B_0, B_1^-	B_0	B_0	B_0
U2	P4	−	+	−	+	B_0, B_1^-	B_1^-	B_1^-	B_0
1	1	+	+	−	−	B_0, B_1^+	B_1^+	B_0	B_1^+
3	2	+	+	+	−	B_0, B_2^-	B_2^-	B_0	B_2^-
5	3	+	+	+	−	B_0, B_2^+	B_0	B_2^+	B_0
7	4	+	−	+	−	B_1^-, B_2^-	B_2^-	B_2^-	B_2^-
8	5	+	−	−	−	B_1^+, B_2^+	B_1^+	B_1^+	B_2^+
1	6	+	−	+	+	B_0, B_1^+	B_1^+	B_1^+	B_1^+
3	7	+	−	−	+	B_0, B_2^-	B_2^-	B_2^-	B_0
5	8	+	−	+	+	B_0, B_2^+	B_0	B_0	B_0
2	9	−	−	+	−	B_0, B_1^+	B_0	B_1^+	B_1^+
4	10	−	−	−	−	B_0, B_2^-	B_2^-	B_2^-	B_2^-
6	11	−	−	+	−	B_0, B_2^+	B_2^+	B_0	B_0
7	12	−	−	+	+	B_1^-, B_2^-	B_1^-	B_2^-	B_2^-
8	13	−	−	−	+	B_1^+, B_2^+	B_1^+	B_1^+	B_1^+
9	14	−	+	+	−	B_1^-, B_1^+	B_1^+	B_1^-	B_1^-
10	15	−	+	−	−	B_2^-, B_2^+	B_2^+	B_2^+	B_2^-
2	16	−	+	−	+	B_0, B_1^+	B_0	B_0	B_1^+
4	17	−	+	+	+	B_0, B_2^-	B_2^-	B_0	B_0
6	18	−	+	+	+	B_0, B_2^+	B_0	B_2^+	B_2^+
9	19	+	+	+	+	B_1^-, B_1^+	B_1^+	B_1^-	B_1^+
10	20	+	+	−	+	B_2^-, B_2^+	B_2^-	B_2^+	B_2^-

NOTE: Each row shows a unique batch. The four preliminary batches labeled as "P1," "P2," "P3," and "P4" are shown in the upper part. The 20 newly conducted batches are shown in the lower part with the sequential run order. The F_5 & F_6 column shows the settings of $\{B_0, B_1^-, B_1^+, B_2^-, B_2^+\}$ for both trials within each batch. The $F_7(+)$ column shows which of these settings was selected for allocation of the $+$ level for factor F_7, with a similar convention for the $F_8(+)$ and $F_9(+)$ columns.

Step 3. Assign levels for F_4 in the current design and decide the run order of the batches.

Step 4. Assign F_7–F_9 levels to trials.

In Step 1, we imbed the preliminary batches into the whole design. In Step 2, we group the trials with the same combination of F_1–F_3 into blocks of size two under the constraint that the blocking scheme for the preliminary batches are retained. More details on Step 2 can be found in Section 3.2. F_4 is left aside temporarily in Step 1 and Step 2. By definition, the batch with F_4 level "−" must be conducted prior to the batch of the same unit with F_4 level "+." Thus, the level of F_4 has to be considered along with the run order of the batches. In Step 3, we assign values of F_4 such that it is balanced and orthogonal to most factors. In the meantime, we decide the run order of the 20 new batches so that temperature and wind speed are changed as infrequently as possible. More details can be found in Section 3.3. Note that each batch of the experiment uses one unit and consists of two trials. The 24 batches designed in Table 2 make up a total of 48 trials. Instead of using 24 units, 12 units are tested in the icing wind tunnel, with each unit undergoing two batches to evaluate the repeated usage of the coating. As a result, a total of four observations are made from one unit. The 20 new batches of the proposed design are shown in the lower part of Table 2 following the run order of the design.

In Step 4, we assign values of F_7–F_9 such that they are balanced and orthogonal to other factors. Though the effects of F_7–F_9 are not of interest by the engineers, random assignment may cause effect confounding. That is, the estimated effects of F_7–F_9 may be aliased to the effects of F_1 to F_6. The detailed assignment is discussed in Section 3.4. It is notable that we design the experiment with orthogonality as an important property. Later (in Section 5), we will provide theoretical basis for such an arrangement.

3. Details of the Design

In this section, we discuss in detail our considerations for designing the icing wind tunnel experiment.

The factors of interest (F_1–F_6) constitute the fixed effects we intend to evaluate. Although these factors can be controlled within each batch, it is possible that there exist some other factors that are unidentified and thus uncontrolled. These unobservable factors may vary from batch to batch, contributing to the variability of the outcome. This can be appropriately modeled using a mixed model framework and a block-wise random effect below:

$$Y_{ij} = \boldsymbol{x}_{ij}^T \boldsymbol{\beta} + a_i + \epsilon_{ij}. \tag{1}$$

As in usual mixed model specifications, $i = 1, \ldots, n$ represents the batches (blockes), $j = 1, 2$ represents the trials within the same batch, $\boldsymbol{x}_{ij}^T$ represents the p-dimensional row vector in the model matrix for the jth trial in the ith batch, and $\boldsymbol{\beta}$ represents the corresponding coefficient vector for the fixed effects, which may include both main effects and interactions. The block-specific random effects $\{a_i : i = 1, \ldots, n\}$ are independently distributed and have a normal distribution with mean 0 and common variance τ^2. The error terms $\{\epsilon_{ij} : i = 1, \ldots, n, j = 1, 2\}$ are also independently distributed and have a normal distribution with mean 0 and common variance σ^2. In addition, $\{a_i : i = 1, \ldots, n\}$ and $\{\epsilon_{i,j} : i = 1, \ldots, n, j = 1, 2\}$ are mutually independent. Let the trials be ordered such that the response vector is defined as $\boldsymbol{Y} = (Y_{11}, Y_{12}, \ldots, Y_{n1}, Y_{n2})^T$ and the corresponding model matrix is defined as $X = (\boldsymbol{x}_{11}, \boldsymbol{x}_{12}, \ldots, \boldsymbol{x}_{n1}, \boldsymbol{x}_{n2})^T$. Then the best linear unbiased estimation of the coefficients for linear models with both fixed effect(s) and random effect(s) can be obtained using generalized least-square equations (Henderson 1975) when σ and τ are known:

$$\hat{\boldsymbol{\beta}} = (X^T W X)^{-1} X^T W \boldsymbol{Y}, \tag{2}$$

where $W = \{I_n \otimes (\sigma^2 I_2 + \tau^2 J_2)\}^{-1} = I_n \otimes \{\frac{1}{\sigma^2} I_2 - \frac{\tau^2}{\sigma^2(\sigma^2+2\tau^2)} J_2\}$,
I_a is the $a \times a$ identity matrix, and J_2 is the 2×2 matrix with all elements being one. The covariance matrix of $\hat{\boldsymbol{\beta}}$ is

$$\text{cov}(\hat{\boldsymbol{\beta}}) = (X^T W X)^{-1}. \tag{3}$$

In reality, σ and τ are unknown. We obtain the empirical best linear unbiased estimator for $\boldsymbol{\beta}$ in two stages (Das, Jiang, and Rao 2004). First, the estimator of $\boldsymbol{\beta}$ is obtained through unknown σ and τ. Then in the second stage, σ and

τ are replaced with their estimates. Kackar and Harville (1981) showed that the two-stage estimator is unbiased for $\boldsymbol{\beta}$.

It is desirable to construct a design to achieve, for example, D-optimality, that is, to minimize the determinant of the covariance matrix of $\hat{\boldsymbol{\beta}}$. Following the ideas from Cheng (1995), we start with choosing the design points from a good design with the absence of block-wise random effects and then group the trials according to some balance criterion. In the subsequent subsections, we discuss in detail the four steps of our design.

3.1. The Factorial and Imbedded Design

The first task is to deal with the related factors F_5 and F_6. In the following we use F_{56} to refer to the five-level factor whose levels are B_0, B_1^-, B_1^+, B_2^-, and B_2^+. A naive approach is to assign equally many trials for these five combinations. However, this may not be the most efficient methodology for making conclusions from the data. Four major questions to be addressed after the experiments are:

1. How does the coating perform compared to the absence of coating? (B_1 & B_2 vs. B_0)?
2. Which of the two coatings is better (B_2 or B_1)?
3. Would the addition of nanoparticles affect ice accumulation (B_1^+ & B_2^+ vs. B_1^- & B_2^-)?
4. Would the addition of nanoparticles affect ice accumulation differently in the two coatings ($B_1^+ - B_1^-$ vs. $B_2^+ - B_2^-$)?

From the balance viewpoint, it is ideal to assign equal number of trials for both sides of the above comparisons. To address questions 2–4, the number of trials for B_1^-, B_1^+, B_2^-, and B_2^+ shall be the same. However, there is no uniformly best assignment on the number of trials for B_0. Using fewer trials is preferred for questions 2–4. Moreover, question 1 is the most important, and allocating more trials for B_0 will give more informative results for this question. As a compromise, we set the number of trials for B_0, B_1, and B_2 to be equal. Consequently, B_0 occurs twice as often as each of the other levels: B_1^-, B_1^+, B_2^-, and B_2^+. This plan aims to place more emphasis on estimating the effect of contrast 1 (β_5 in the model of Section 4 and Table 7). Special techniques, as shall be discussed in Section 4 when the model is introduced, are needed to analyze effects of F_{56}.

The preliminary batches consist of a factorial design for factors F_1, F_2, and F_{56}. We imbed it into the whole design, which is a factorial in (F_1, F_2, F_3, F_{56}). The cross array after Step 1 is shown in the columns "F_1–F_3" and "Step 1" of Table 3, with the preliminary batches shown in boldface. Under each level combination of (F_1, F_2, F_3) in the outer array, the inner array consists of six trials $\{B_0, B_0, B_1^-, B_1^+, B_2^-, B_2^+\}$, which will be grouped into three batches by Step 2. Note that the grouping of the preliminary trials is fixed, which is shown with the "$B_0B_1^-$" symbol highlighted in boldface in the column "Step 2" of Table 3.

Regular factorial designs have long been recognized as D-optimal designs for two-level factors under models with uncorrelated errors. Although a D-optimal design can be constructed by designing fixed factors and random factors simultaneously, an orthogonal design for $\{F_1, F_2, F_3, F_{56}\}$. We temporarily put aside $\{F_4, F_7\text{–}F_9\}$ and the random effect in Step 1 for several reasons. First, determining a D-optimal design requires knowledge of the intrablock correlation, which we do not have. Second, D-optimal design is usually obtained by solving an optimization problem with iterations, which is hard in our case because there are restrictions on the incomplete blocking scheme for the inner array and the value of F_4 is subject to the assignment of blocks. Third, as will be shown in Section 5, this orthogonal design can ensure uncorrelated parameter estimates for the effects of F_1, F_2, F_3, and F_{56}, regardless of the intrablock correlation.

Table 3. The proposed design after Steps 1 and 2.

			F_{56}	
F_1	F_2	F_3	Step 1	Step 2
−	−	−	$\boldsymbol{B_0}, B_0, \boldsymbol{B_1^-}, B_1^+, B_2^-, B_2^+$	$\boldsymbol{B_0 B_1^-}, B_0B_2^-, B_1^+B_2^+$
+	−	−	$\boldsymbol{B_0}, B_0, \boldsymbol{B_1^-}, B_1^+, B_2^-, B_2^+$	$\boldsymbol{B_0 B_1^-}, B_0B_2^-, B_1^+B_2^+$
−	+	−	$\boldsymbol{B_0}, B_0, \boldsymbol{B_1^-}, B_1^+, B_2^-, B_2^+$	$\boldsymbol{B_0 B_1^-}, B_0B_1^+, B_2^-B_2^+$
+	+	−	$\boldsymbol{B_0}, B_0, \boldsymbol{B_1^-}, B_1^+, B_2^-, B_2^+$	$\boldsymbol{B_0 B_1^-}, B_0B_1^+, B_2^-B_2^+$
−	−	+	$B_0, B_0, B_1^-, B_1^+, B_2^-, B_2^+$	$B_0B_1^+, B_0B_2^+, B_1^-B_2^-$
+	−	+	$B_0, B_0, B_1^-, B_1^+, B_2^-, B_2^+$	$B_0B_1^+, B_0B_2^+, B_1^-B_2^-$
−	+	+	$B_0, B_0, B_1^-, B_1^+, B_2^-, B_2^+$	$B_0B_2^-, B_0B_2^+, B_1^-B_1^+$
+	+	+	$B_0, B_0, B_1^-, B_1^+, B_2^-, B_2^+$	$B_0B_2^-, B_0B_2^+, B_1^-B_1^+$

NOTE: Each of the symbols $\{B_0, B_1^-, B_1^+, B_2^-, B_2^+\}$ represents a single trial. Whenever two symbols are concatenated in the "Step 2" column, the corresponding trials are grouped into one batch. For the preliminary batches, the symbols are bold.

3.2. Grouping Trials Into Batches

The design after Step 1 mandates that the six trials under each combination of F_1, F_2, and F_3 must consist of exactly $\{B_0, B_0, B_1^-, B_1^+, B_2^-, B_2^+\}$ for the combination of the within-block factors F_5 and F_6. The next step is to group the six trials into blocks of size two. For example, the symbol "$B_0B_1^-$" represents a batch in which B_0 pairs with B_1^-. There are in total 15 possible grouping schemes, five of which have the same combination of F_5 and F_6 levels (B_0B_0, $B_1^-B_1^-$, $B_1^+B_1^+$, $B_2^-B_2^-$, $B_2^+B_2^+$) and the other 10 have different combinations of F_5 and F_6 levels ($B_0B_1^-$, $B_0B_1^+$, $B_0B_2^-$, $B_0B_2^+$, $B_1^-B_1^+$, $B_2^-B_2^+$, $B_1^-B_2^-$, $B_1^+B_2^+$, $B_1^-B_2^+$, $B_1^+B_2^-$). Let n_{xy} denote the number of batches in which x and y are paired. Note that n_{xy} must always be an even integer because each unit is tested twice. According to the levels of F_5 and F_6, we can further classify the 15 possible groupings into six categories:

1. $n_{00} = n_{B_0B_0}$, when B_0 is paired with B_0;
2. $n_{SS} = n_{B_1^-B_1^-} + n_{B_1^+B_1^+} + n_{B_2^-B_2^-} + n_{B_2^+B_2^+}$, when the pair $x, y \in \{B_1^-, B_1^+, B_2^-, B_2^+\}$ have the same level of F_5 and the same level of F_6;
3. $n_{0.} = n_{B_0B_1^-} + n_{B_0B_1^+} + n_{B_0B_2^-} + n_{B_0B_2^+}$, when B_0 is paired with $x \in \{B_1^-, B_1^+, B_2^-, B_2^+\}$;
4. $n_{SD} = n_{B_1^-B_1^+} + n_{B_2^-B_2^+}$, when $x, y \in \{B_1^-, B_1^+, B_2^-, B_2^+\}$ have the same level of F_5 but different levels of F_6;
5. $n_{DS} = n_{B_1^-B_2^-} + n_{B_1^+B_2^+}$, when $x, y \in \{B_1^-, B_1^+, B_2^-, B_2^+\}$ have different levels of F_5 but the same level of F_6;
6. $n_{DD} = n_{B_1^-B_2^+} + n_{B_1^+B_2^-}$, when $x, y \in \{B_1^-, B_1^+, B_2^-, B_2^+\}$ have different levels of F_5 and different levels of F_6.

It is apparent that the allocation of the 24 batches into the above six categories affects the covariance matrix of $\hat{\boldsymbol{\beta}}$ and thus is closely related with the optimality of the design. BIBDs have been employed to construct D-optimal block minimum-support designs (Cheng 1995) and have been shown to be robust to the ratio of the random effect variance to the random error variance (Atkins and Cheng 1999). However, these designs consist of only within-block treatment factor(s) but not between-block factor(s). Thus, directly applying BIBDs lacks theoretical support in our problem. We therefore propose a criterion named "grouping balance" to replace the balance condition of BIBD. For our problem, the design is said to be balanced in grouping if the following conditions meet:

1. $n_{B_1^- B_1^-} = n_{B_1^+ B_1^+} = n_{B_2^- B_2^-} = n_{B_2^+ B_2^+} = n_{SS}/4$;
2. $n_{B_0 B_1^-} = n_{B_0 B_1^+} = n_{B_0 B_2^-} = n_{B_0 B_2^+} = n_{0.}/4$.
3. $n_{B_1^- B_1^+} = n_{B_2^- B_2^+} = n_{SD}/2$;
4. $n_{B_1^- B_2^-} = n_{B_1^+ B_2^+} = n_{DS}/2$;
5. $n_{B_1^- B_2^+} = n_{B_1^+ B_2^-} = n_{DD}/2$.

The "grouping balance" conditions for F_5 and F_6 make sure that the inverse of the sub-matrix in $\mathrm{cov}(\hat{\boldsymbol{\beta}})$ representing F_{56} is diagonal. It will be shown that under conditions specified in Section 5, a D-optimal design for the icing wind tunnel experiment can be obtained by setting

$$n_{00} = n_{SS} = n_{DD} = 0, \quad \text{and } n_{SD} = n_{DS} = 4. \tag{4}$$

The grouping balance condition is weaker than the conditions for a BIBD in that not all pairs of levels need to appear in the same blocks for the same number of times. For example, $n_{B_1^- B_1^+}$ is not required to be equal to $n_{B_1^- B_2^+}$. However, as we shall show in Section 5, the grouping balance condition, together with the orthogonality among all factors, guarantee a diagonal covariance matrix for the parameter estimates with the presence of the block-specific random effect. The proposed design after Step 2 is shown in Table 3. The four preliminary batches involving $B_0B_1^-$ are in boldface to emphasize that they have been fixed before grouping.

3.3. The Run Order of the Batches

After Step 2 of the design, all factors of interest except for F_4 have been considered. From the definition of the factor "repetition" (F_4), for any unit, the batch with F_4 level (−) must be carried out before the batch with F_4 level (+). As a result, the assignment of F_4 is closely related to the run order of the batches.

In the design of experiment field, it is common to determine the run order by randomization. However, randomized run order may lead to unfavorable sequences when there are hard-to-change factors (Ju and Lucas 2002). In the icing wind tunnel experiment, the changes of temperature (F_1) and wind speed (F_2) involve adjustments in many system parameters and are therefore hard to control. Thus, we adopt a specific run order that minimizes the number of level changes for temperature and wind speed. The usual one-at-a-time run order (Daniel 1973; Lin and Lam 1997) ensures the most economical arrangement of an experiment. Specifically, for two two-level factors, the run order can be $(++)$, $(+-)$, $(--)$, and $(-+)$, which results in a total of three level changes across the design. This run order scheme is simple but cannot be directly applied to the icing wind tunnel experiment because the constraint from the factor F_4. To reduce confounding, it is desirable that the two batches on the same unit are not conducted under exactly the same combinations of F_1 and F_2. In addition, it is preferred that under the same combination of F_1 and F_2, there is at least one batch of experiment with each level of F_4. The minimum number of level changes under one-at-a-time run order cannot be attained under the above requirements. Therefore, the minimum number of level changes in our study is at least four.

We now propose an approach that ensures the minimum number of level changes for F_1 and F_2. In Section 5, we will show that this method guarantees that F_4 is orthogonal to all other factors, which is important for the theoretical properties of our design. We call our method "alternate shifting." Our run order and the corresponding levels of F_4 is shown in Table 4.

Based on the design in Table 3, for the 20 new experiments, we first group them according to the combinations of F_1 and F_2, say in order $R_1 = (+, +)$, $R_2 = (+, -)$, $R_3 = (-, -)$, and $R_4 = (-, +)$. Since the units in the group R_4 and F_4 level "−" must have their repeated batch with F_4 level "+" under some combination of (F_1, F_2) other than in R_4, one of the R_1, R_2, and R_3 should be repeated at least once. As a result, we add $R_5 = (+, +)$ in which all batches are with F_4 level "+." This makes four times of level changes in total. Not considering the preliminary batches, there are five batches under each combination of F_1 and F_2. For R_1, we arrange three batches, all under $F_4 = -$. Then these three batches (same units as above) are shifted down to R_2 with $F_4 = +$. In addition, two batches (new units) are added under R_2 with $F_4 = -$. In the next step, the two batches under R_2 with $F_4 = -$ are shifted down to R_3 with $F_4 = +$. This procedure goes on until R_5, in which the two batches under R_4 with $F_4 = -$ are shifted down and no new units can be added. Details about the arrangement, together with the unit number, can be found in Table 4.

Table 4. Run order determination and assignment of F_4.

Run order	(F_1, F_2)	F_3	$F_4 : -$	$F_4 : +$
1–3	$R_1 = (+, +)$	−	$B_0B_1^+$ (Unit 1)	
		+	$B_0B_2^-$ (Unit 3), $B_0B_2^+$ (Unit 5)	
4–8	$R_2 = (+, -)$	−	$B_1^+B_2^+$ (Unit 8)	$B_0B_2^-$ (Unit 3)
		+	$B_1^-B_2^-$ (Unit 7)	$B_0B_1^+$ (Unit 1), $B_0B_2^+$ (Unit 5)
9–13	$R_3 = (-, -)$	−	$B_0B_2^-$ (Unit 4)	$B_1^+B_2^+$ (Unit 8)
		+	$B_0B_1^+$ (Unit 2), $B_0B_2^+$ (Unit 6)	$B_1^-B_2^-$ (Unit 7)
14–18	$R_4 = (-, +)$	−	$B_2^-B_2^+$ (Unit 10)	$B_0B_1^+$ (Unit 2)
		+	$B_1^-B_1^+$ (Unit 9)	$B_0B_2^-$ (Unit 4), $B_0B_2^+$ (Unit 6)
19, 20	$R_5 = (+, +)$	−		$B_2^-B_2^+$ (Unit 10)
		+		$B_1^-B_1^+$ (Unit 9)

Table 5. Example for the assignment of factors F_7 to F_9.

F_1–F_3	F_{56}	F_7	F_8	F_9
$(++-)$	$\boldsymbol{B_0}$	**+**	**−**	**+**
$(++-)$	$\boldsymbol{B_1^-}$	**−**	**+**	**−**
$(++-)$	B_0	−	+	−
$(++-)$	B_1^+	+	−	+
$(++-)$	B_2^-	+	−	+
$(++-)$	B_2^+	−	+	−

3.4. The Design for F_7 to F_9

We treat F_7 to F_9 as nuisance factors that may be present in the full model. However, to save the degrees of freedom for estimating the factors of interest, we do not intend to include these factors into the final analysis. Thus, it is desirable to design F_7–F_9 such that our inference for the factors of interest is unaffected by employing a reduced model.

In general, for a response Y, suppose the potential effects are $\boldsymbol{\beta}$ and $\boldsymbol{\gamma}$, in which only $\boldsymbol{\beta}$ is of interest. Assume the underlying true model (unknown to us) to be

$$M_1 : \boldsymbol{Y} = X\boldsymbol{\beta} + Z\boldsymbol{\gamma} + \boldsymbol{\epsilon_1}, \tag{5}$$

but we only fit the reduced model

$$M_2 : \boldsymbol{Y} = X\boldsymbol{\beta} + \boldsymbol{\epsilon_2}, \tag{6}$$

where $\boldsymbol{\epsilon_1}$ and $\boldsymbol{\epsilon_2}$ are the error vector with zero mean and covariance matrix Σ. In the special case of the icing wind tunnel experiment, (6) reduces to (1) if $\Sigma = W^{-1}$. The generalized least-square estimation of $\boldsymbol{\beta}$ using the reduced model is $\hat{\boldsymbol{\beta}} = (X^T\Sigma^{-1}X)^{-1}(X^T\Sigma^{-1}\boldsymbol{Y})$, and its expectation is

$$\begin{aligned} E(\hat{\boldsymbol{\beta}}) &= (X^T\Sigma^{-1}X)^{-1}X^T\Sigma^{-1}(X\boldsymbol{\beta} + Z\boldsymbol{\gamma}) \\ &= \boldsymbol{\beta} + (X^T\Sigma^{-1}X)^{-1}(X^T\Sigma^{-1}Z)\boldsymbol{\gamma}. \end{aligned} \tag{7}$$

When the aliased matrix $X^T\Sigma^{-1}Z = 0$, $\hat{\boldsymbol{\beta}}$ is unbiased (Box and Draper 1987). If the error terms are homoscedastic and uncorrelated, the orthogonality between the columns of X and the columns of Z leads to a zero alias matrix. In our case, the error terms are correlated. As will be shown in Section 5, the orthogonality of F_7–F_9 with the other factors still guarantees the unbiasedness of $\hat{\boldsymbol{\beta}}$. So our goal is to make F_7–F_9 orthogonal to F_1–F_6. Note that within the same batch, the two trials have the same level of F_1–F_4. By the definition of F_7–F_9, the two trials within the same batch have exactly opposite levels of F_7–F_9. Immediately, we have that F_7–F_9 are orthogonal to F_1–F_4. So we only need to assign F_7–F_9 according to F_{56}.

We proceed by looking at each combination of F_1–F_3 separately. Take the six trials with F_1 to F_3 being $(+, +, -)$ as an example. For the two trials done in the preliminary batches, levels of F_7 to F_9 have been assigned, as shown in boldface in Table 5. Because F_i $(i = 7, 8, 9)$ should be balanced to B_0, the third row of Table 5 is $(-, +, -)$. Then we only need to consider the assignments for $F_5 = B_1$ or B_2. For these four rows, since the F_5 and F_6 columns are orthogonal, assigning values for F_7 to F_9 is equivalent to finding a third column orthogonal to them. The only two choices for the third column are $(-, +, +, -)^T$ and $(+, -, -, +)^T$. As the second row is assigned in the preliminary batches, the choice for the third orthogonal column is now determined. Other combinations of F_1–F_3 can be dealt with similarly.

4. Data Analysis

In this section, we present the analysis of the data generated from our proposed design for the icing wind tunnel experiment. The original data is available from the authors by request.

We use a mixed effects model with block-specific random effects as in (1). The response Y is the weight of ice accumulated in each trial. The model matrix is made up of nine columns: $X = (\boldsymbol{X}_0, \ldots, \boldsymbol{X}_7, \boldsymbol{X}_{67})$. Variable $\boldsymbol{X}_0$ corresponds to the intercept parameter β_0. Variable $\boldsymbol{X}_1$ corresponds to the main effect (β_1) of the average temperature recorded during the experiment. Note that in the real analysis, actual average temperature instead of the designed levels of -1 and 1 are used to make better use of the data. Variables $\boldsymbol{X}_2 - \boldsymbol{X}_4$ correspond to the main effects (β_2–β_4) of factors F_2–F_4 (a value of -1 for low level $-$, and a value of 1 for high level $+$). Further, we use four variables $\boldsymbol{X}_5$–$\boldsymbol{X}_7$ and $\boldsymbol{X}_{67}$ to represent the effects of F_{56} in the model matrix, with the corresponding parameters β_5–β_7 and β_{67}. The detailed coding is provided in Table 6. Variable $\boldsymbol{X}_5$ corresponds to the contrast between the deicing coating (B_1 or B_2) and aluminum (B_0). Variable $\boldsymbol{X}_6$ corresponds to the contrast between B_2 and B_1. Variable $\boldsymbol{X}_7$ corresponds to the contrast between adding nanoparticles and the absence of additional nanoparticles when a deicing coating (B_1 or B_2) is used. Variable $\boldsymbol{X}_{67}$ corresponds to the interaction of $\boldsymbol{X}_6$ and $\boldsymbol{X}_7$, that is, whether addition of nanoparticles has differential impact on B_1 and B_2. Variables $\boldsymbol{X}_5$–$\boldsymbol{X}_7$ and $\boldsymbol{X}_{67}$ represent the comparison between different icing surfaces (e.g., aluminum vs. coating or one coating vs. another coating) and their corresponding effects provide answers to the questions raised in Section 3.1. Therefore, their effects are of the greatest importance in our study. The between-block variables $\boldsymbol{X}_1$–$\boldsymbol{X}_4$ are our secondary goal. So variables $\boldsymbol{X}_1$–$\boldsymbol{X}_7$ and $\boldsymbol{X}_{67}$ all enter our model. In case of insignificance of these factors, it is of interest to learn about the effect size of each potential factor to guide further investigation of the coating. We apply a log transformation to the response variable to stabilize variance and to improve normality. Figure A.1 in Appendix A confirms the validity of the transformation. We have also added two-way interactions among $\boldsymbol{X}_1$–$\boldsymbol{X}_7$ but none of these interactions appear significant at the 0.05 level. Results of fitting the mixed model are shown in Table 7.

The most important conclusion we reach from the icing wind tunnel experiment is that the deicing coating can effectively reduce ice accumulation as compared with aluminum (p-value of β_5 <0.05). Furthermore, there is confidence that both compositions of the coating, B_1 and B_2, reduce ice accumulation. Although B_2 possesses a slightly better parameter estimate than B_1, the difference is not statistically significant. The addition of nanoparticles does not tend to adversely affect ice accumulation on the coating. This finding shows that with lifetime lengthened and mechanical properties enhanced, the coating with nanoparticles may have almost the same icephobic properties.

Table 6. The coding for the model matrix of factors F_5 and F_6.

F_{56}	X_5	X_6	X_7	X_{67}
B_0	−2	0	0	0
B_1^-	1	−1	−1	1
B_1^+	1	−1	1	−1
B_2^-	1	1	−1	−1
B_2^+	1	1	1	1

Table 7. Results on the mixed effect model for the icing wind tunnel experiment.

Variable	Effect	Estimate	Standard Error	Test statistic	p-Value
Intercept (X_0)	β_0	1.9095	0.1272	15.02	<0.0001
Temperature (X_1)	β_1	− 0.1265	0.0142	− 8.92	<0.0001
Speed (X_2)	β_2	0.2161	0.0329	6.57	<0.0001
Vibration (X_3)	β_3	− 0.0319	0.0328	− 0.97	0.3441
Repetition (X_4)	β_4	− 0.0623	0.0334	− 1.86	0.0772
Coating versus aluminum (X_5)	β_5	− 0.0859	0.0181	− 4.74	0.0001
B_2 versus B_1 (X_6)	β_6	− 0.0494	0.0337	− 1.47	0.1577
Nanoparticle addition (X_7)	β_7	− 0.0501	0.0337	− 1.49	0.1527
X_{67}	β_{67}	0.001825	0.0314	0.06	0.9542
Random effect variance	τ^2	0.0120	0.0106	1.13	0.1298
Residual error variance	σ^2	0.0279	0.0090	3.08	0.001

Atmospheric conditions appear to affect ice accumulation. A lower temperature or a higher wind speed significantly increases ice accumulation. However, vibration does not show significant impact. Finally, results support that the performance of the coating is not degraded upon second usage.

Conventional deicing coatings such as electro-thermal coatings, opto-thermal coatings, super-hydrophobic coatings, and coatings containing slowly released freezing point depressants or lubricating oils, suffer from problems such as low icephobicity, flashover in the power system, and severe degradation upon repeated usage. The novel deicing coating we investigate is promising in effectively reducing ice accumulation. The coating is additionally less subject to problems associated with aging due to the slow release mechanism induced by the addition of nanoparticles. As a result, this deicing coating may offer a more convenient, efficient, and durable alternative to existing deicing methods.

5. Theoretical Results

5.1. Theoretical Conclusions of the Experiment

In this section, we provide some theoretical considerations for our design. The propositions and the theorem are specific to our study. The three lemmas, however, are very general.

Here, we use $X = (\boldsymbol{X}_0, \dots, \boldsymbol{X}_8)$ to represent the model matrix of a design, with $\boldsymbol{X}_0$ being the intercept vector, $\boldsymbol{X}_1$–$\boldsymbol{X}_4$ being 1 (high level +) or −1 (low level −) and the coding for $\boldsymbol{X}_5$–$\boldsymbol{X}_7$ defined as in Table 6. For ease of presentation, we use $\boldsymbol{X}_8$ here to represent the interaction effect $\boldsymbol{X}_{67}$ as defined in Table 6. As discussed in Section 4, the actual temperature is used as the covariate representing F_1 in the data analysis. However, at the design stage, since only temperature levels (high or low) can be planned, only 1 or −1 values are allowed in $\boldsymbol{X}_1$. Let β_0 to β_8 denote the corresponding parameters associated with $\boldsymbol{X}_0$ to $\boldsymbol{X}_8$ and $\boldsymbol{\beta} = (\beta_0, \dots, \beta_8)^T$. Let $\boldsymbol{Y}$ denote the response vector, $\boldsymbol{a}$ denote the vector containing all block-specific random effects, and $\boldsymbol{\epsilon}$ denote the vector containing random errors. The model for analyzing the data takes the mixed model form:

$$Y = X\boldsymbol{\beta} + \boldsymbol{a} + \boldsymbol{\epsilon}.$$

As in Section 3, the covariance matrix of Y is W^{-1} if two trials within the same batch are one after another in $\boldsymbol{Y}$. Then our primary goal is to design the experiment to minimize the determinant of the covariance matrix of $\hat{\boldsymbol{\beta}}$ (specified in (3)), that is, to achieve D-optimality.

Before proceeding to the main conclusion about D-optimality and D-efficiency of our design, we first present a useful lemma and a few propositions. Each one addresses the theoretical properties for one step of our design.

Lemma 1. Let $\boldsymbol{x}$ and $\boldsymbol{z}$ represent two column vectors and W^{-1} represent a covariance matrix. Then $\boldsymbol{x}^T W \boldsymbol{z} = 0$, if the following two properties hold:

P_1. $\boldsymbol{z}$ is orthogonal to $\boldsymbol{x}$;

P_2. $\boldsymbol{z}$ is an eigenvector of W.

Lemma 1 shows that a diagonal covariance matrix for parameter estimates of a mixed model may be obtained from a mutually orthogonal model matrix X if property P_2 is satisfied for the columns of X. Let W denote the inverse of the covariance matrix of the outcome vector as in (2) with $n = 24$ batches and let $U = (U_{i,j})$ denote $X^T W X$, the matrix whose determinant we would like to maximize to achieve D-optimality of the design. By Lemma 1 we have the following Proposition.

Proposition 1. For any $i = 0, \dots, 4$, if $\boldsymbol{X}_i$ is orthogonal to all other columns in the model matrix X, then

$$U_{i,j} = 0,$$

for $i = 0, \dots, 4$, $j = 0, \dots, 8$ and $j \neq i$.

All proofs are provided in Appendix B (available online). A key condition to prove Proposition 1 is the validity of Property P_2. In the icing wind tunnel experiment, the values of $\boldsymbol{X}_0$–$\boldsymbol{X}_4$ are the same within the same batch. This special structure results in Property P_2 for $\boldsymbol{X}_0$–$\boldsymbol{X}_4$. Proposition 1 shows that orthogonality is a sufficient condition to ensure that the off-diagonal entries related with $\boldsymbol{X}_0$ to $\boldsymbol{X}_4$ are all zero in U. So in Step 1 of our design, we employ a cross array for F_1–F_3 and F_{56}. Proposition 1 does not deal with $U_{i,j}$ for $i, j = 5, \dots, 8$. We deal with this part in Step 2 of our design, with the properties given in the following proposition.

Proposition 2. If $\boldsymbol{X}_5$ to $\boldsymbol{X}_8$ satisfy the "grouping balance" conditions in Section 3.2, then $U_{i,j} = 0$ for $i, j = 5, \dots, 8$ and $i \neq j$.

The "grouping balance" conditions make sure that the corresponding part of U is diagonal. After Step 2, the only factor that needs to be determined is F_4. If F_4 is orthogonal to other factors, the whole matrix U is diagonal according to Propositions 1 and 2. In Section 3.3, we have explained that the minimum possible number of level changes is four. We assign levels for F_4 and arrange the run order by Table 4 following the "alternate shifting" run order method, which, according to Proposition 4 in Section 5.2, ensures that F_4 is orthogonal to F_1–F_3.

The last step in our design process relies on the following proposition, which shows that correct inference about F_1–F_6 can be made under the reduced model (6) even if F_7–F_9 affects the final outcome and should be included in the full model.

Proposition 3. If F_7 to F_9 are orthogonal to the columns in the model matrix X, then $\hat{\boldsymbol{\beta}} = (X^T\Sigma^{-1}X)^{-1}X^T\Sigma^{-1}\mathbf{Y}$ is unbiased under the full model M_1 in (5).

Next, we proceed to the main conclusion about the D-optimality of the design. It is mainly based on the following lemmas (Graybill 2001).

Lemma 2. The determinant of an $m \times m$ positive definite matrix $A = (a_{ij})$ satisfies

$$\det(A) \leq \prod_{i=1}^{m} a_{ii},$$

with equality if and only if A is a diagonal matrix.

Lemma 3. The determinant of an $m \times m$ positive definite matrix $A = (a_{ij})$ satisfies

$$\det(A) \leq \left(\prod_{i=1}^{m-k} a_{ii}\right) \det(A_2),$$

where A_2 is the lower right $k \times k$ submatrix of A, for $k \leq m$, with equality if and only if $a_{ij} = 0$ for $i = 1, \ldots, m-k$, $j = 1, \ldots, m$, $j \neq i$.

Lemma 2 shows that among positive definite matrices with the same diagonal elements, the diagonal matrix has the largest determinant. Lemma 3 assumes that there are two positive definite matrices that have the first $m-k$ diagonal elements identical. When this holds true, the determinants of the lower right parts are crucial to the comparison of determinants of the two matrices. The search for the D-optimal design thus follows two major steps: (1) comparing the values of the product of the diagonal elements and (2) looking for the diagonal matrix, which can achieve the above product as the determinant. The proofs of Lemmas 2 and 3 take advantage of simple matrix algebra (Graybill 2001) and are omitted here.

Now, we present our main result about the D-optimality of a design following the four steps (from Section 3.1 to Section 3.4). Designs under our consideration satisfy the following set of conditions.

C_1. The total number of batches is $n = 24$ with each batch consisting of two trials;

C_2. The total numbers of trials involving B_0, B_1, and B_2 are equal;

C_3. The total numbers of trials involving $F_6 = -$ and $F_6 = +$ are equal;

C_4. The number of $n_{B_0B_1^-}$ is no less than four.

Condition C_1 constrains the number of trials under our budget. Conditions C_2 and C_3 require that the levels of F_5 and F_6 have equal numbers of observations. Condition C_4 is due to the four preliminary batches with $B_0B_1^-$.

Theorem 1. Given that each unit undergoes the initial experiment and the repeated experiment, and under Conditions C_1–C_4, an orthogonal array with "grouping balance" that satisfies $n_{00} = n_{SS} = n_{DD} = 0$ and $n_{SD} = n_{DS} = 4$ is a D-optimal design for the effects of β_0–β_8.

Remark 1. Cheng (1995) proposed the D-optimal block design within the group of minimum-support designs. The optimal design depends on the value of $\rho = \tau^2/\sigma^2$. The D-optimality of the design as defined in Theorem 1 does not depend on ρ and thus can be applied with no prior knowledge of ρ.

The proposed design is constructed following the above considerations for D-optimality and it satisfies all requirements in Theorem 1 except that factors F_1 and F_4 are not orthogonal to each other. The "alternate shifting" method guarantees that F_4 is orthogonal to F_1–F_3 for the 20 new batches. However, as can be seen from Table 2, F_1 is fully aliased with F_4 in the four preliminary batches. While there is a way to construct a design to achieve the orthogonality between F_1 and F_4, the number of level changes must be increased. So there is a balance between the orthogonality and the number of level changes for the design.

Define the D-efficiency of a design D to be

$$D_{\text{efficiency}}(D) = \left\{\frac{\det(D)}{\det(D^*)}\right\}^{1/9},$$

where D^* is the D-optimal design specified in Theorem 1. By direct calculation, the D-efficiency of the proposed design is as high as 0.997 and is independent of the value of ρ. Therefore, our final design sacrifices a small amount of efficiency to achieve the minimum number of level changes for F_1 and F_2.

5.2. General Applications

In this section, we provide theoretical results for general application. Suppose the experiment has m two-level environment factors denoted by $F_1, F_2, \ldots, F_m$ and one treatment factor with t levels, $\xi_1, \xi_2, \ldots, \xi_t$. We use a cross array to arrange the two types of factors, of which the outer array is denoted by D_1 with run size of N_1, and the inner array is denoted by D_2 in which the treatment level ξ_i appears t_i times, for $i = 1, \ldots, t$ ($\sum_{i=1}^t t_i = 2q$). Under the cross array structure, for each row of D_1, there are $2q$ trials that will be grouped into q blocks of size two.

Assume the underlying model is

$$Y = \beta_0 + \sum_{i=1}^{m} \beta_i X_i + \sum_{j=1}^{t-1} \alpha_j C_j + Z\gamma + \epsilon, \tag{8}$$

where β_i is the main effect of F_i, α_j is the jth treatment contrast effect, $\gamma = (\gamma_1, \gamma_2, \ldots, \gamma_{N_1q})'$ with γ_k being the random effect of the kth block, Z is the matrix of $\mathbf{I}_{\mathbf{N_1q}} \otimes [1, 1]'$, and ϵ is the random error. Further assume that $\gamma \sim N(0, \tau^2\mathbf{I}_{\mathbf{N_1q}})$, $\epsilon \sim N(0, \sigma^2\mathbf{I}_{\mathbf{2N_1q}})$ and γ and ϵ are independent to each other.

Let $P = (X_0, X_1, \ldots, X_m)$, $Q = (C_1, C_2, \ldots, C_{t-1})$, and $X = (P, Q)$. The information matrix is $X'WX$, where $W = \mathbf{I}_{\mathbf{N_1q}} \otimes (a\mathbf{I_2} + c\mathbf{J_2})$ with $a = 1/\sigma^2$ and $c = -\tau^2/(\sigma^2(\sigma^2 + 2\tau^2))$. Then we have

$$X'WX = \begin{pmatrix} P'WP & P'WQ \\ Q'WP & Q'WQ \end{pmatrix}.$$

Based upon the cross array structure, we have $P = D_1 \otimes \mathbf{1_{2q}}$, and $Q = [Q'_1, Q'_2, \dots, Q'_{N_1}]'$, where Q_1 is a $2q \times (t-1)$ matrix, and Q_i can be obtained from Q_1 by row permutations, for $i = 2, \dots, N_1$. Suppose D_1 is an orthogonal design of two levels ± 1, and $Q'_1 \cdot \mathbf{1}_{2q} = \mathbf{0}_{t-1}$, then

(i) $P'WP = 2N_1 q(a+2c)\mathbf{I_{m+1}}$; and
(ii) $P'WQ = \mathbf{0}$.

The information matrix can thus be simplified to

$$X'WX = \begin{pmatrix} d \cdot \mathbf{I_{m+1}} & \mathbf{0} \\ \mathbf{0} & Q'WQ \end{pmatrix},$$

where $d = 2N_1 q(a+2c)$. Thus, the D-optimality of the design is determined by $\|Q'WQ\|$. It can be seen that the matrix $Q'WQ$ dependents on how the $2q$ trials under each row of D_1 are paired into blocks.

Remark. In the design of the icing wind tunnel experiment, we have $m = 3$, $t = 5$, $N_1 = 8$, $t_1 = 2$, $t_2 = \cdots = t_5 = 1$, and $q = 3$. Under the cross array structure, the outer array D_1 is a factorial design $\mathbf{2^3}$ and the inner array for F_{56} is blocked by the "grouping balance" incomplete blocking scheme. The $X'WX$ is a diagonal matrix with the first four diagonal elements being $48(a+2c)$, and the last four diagonal elements being $48(2a+c)$, $32(a+c)$, $32(a+c)$, and $16(2a+c)$, respectively.

The general set for minimum number of level changes is discussed next. Suppose there are m factors, $F_1, \dots, F_m$, each of two levels, where the first s factors are hard-to-change, and the last $m-s$ factors are easy-to-change. Each unit is experimented under two different level combinations of $F_1, \dots, F_m$, the initial batch and the repeated batch. Let F_{m+1} be the repetition factor, where $F_{m+1} = -1$ and $+1$ for the initial and repeated batch, respectively. Consider the designs that satisfy the following three conditions:

C_5. Each unit undergoes two batches, where the initial batch must be carried out before the repeated batch.
C_6. Under each of the 2^s combinations of F_1–F_s, there are at least one initial batch and at least one repeated batch.
C_7. The initial and repeated batches of the same unit are conducted under different combinations of F_1 to F_s.

Condition C_5 is natural for testing the effect of repetition factor. Conditions C_6 and C_7 are proposed to avoid confounding between the effects of F_1–F_s and F_{m+1}, or confounding between the effects of F_1–F_s and any potential random effects related to units. It can be seen that the minimum number of level changes under constraints of C_5–C_7 cannot be $2^s - 1$ as given by the ordinary minimum level change method. In the following, we propose the "alternate shifting" run order method.

Let $n_1 = 2^s$ and $n_2 = 2^{m-s}$. Let $R_1, R_2, \dots, R_{n_1}$ be the n_1 level combinations of factors $F_1, \dots, F_s$, such that the sequence $R_1, R_2, \dots, R_{n_1}$ is in the one-at-a-time run order, and $r_1, r_2, \dots, r_{n_2}$ be the n_2 level combinations of factors $F_{s+1}, \dots, F_m$. Let g^k_{i1} and g^k_{i2} denote the units conducted under $(F_1, \dots, F_s) = R_i$ and $(F_{s+1}, \dots, F_m) = r_k$ with the initial experiment and the repeated experiment, respectively. Table 8 illustrates the run order scheme. Let $G_{ij} = \{g^1_{ij}, \dots, g^{n_2}_{ij}\}$; set $G_{12} = G_{n_1+1,1} = \emptyset$ and $G_{i,2} = G_{i-1,1}$. The run order of this scheme is $G_{11} \to G_{21} \to G_{22} \to \cdots \to G_{n_1,1} \to G_{n_1,2} \to G_{n_1+1,2}$, with the number of level changes for the hard-to-change factors being 2^s. Proposition 4 shows its theoretical properties.

Table 8. Run order scheme.

Factors F_1 to F_m		Repeated factor F_{m+1}	
Level combination of F_1 to F_s	Level combination of F_{s+1} to F_m	Units under $F_{m+1} = -$	Units under $F_{m+1} = +$
R_1	r_1	g^1_{11}	$\emptyset$
$\vdots$	$\vdots$	$\vdots$	$\vdots$
R_1	r_{n_2}	$g^{n_2}_{11}$	$\emptyset$
R_2	r_1	g^1_{21}	g^1_{22}
$\vdots$	$\vdots$	$\vdots$	$\vdots$
R_2	r_{n_2}	$g^{n_2}_{21}$	$g^{n_2}_{22}$
$\vdots$	$\vdots$	$\vdots$	$\vdots$
R_i	r_1	g^1_{i1}	g^1_{i2}
$\vdots$	$\vdots$	$\vdots$	$\vdots$
R_i	r_{n_2}	$g^{n_2}_{i1}$	$g^{n_2}_{i2}$
$\vdots$	$\vdots$	$\vdots$	$\vdots$
R_{n_1+1}	r_1	$\emptyset$	$g^1_{n_1+1,2}$
$\vdots$	$\vdots$	$\vdots$	$\vdots$
R_{n_1+1}	r_{n_2}	$\emptyset$	$g^{n_2}_{n_1+1,2}$

NOTE: Each row represents an experimental run. The first and second columns list level combinations of hard-to-change factors (F_1–F_s) and easy-to-change factors (F_{s+1}–F_m), respectively. The third and fourth columns list the labels of units undergoing initial and repeated experiments, respectively.

Proposition 4. Under C_5–C_7, the "alternate shifting" method attains the minimum number of level changes and also ensures that F_{m+1} is orthogonal to F_1–F_m.

Remark. In the icing wind tunnel experiment, we have three environment factors F_1, F_2, and F_3, where F_1 and F_2 are two hard-to-change factors. In this case $n_1 = 4$ and $n_2 = 2$, and the run order is

$$G_{11} \to G_{21} \to G_{22} \to G_{31} \to G_{32} \to G_{41} \to G_{42} \to G_{52},$$

in which the factors F_1 and F_2 change four times. Table 4 displays this order.

6. Conclusions and Discussion

In this article, we present our design and analysis for the icing wind tunnel experiment for a newly developed deicing coating. Results confirm that the deicing coating is a promising strategy for solving the aircraft icing problem in that it will significantly reduce ice accumulation as compared with aluminum. Addition of nanoparticles or one repeated usage of the coating do not significantly impact the deicing property of the coating, which is a good sign for the durability of such coatings. As expected, atmospheric conditions such as wind speed and temperature significantly affect the amount of ice accumulated. As compared with traditional deicing coatings, the novel coating offers both high icephobicity and prolonged service lifetime. Thus, this new deicing coating offers a more efficient and durable alternative to existing deicing methods.

The icing wind tunnel experiment raises multiple challenges on different aspects of design of experiment (DOE) theory and practice. Conventional DOE methods can address one challenge but fail to account for the others in such a complex experiment. We approach the problem by decomposing the complex problem into several steps, with each step addressing a particular challenge. In addition, these steps are properly arranged so that the overall design possesses nice properties.

The icing wind tunnel experiment involves multiple between-block factors, multiple within-block factors, and random block effects. Each block contains two trials and the variance of the block effect is unknown prior to the experiments. We propose a cross array structure with orthogonal design as the outer array and the "grouping balance" incomplete blocking scheme as the inner array for designing such an experiment. Generally, an orthogonal design can be chosen as the outer array to produce some uncorrelated estimations, and the D-efficiency of the whole design will depend on the incomplete blocking schemes. In the meantime, we reach several conclusions. First, orthogonality is still a desirable property when dealing with a mixed-effect model. For experiments with fixed effects only, orthogonality guarantees uncorrelated estimates on main effects. This is not true, however, for general experiments with mixed effects. In our experiment, F_1–F_4 take the same value in each block; F_7–F_9 take opposite values in each block. For such fixed factors, orthogonality is still a sufficient condition for uncorrelated parameter estimates. For F_5 and F_6, which can take either the same or different values within a block, we propose additional "grouping balance" conditions. We show that orthogonality in addition to grouping balance guarantees uncorrelated parameter estimates. It will be interesting to study the sufficiency and necessity of orthogonality and grouping balance for other cases.

Not only does our design strategy give uncorrelated parameter estimates, but it also leads to D-optimal designs. For complex situations with random factors, it may be hard to search for D-optimal designs directly. Our example suggests a multi-step designing scheme: work on the most important effects first and worry about secondary effects second. In both steps, balance, orthogonality, and grouping balance are considered to be the principles. In such a scheme, the global optimality may not be guaranteed but high D-efficiency is to be expected.

In addition, for nuisance factors F_7 to F_9 that have opposite levels within the same block, we show that orthogonality for these factors to factors of interest (F_1 to F_6) is important in ensuring that the parameter estimates for the factors of interest are unbiased. This holds regardless of whether or not F_7 to F_9 affect the response variable. To focus on important factors, we made F_7 to F_9 orthogonal to $\boldsymbol{X}_5$–$\boldsymbol{X}_7$. However, not all of them are orthogonal to $\boldsymbol{X}_{67}$. Finally, we also discuss how to arrange different batches so that the number of level changes for temperature and wind speed is as small as possible. We propose the "alternate shifting" method, which shifts between F_4 being low level ($-$) and F_4 being high level ($+$). We also show that such a method guarantees that F_4 is orthogonal to other factors. The final design applies the "alternate shifting" method to the 20 new experiments, which results in a low correlation between F_1 and F_4 (correlation coefficient of 1/6). Nevertheless, the efficiency of the design is very high and parameter estimation is not affected much. Had the four preliminary batches been conducted under conditions that satisfy the "alternate shifting" method, the design would have guaranteed D-optimality and the minimum number of level changes simultaneously.

Although most theoretical results in this article (Propositions 1, 2, 3 and Theorem 1) are presented under the context of the current design, some general principles provide guidance for designs of this type of problems. The search for a D-optimal design under a nondiagonal covariance structure may proceed in a step-by-step fashion. First, one can start from a design with fixed-effect factors only, taking into consideration of good properties of standard design of experiments. For example, when considering a block design with random block effects in which both between-block factors and within-block factors are present, we can adopt a cross array structure for the whole design. The between-block factors form the outer array, and the within-block factors form the inner array. Then, experiments should be grouped into blocks according to some balance criteria, for example, criteria for a BIBD or the concept of "grouping balance" proposed in this article. In addition, for incomplete block designs, it is sensible not to put the same level of within-block factors within one block (e.g., not to pair B_0 with B_0). This usually results in higher efficiency, as the different treatment levels are compared directly. After that, one proceeds to take care of other considerations if any, for example, the presence of hard-to-change factors or nuisance parameters. In the particular case of the icing wind tunnel experiment, the orthogonality of the nuisance factors to the factors of interest guarantees a zero aliased matrix, ensuring the unbiasedness of parameter estimates under the reduced model. Under a general covariance structure, the same conclusion applies when the aliased matrix can be made zero. More generalizations to the current design can be of interest and will be investigated in the future, including the "grouping balance" conditions for general type of factors, D-optimal block design with block size of more than two, and the case with multiple random effects.

In this article, we considered batch-specific random effect as the major source of variability, rather than random noise in the icing wind tunnel experiment. It is arguable whether unit-specific random effect (repeated usage of the same unit) and random effect arising from whole plot of a split-plot like design (due to nonrandom run order) are also possible sources of variation. We consider these as less important in the icing wind tunnel experiment for two reasons. First, there is much more variability associated with the environment within the icing wind tunnel than the environment in the laboratory to produce the units. Second, the actual value of F_1, the most hard-to-change factor can be recorded during the experiment and was used for the final data analysis. This largely reduces the temporal impact of F_1 from the nonrandom run order we have used. So at the design stage, we only assumed the existence of the batch-specific random effect and worked out a nice solution for an efficient design. At the analysis stage, the incorporation of these additional random effects was also considered but showed minimal impact on the analysis, which confirms our assumption at the design stage.

Modern experiments typically involve many complicated setups. The proposed design method provides a step-by-step procedure for solving such a complex problem. Experimental scientists are becoming aware that conventional designs may not be appropriate for experiments with high complexities. This is indeed the motivation of our work. As a result, the proposed

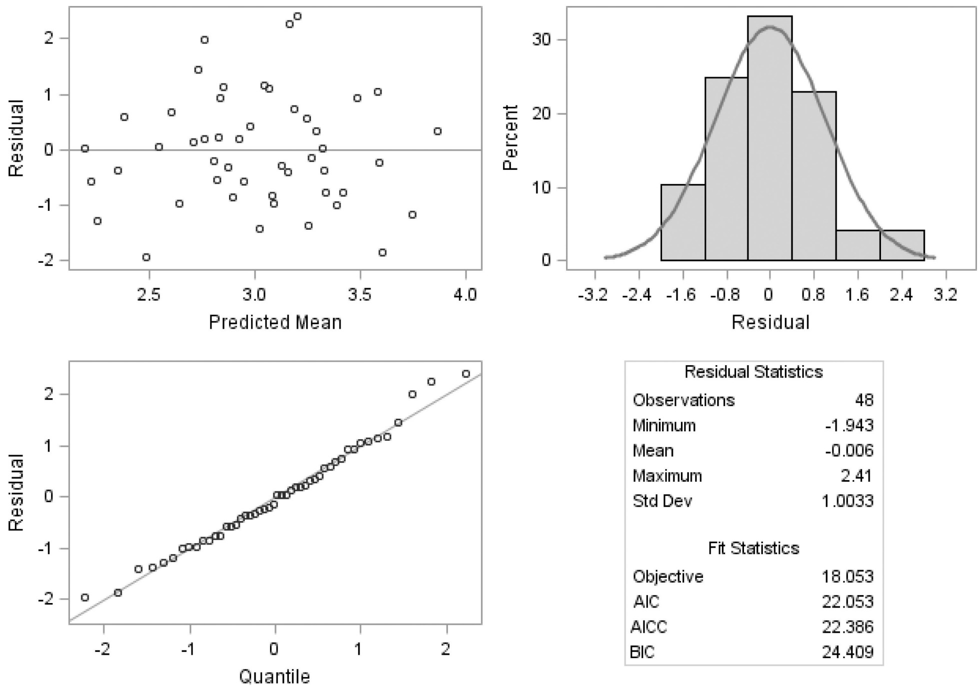

Figure A.1. The residual plot, the histogram of the residuals, and the Q-Q plot of the residuals are used to assess the goodness of fit of our model.

method and strategy is anticipated to find many applications in modern experimental sciences. Due to confidentiality concerns, some other potential applications are not discussed here.

Appendix A: Diagnosis

The residual plot, the histogram of the residuals and the Q-Q plot of the residuals are used to assess the goodness of fit of our model. The plots indicate a good fit after the log transformation for the response variable.

Supplementary Material

The online supplementary material contains Appendix B.

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References

Atkins, J. E., and Cheng, C. S. (1999), "Optimal Regression Designs in the Presence of Random Block Effects," *Journal of Statistical Planning and Inference*, 77, 321–335. [1418,1422]

Ayres, J., Simendinger, W., and Balik, C. (2007), "Characterization of Titanium Alkoxide Sol-Gel Systems Designed for Anti-Icing Coatings," *Journal of Coatings Technology and Research*, 4, 463–471. [1417]

Bose, R. C. (1939), "On the Construction of Balanced Incomplete Block Designs," *Annals of Eugenics*, 9, 353–399. [1418]

Bose, R. C., and Nair, K. (1939), "Partially Balanced Incomplete Block Designs," *Sankhyā: The Indian Journal of Statistics*, 4, 337–372. [1418]

Box, G., and Draper, N. R. (1987), *Empirical Model Building and Response Surface*, New York: Wiley. [1423]

Cheng, C. S. (1995), "Optimal Regression Designs Under Random Block-Effect Models," *Statistica Sinica*, 5, 485–497. [1418,1421,1425]

Cochran, W. G., and Cox, G. (1957), *Experimental Designs*, New York: Wiley. [1418]

Daniel, C. (1973), "One-At-A-Time Plans," *Journal of the American Statistical Association*, 68, 353–360. [1422]

Das, K., Jiang, J., and Rao, J. (2004), "Mean Squared Error of Empirical Predictor," *The Annals of Statistics*, 32, 818–840. [1420]

Goos, P. (2006), "Optimal Versus Orthogonal and Equivalent-Estimation Design of Blocked and split-Plot Experiments," *Statistica Neerlandica*, 60, 361–378. [1418]

Goos, P., and Vandebroek, M. (2003), "D-Optimal Split-Plot Designs With Given Numbers and Sizes of Whole Plots," *Technometrics*, 45, 235–245. [1418]

Graybill, F. (2001), *Matrices With Applications in Statistics*, London: Duxbury Press. [1425]

Guan, L., Zhang, H., Yang, Y., Zhang, Z., and Wang, C. (2010), "Deicing Silicone Coating Based on Bidirectional Migration Effect of Alkane Molecules," Technical Report, National Center for Nanoscience and Technology, China. [1417]

Henderson, C. R. (1975), "Best Linear Unbiased Estimation and Prediction Under a Selection Model," *Biometrics*, 31, 423–447. [1420]

Huneault, M., Langheit, J., and Caron, J. (2005), "Combined Models for Glaze Ice, Accretion and De-Icing of Current-Carrying Electrical Conductors," *IEEE Transactions on Power Delivery*, 20, 1611–1616. [1417]

Johns, B., and Goos, P. (2009), "D-Optimal Designs of Split-Split-Plot Experiments," *Biometrika*, 96, 67–82. [1418]

——— (2012), "An Algorithm for Finding D-Efficient Equivalent-Estimation Second-Order Split-Plot Designs," *Journal of Quality Technology*, 44, 363–374. [1418]

Ju, H., and Lucas, J. (2002), "Lk Factorial Experiments With Hard-to-Change and Easy-to-Change Factors," *Journal of Quality Technology*, 34, 411–421. [1418,1422]

Kackar, R., and Harville, D. (1981), "Unbiasedness of Two-Stage Estimation and Prediction Procedures for Mixed Linear Models," *Communications in Statistics—Theory and Methods*, 10, 1249–1261. [1421]

Kulinich, S., and Farzaneh, M. (2009), "Ice Adhesion on Super-Hydrophobic Surfaces," *Applied Surface Science*, 255, 8153–8157. [1417]

Liao, W., Jia, Z., Guan, Z., Wang, L., Yang, J., Fan, J., Su, Z., and Zhou, J. (2007), "Reducing Ice Accumulation on Insulators by Applying Semi-conducting RTV Silicone Coating," *IEEE Transactions on Dielectrics and Electrical Insulation*, 14, 1446–1454. [1417]

Lin, D. K. J., and Lam, A. W. (1997), "Connections Between Two-Level Factorials and Venn Diagrams," *The American Statistician*, 51, 49–51. [1422]

Macharia, H., and Goos, P. (2010), "D-Optimal and D-Efficient Equivalent-Estimation Second-Order Split-Plot Designs," *Journal of Quality Technology*, 42, 358–372. [1418]

Menini, R., and Farzaneh, M. (2009), "Elaboration of Al_2O_3/PTFE Icephobic Coatings for Protecting Aluminum Surfaces," *Surface and Coatings Technology*, 203, 1941–1946. [1417]

Miles, B. (2000), "Solar Heated Windshield," US Patent 6,066,372. [1417]

Mishchenko, L., Hatton, B., Bahadur, V., Taylor, J., Krupenkin, T., and Aizenberg, J. (2010), "Design of Ice-Free Nanostructured Impacting Water Droplets," *ASC NANO*, 4, 7699–7707. [1417]

Saito, H., Takai, K., and Yamauchi, G. (1997), "Water- and Ice-Repellent Coatings," *Surface Coatings International*, 80, 168–171. [1417]

Shah, K. R., and Sinha, B. K. (1989), *Theory of Optimal Designs*, New York: Springer-Verlag. [1418]

Simendinger, W. H. (2004), "Anti-Icing Composition," US Patent 6,702,953. [1417]