Lab-Scale Fiber Spinning Experimental Design Cost Comparison

Jeffrey C. Moreland, Julia L. Sharp, Ph.D. , Philip J. Brown, Ph.D.

Clemson University, Clemson, South Carolina UNITED STATES

Correspondence to:
Jeffrey C. Moreland email: jmorela@clemson.edu

ABSTRACT
Many statistical experimental designs are too costly or require too much raw material to be feasible for lab-scale fiber spinning experiments. In this study a four-factor response surface design is presented to study the fiber spinning process in detail at the lab scale. The time, cost, and amount of raw material required to execute the proposed design are compared to the typical completely randomized 2^4 factorial design used in fiber spinning experiments and also to a standard four-factor response surface design. Sample fiber data as well as analysis from a typical statistical software package is provided to further demonstrate the differences between each design. By designating some treatment factors in the design as hard-to-change, split-plotting is used to reduce the time, cost, and amount of raw material required to complete the experiment. The proposed split-plot design is faster and less expensive than a typical factorial design and has the advantage of fitting a more complex second-order model to the system. When compared to a standard response surface design, the proposed split-plot design provides the same second-order modeling capabilities but reduces the cost of the experiment by 53%, the total time by 36%, and the amount of polymer required by 24%. Thus, a split-plot response surface design based on hard-to-change factors is recommended in lab-scale spinning.

INTRODUCTION
Lab-scale experiments are important in the development of new fibers and spinning processes because they allow researchers to study the fiber formation process under more controlled and managed conditions and with less time and cost than is possible with large-scale equipment. However, the low capacity and relatively short operating times of lab-scale equipment limit the number of runs that can be conducted during the experiment and make it difficult to conduct experiments with many factors (e.g., speed) and levels (e.g., slow and fast) . In addition, the random assignment of the factor levels to experimental runs must be considered for an appropriate statistical analysis to be conducted on the resulting experimental data. A design that is completely randomized indicates that factor levels are completely reset between runs, which makes many statistical designs impractical for lab-scale finer spinning experiments since many factors of interest are difficult or costly to change and reset. Thus, the basis for this research is a statistical experimental design that is feasible and practical for lab-scale spinning experiments.

Traditionally the fiber spinning process has been studied using a one-at-a-time approach by varying the levels of a single treatment factor while keeping the levels of all other factors constant. The experiment would then be repeated for each factor of interest until all factors had been studied. This type of experiment is not only costly and inefficient to run, it also only provides limited information about the process as interaction effects between the factors are not studied [1]. Some fiber spinning processes such as melt spinning of polypropylene and wet spinning of acrylic have been studied using factorial designs [2-4], but the details of the experiment (e.g., randomization, replication, blocking) are unspecified or unclear, which leads to a lack of reproducibility.

The lack of knowledge regarding randomization, replication, and blocking can lead to improper analysis of the experimental data. The standard analysis of a two-level factorial design that is common in many scientific and industrial studies requires complete randomization and resetting of the treatment factor levels. In many cases, changing the levels of certain factors may be too costly or time consuming to allow for complete randomization so the experiment will be run with the levels of these hard-to-change factors fixed before resetting the experiment and moving on to the next level.

When the levels of the factors are not completely reset as in the example above, the experiment takes on a statistical design known as a split-plot that requires a different statistical analysis than a completely randomized design. In a split-plot design, the treatments are applied at different times or stages during the experiment resulting in experimental units or plots of different sizes *Figure 1* [1]. In the case above, the researcher inadvertently created a spilt-plot design by first applying the hard-to-change treatments to create large whole plots and then applying the easy-to-change treatments to smaller subplots. Unless the split-plot structure is accounted for in the statistical analysis of the experimental data, the researcher may draw incorrect conclusions about the effects of the treatment factors [1, 5].

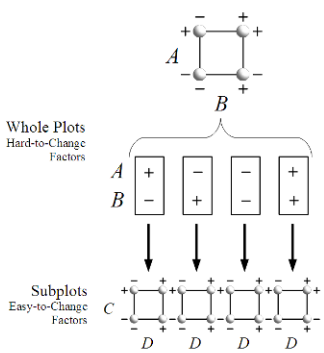

FIGURE 1. A split-plot design with two whole-plot factors and two subplot factors (adapted from Figure 14.7 in Montgomery [1]).

If the factors of the experiment are recognized as either easy-to-change (ETC) or hard-to-change (HTC) before the experiment, split-plotting can be used to limit the number of full resets between runs and create a more efficient experiment [6, 7]. This split-plot approach can also be applied to more advanced experimental designs like response surface designs [8] to create highly powerful and efficient experiments that can be used to study the fiber spinning process in great detail.

To demonstrate the benefits of a split-plot design, a four-factor response surface experiment for studying a lab-scale wet-spinning process will be designed and compared to a completely randomized 2^4 factorial design and a standard four-factor response surface design. The experimental design will be established so that the lab experiment can be conducted in a single day or shift. By setting up the experiment in this manner, the impact of nuisance factors such as multiple operators, environmental changes, and excessive down time between runs may be decreased or prevented. The total volume of polymer solution required for the experiment should be less than or equal to the capacity of the lab-scale equipment to prevent a multiple batch effect on the results. Each experimental run should also produce a sufficient length of testable yarn.

METHODS

A typical lab-scale wet spinning line *Figure 2* consists of three zones: (A) spinning, (B) drawing, and (C) take-up [9]. The spinning zone includes a polymer tank to hold the polymer solution during spinning and a metering pump to deliver an accurate amount of polymer solution to the spinneret. The spinneret contains many small holes that form the individual filaments of the yarn as the solution is pumped through the device. The liquid solution is solidified in the coagulation bath and excess solvent is removed by a wash roll. The drawing zone consists of a heated wash bath to assist in the drawing process and solvent removal. This zone also has a drying roll to remove excess moisture from the yarn before it enters the final take-up zone where it is wound onto a yarn tube using a tension-controlled winder.

The process conditions (solvent, speed, etc.) in the spinning and drawing zones will affect the structure and properties of the final fiber and are often used as treatment factors in experimental designs. The length of fiber produced and the time required to complete the experiment are also dependent on the spinning conditions and the speed of the final take-up.

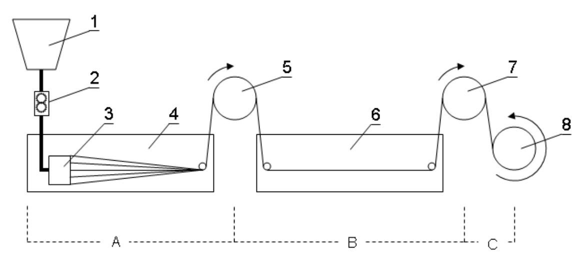

FIGURE 2. Schematic of a wet spinning line with three process zones: (A) Spinning, (B) Drawing, and (C) Take-up. The core components of the line are (1) polymer tank, (2) metering pump, (3) spinneret, (4) coagulation bath, (5) wash roll, (6) wash bath, (7) dry roll, and (8) take-up winder.

<u>Line And Experimental Parameters</u>

To determine the appropriateness of several experimental designs for a lab-scale spinning line, the parameters listed in *Table I* will be considered.

TABLE I. Processing parameters used to calculate the cost of a fiber spinning experiment.

Parameter	Description	Units
k_{ETC}	Number of easy-to-change (ETC) factors	
k_{HTC}	Number of hard-to-change (HTC) factors	
r	Number of experimental runs required	
b	Number of blocks (full resets) required	
M_L	Volume capacity of the polymer tank	mL
Q	Volumetric flow rate of pump	cm^3/min
N_h	Number of holes in the spinneret	
D_h	Diameter of each hole in the spinneret	cm
V_0	Average extrusion velocity defined in Eq. (1) [10]	m/min
V_L	Velocity of the take-up roll	m/min
S	Total stretch ratio defined in Eq, (2)	
L_r	Minimum length of fiber required for each experimental run	m
T_{HTC}	Time required to change/reset HTC factors (constant value)	
L_L	Length of the Spin Line	m
T_L	Time to reset/rethread line defined by Eq. (3)	hr

$$V_0 = \frac{\frac{Q}{N_h}}{\pi\left(\frac{D_h}{2}\right)^2(100)} \tag{1}$$

$$S = \frac{V_L}{V_0} \tag{2}$$

$$T_L = \frac{L_L}{V_L} \tag{3}$$

Note that in Eq. (3), the take-up velocity (**V_L**) is the same as the average extrusion velocity **(V_0)** so that the yarn is not stretched during rethreading (**$V_L = V_0$).**

Time Calculations

The total time required to complete the experiment can be calculated using the parameters in *Table I* and the following equations:

$$T_r = \frac{L_r}{V_L} \tag{4}$$

where **T_r** is the time required to collect samples for each experimental run.

$$T_R = \sum_{i=1}^{r} T_{r,i} = \sum_{i=1}^{r} \frac{L_{r,i}}{V_{L,i}} \tag{5}$$

where **T_R** is the total time to collect samples from all experimental runs.

$$T_b = T_{HTC} + T_L \tag{6}$$

where **T_b** is the time required to change HTC factors and reset line for each block.

$$T_B = bT_b \tag{7}$$

where **T_B** is the time required to change HTC factors and reset line for all *b* blocks.

$$T_E = T_R + T_B \tag{8}$$

where **T_E** is the total time required to complete experiment. The time required to change the ETC factors is considered negligible and is not included in the calculation of **T_E.**

Polymer Solution Calculations

The total volume of polymer solution required to complete the experiment can be calculated using the aforementioned time calculations, the spinning parameters listed in *Table I*, and the following equations:

$$M_r = T_r \cdot Q \tag{9}$$

where **M_r** is the volume of polymer solution required for each experimental run.

$$M_R = \sum_{i=1}^{r} M_{r,i} = \sum_{i=1}^{r} T_{r,i} \cdot Q \tag{10}$$

where **M_R** is the volume of polymer solution required for all experimental runs.

$$M_b = \left(\frac{L_L}{V_L}\right)Q \tag{11}$$

where **M_b** is the volume of polymer solution wasted for each block reset of the experiment and **$V_L = V_0$**.

$$M_B = bM_b \tag{12}$$

where **M_B** is the volume of polymer solution wasted for all *b* block resets.

$$M_E = M_R + M_B \quad (13)$$

where M_E is the total volume of polymer solution required to the experiment. Because the time required to change the ETC factors is negligible, the volume of polymer wasted during the change is also considered negligible and is not included in the calculation of M_E.

General Cost

In addition to the time and polymer solution required to carry out the experiment, other costs such as the cost of supplies, raw materials, or utilities should also be considered and can be expressed in terms of actual dollars or as a generic cost unit *C*. The cost to change the levels of the HTC and ETC factors can then be calculated based on a simple cost model using the following equations [6]:

$$C_R = rC_r, \quad (14)$$

where C_R is the general cost associated with changing all ETC factors, C_r is the general cost unit to change each ETC factor, and r is the number of experimental runs.

$$C_B = bC_b, \quad (15)$$

where C_B is the general cost associated with changing all HTC factors, C_b is the general cost unit to change each HTC factor, and b is the number of blocks (full resets).

$$C_E = C_R + C_B \quad \text{(Eq. 16)}$$

where C_E is the general cost of changing the HTC and ETC factors in the experiment.

RESULTS AND DISCUSSION

Example: Wet Spinning with 2 Hard-to-Change Factors and 2 Easy-to-Change Factors

Consider a four-factor lab-scale wet spinning experiment to study the effect of two coagulation additives and two stretch ratios on the initial modulus (*Y*) of the resulting fibers. Changing the coagulation conditions requires a full reset of the spin line, meaning that the coagulation tank must be emptied, cleaned, and refilled with a new coagulation mixture and that the line must be rethreaded before the each run; thus, the two coagulation factors (A and B) are designated as hard-to-change (HTC). The levels of the two stretch ratios (C and D) can be easily changed and reset while the line is running; thus, these factors are designated as easy-to-change (ETC). By utilizing a split-plot response surface design, this experiment can be efficiently conducted at the lab-scale to provide a detailed model of the effects of processing conditions on initial modulus. To demonstrate the benefits of this design, the data from a wet-spinning spinning experiment was analyzed as both a completely randomized 2-level full factorial with replicated center points (Design I) and a response surface design (Design II). The results of Designs I and II were compared to the conducted split-plot response surface design (Design III).

The lab-scale wet-spinning line used has a much smaller capacity and throughput than a typical full-scale production-scale line. The capacity of the polymer tank of the lab-scale line (M_L) is only 1 L (1000 mL) and the flow rate of the metering pump (Q) is 1 mL/min, which is 50-100 times less than some production lines [11]. The spinneret used in lab-scale experiments is also much smaller than a typical production spinneret containing only 20 holes (N_n) each with a diameter of 0.015 cm (D_h) resulting in an average extrusion velocity (V_0) of 2.83 m/min Eq. (1). A typical production spinneret could have hundreds of holes and have an extrusion velocity 10 to 20 times higher than the lab-scale line [11]. The length of the lab-scale spin line (L_L) is 36 m; thus, the time to rethread the line (T_L) is 12.7 min since the speed of the takeup roll (V_L) is the same as V_0 during rethreading Eq. (3). Because the pump runs at 1 mL/min, 12.7 mL of the polymer solution is required to rethread the line before each block (M_b). Twenty minutes are required to change the levels of the HTC factors (T_{HTC}) and when combined with the time to rethread the line (T_L), the total time lost per block (T_b) is 32.7 minutes. Each run should produce a minimum of 50 m of testable fiber to allow for characterization techniques such as tensile testing and examination of the fiber cross-sections to be performed. These processing parameters are summarized in *Table II*.

TABLE II. The processing parameters of the spin line used in the experiment.

Process Variable	Value
M_L	1000 mL
Q	1 mL/min
N_h	20
D_h	0.0150 cm
V_o (Eq. 1)	2.83 m/min
L_r	50 m
T_{HTC}	20 min
L_L	36 m
T_L (Eq. 3)	12.7 min
T_b (Eq. 6)	32.7 min
M_b (Eq. 11)	12.7 mL
C_r	1 cost unit
C_b	100 cost units

Design I: 2^4 Full Factorial with Replicated Center Points

A 2^4 factorial design is an experimental design in which four factors each having two levels are run in combinations to study the joint effect of the factors on the response [1]. The two levels of each factor are general coded as "-1" for the "low" level and "1" for the "high" level of the factor. In order to perform hypothesis tests for the effects of the factors, the experimental error must be estimated by replicating some or all of the experimental runs. A common practice is to augment the factorial design with replicated center points (coded as "0") midway between the low and high levels of the design rather than replicate all of the runs [1, 12]. The coded and uncoded levels of each factor are listed in *Table III.*

TABLE III. Coded and uncoded levels of each factor for Design I.

Factor	Factor Type	Low Level	Center Level	High Level	Level Type
		-1	0	1	Coded
A	HTC	1	2	3	Uncoded
B	HTC	1	2	3	
C	ETC	0.90	1.00	1.10	
D	ETC	1.075	1.15	1.225	

To calculate the time and amount of polymer needed to complete the experiment, the total stretch ratio (S) of each experimental run is first determined by multiplying the spin-stretch ratio (Factor C) and the draw-stretch ratio (Factor D) for each experimental run. The final take-up velocity (V_L) for each run is then calculated by multiplying the average extrusion velocity (V_0=2.83 m/min) by S Eq. (2). The time and amount of polymer required for each experimental run (T_r and M_r) can then be calculated using Equations 4 and 9, respectively. The individual times and amounts are then summed over all runs resulting in a total time of T_R = 341.6 min and a total amount of polymer M_R = 341.6 mL required to collect samples from all experimental runs. Because the pump is operating at 1 ml/min, the time and amount of polymer will have the same value (i.e., 1 minute of operating time requires 1 mL of polymer). The coded factor levels, resets, and resulting cost parameters and initial modulus response results (data collected during the execution of Design III) for each experimental run are listed in *Table IV*.

TABLE IV. Coded factor levels, resets, cost parameters, and responses for each run of Design I*.

Run	HTC A	HTC B	ETC C	ETC D	Reset	T_r (min)	M_r (mL)	*Y (cN/tex)*
1	-1	-1	-1	-1	1	18.3	18.3	719
2	-1	-1	1	-1	2	14.9	14.9	748
3	-1	-1	-1	1	3	16.0	16.0	879
4	-1	-1	1	1	4	13.1	13.1	969
5	1	-1	-1	-1	5	18.3	18.3	700
6	1	-1	1	-1	6	14.9	14.9	719
7	1	-1	-1	1	7	16.0	16.0	883
8	1	-1	1	1	8	13.1	13.1	874
9	-1	1	-1	-1	9	18.3	18.3	700
10	-1	1	1	-1	10	14.9	14.9	718
11	-1	1	-1	1	11	16.0	16.0	850
12	-1	1	1	1	12	13.1	13.1	952
13	1	1	-1	-1	13	18.3	18.3	698
14	1	1	1	-1	14	14.9	14.9	745
15	1	1	-1	1	15	16.0	16.0	814
16	1	1	1	1	16	13.1	13.1	821
17	0	0	0	0	17	15.4	15.4	798
18	0	0	0	0	18	15.4	15.4	761
19	0	0	0	0	19	15.4	15.4	755
20	0	0	0	0	20	15.4	15.4	773
21	0	0	0	0	21	15.4	15.4	823
22	0	0	0	0	22	15.4	15.4	731

*For clarity, the runs are listed in a standard order. During execution of the experiment, the run order is randomized.

Since a complete reset is required between each experimental run to ensure randomization, a total of 22 complete resets (which requires the additive mixture to be dumped 22 times and the machines to be completely shut down) are required for this design; thus the total time lost to change the HTC factors and reset the line is T_B = 719.4 min (12.0 hours) Eq. (7) and the total volume of polymer wasted is M_B = 279.4 mL Eq. (12). When added to the time and amount of polymer necessary for each run, the total time of the experiment is T_E= 1061.0 min or 17.7 hours Eq. (8) and the total amount of polymer required is M_E = 621.0 mL Eq. (13). The general cost for changing the ETC factors in all experimental runs is 22 cost units Eq. (14) and 2200 cost units for changing the HTC factors in all line resets Eq. (15) so the general cost of the experiment (C_E) is 2222 cost units.

Factorial designs can be analyzed using a procedure called analysis of variance (ANOVA) that is commonly performed in many statistical software packages. The resulting ANOVA can then be used to construct a model for the experiment and determine which treatment factors are significant in the model. Since each factor in a two-level factorial design has only two levels, only linear effects (i.e., first order models) can be estimated with this type of design. The addition of center points to the design can be used to detect curvature of the linear model, which is an indication that a higher order model should be used to describe the data [1].

The data for Design I was analyzed using the *proc glm* procedure of the SAS/STAT software, Version 9.1 of the SAS System for Windows [13]. The resulting ANOVA table including a test for curvature [1] is listed in *Table V* with the estimated treatment effects listed in *Table VI*. The significance of each effect is determined by comparing the effect estimate to its standard error using a *t*-test [1] with a critical *t* value of 2.201 at α=0.05. The effects are displayed graphically by the Pareto chart in *Figure 3* showing the *t* value for each factor in relation to the critical *t* value and by the main effect plots in *Figure 4*. The analysis revealed that ETC-D has the greatest effect on *Y* followed by ETC-C, and that both have an increasing effect. There is also no evidence of curvature in the model indicating that a first order model is appropriate.

TABLE V. ANOVA table for Design I.

Source	DF	Sum of Squares	Mean Square	F	P
Model	10	124957	12495.7	10.84	<0.001
Main Effects	4	117815	29453.8	25.54	<0.001
2-Way Interactions	6	7141	1190.2	1.03	0.452
Total Error	11	12686	1153.2		
Curvature	1	2908	2907.5	2.71	0.160
Lack of Fit	5	4423	884.5		
Pure Error	5	5356	1071.1		
Total	21	137642			

TABLE VI. Model parameter estimates for the treatments effects of Design I.

Parameter	Estimate	Standard Error	t Value	P
Intercept	792.3	7.2	109.43	<.0001
ETC-D	80.9	8.5	9.53	<.0001
ETC-C	18.9	8.5	2.23	0.0475
HTC-A	-17.6	8.5	-2.07	0.0629
HTC-A*ETC-D	-14.7	8.5	-1.73	0.1115
HTC-B	-12.1	8.5	-1.42	0.1831
HTC-A*ETC-C	-10.9	8.5	-1.29	0.2241
HTC-B*ETC-D	-8.9	8.5	-1.05	0.3150
ETC-C*ETC-D	4.8	8.5	0.57	0.5822
HTC-B*ETC-C	2.8	8.5	0.33	0.7467
HTC-A*HTC-B	-0.2	8.5	-0.02	0.9828

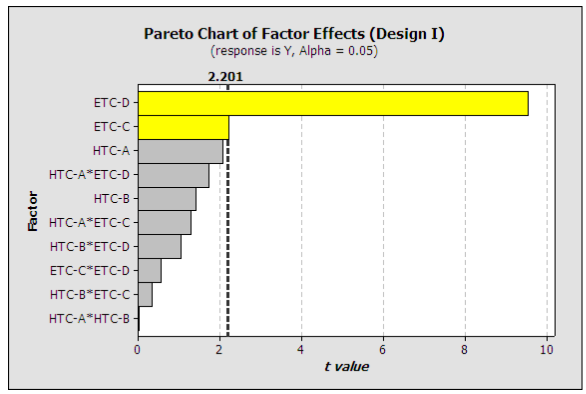

FIGURE 3. Pareto chart of the treatment factor effects showing statistical significance. Factors in yellow are significant at α=0.05 (plot created using Minitab 15 statistical software [14]).

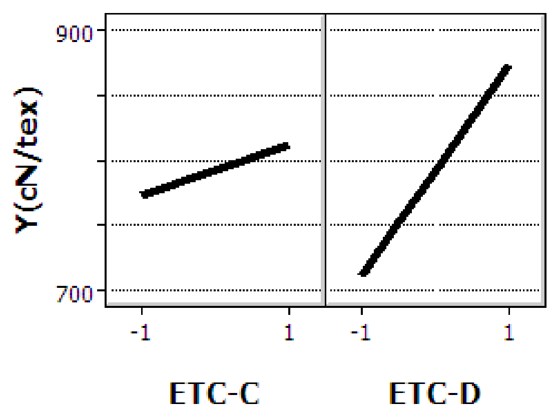

FIGURE 4. Main effect plots for Design I showing the effect of each factor on Initial Modulus.

Design II: 4-Factor Response Surface Design (Completely Randomized)

A 2-level factorial design such as Design I is only capable of fitting a first-order model since each factor is only tested at two levels. A first-order model may not adequately describe a complex system such as fiber spinning, so a higher second-order model to analyze such a system may be necessary and preferred. A second-order model can be fit using a response surface design with more treatment levels; a rotatable central composite design (RCCD) is a response surface experimental design that appropriately considers higher-order models. A RCCD consists of the same 2^4 full factorial with replicated center points as Design I but is augmented with additional axial points at some distance α from the coded center point of 0. The value of $\alpha = 2^{k/4}$ where k is the number of treatment factors; for the four-factor design $\alpha = 2^{4/4} = 2$ [1, 12, 15]. The coded and corresponding uncoded levels of each factor for Design II are listed in *Table VII*.

TABLE VII. Coded and uncoded factor levels for Design II.

Factor	Low Axial Level	Low Level	Center Level	High Level	High Axial Level	Level Type
	-2	-1	0	1	2	Coded
HTC-A	0	1	2	3	4	Uncoded
HTC-B	0	1	2	3	4	
ETC-C	0.800	0.900	1.000	1.100	1.200	
ETC-D	1.000	1.075	1.150	1.225	1.300	

The time and amount of polymer necessary for Design II are calculated in the same manner as for Design I resulting in T_R = 466.4 min (7.8 hours) and M_R = 466.4 mL. The coded factor levels, resets, and resulting cost parameters and initial modulus response results (data collected during the execution of Design III) for each experimental run are listed in *Table VIII.*

TABLE VIII. Coded factor levels, resets, cost parameters, and responses for each run of Design II*.

Run	HTC A	HTC B	ETC C	ETC D	Reset	T_r (min)	M_r (mL)	*Y (cN/tex)*
1	-1	-1	-1	-1	1	18.3	18.3	719
2	-1	-1	1	-1	2	14.9	14.9	748
3	-1	-1	-1	1	3	16.0	16.0	879
4	-1	-1	1	1	4	13.1	13.1	969
5	1	-1	-1	-1	5	18.3	18.3	700
6	1	-1	1	-1	6	14.9	14.9	719
7	1	-1	-1	1	7	16.0	16.0	883
8	1	-1	1	1	8	13.1	13.1	874
9	-1	1	-1	-1	9	18.3	18.3	700
10	-1	1	1	-1	10	14.9	14.9	718
11	-1	1	-1	1	11	16.0	16.0	850
12	-1	1	1	1	12	13.1	13.1	952
13	1	1	-1	-1	13	18.3	18.3	698
14	1	1	1	-1	14	14.9	14.9	745
15	1	1	-1	1	15	16.0	16.0	814
16	1	1	1	1	16	13.1	13.1	821
17	-2	0	0	0	17	15.4	15.4	765
18	2	0	0	0	18	15.4	15.4	729
19	0	-2	0	0	19	15.4	15.4	849
20	0	2	0	0	20	15.4	15.4	777
21	0	0	-2	0	21	19.2	19.2	771
22	0	0	2	0	22	12.8	12.8	880
23	0	0	0	-2	23	17.7	17.7	662
24	0	0	0	2	24	13.6	13.6	1022
25	0	0	0	0	25	15.4	15.4	798
26	0	0	0	0	26	15.4	15.4	761
27	0	0	0	0	27	15.4	15.4	755
28	0	0	0	0	28	15.4	15.4	773
29	0	0	0	0	29	15.4	15.4	823
30	0	0	0	0	30	15.4	15.4	731

*For clarity, the runs are listed in a standard order. During execution of the experiment, the run order is randomized.

Design II requires a total of 30 complete resets (8 more resets than in Design I); thus more time is lost changing the HTC factors and resetting the line (T_B = 981.0 min (16.4 hr)) (Eq. 7), and more polymer is wasted (M_B = 381.0 mL) (Eq. 12) than for Design I. The total time of the experiment for Design II is T_E = 1447.4 min (24.1 hr) (Eq. 8) and the total amount of polymer is M_E = 847.4 mL (Eq. 13). The general cost for all experimental runs is 30 cost units (Eq. 14) and 3000 cost units for all line resets (Eq. 15) making the general cost of the experiment for Design II (C_E) 3030 cost units.

The *proc rsreg* procedure of SAS/STAT [13] used to analyze the response surface data of Design II, and the ANOVA (*Table IX*) revealed that a second-order model is a better fit than the first-order model indicated by Design I. In addition, all four main factors, HTC-A, HTC-B, ETC-C, and ETC-D, have a significant effect on Y with ETC-C and ETC-D having the greatest effect at α=0.05 (*Table X and Figure 5*). The main effects plot (*Figure 6*) and contour plots of factors ETC-C and ETC-D versus predicted values of *Y* (*Figure 7)* illustrate the second-order relationship.

TABLE IX. ANOVA table for Design II.

Source	DF	Sum of Squares	Mean Square	F value	P
Model	14	211811		19.79	< 0.001
Linear	4	190410		62.26	< 0.001
Quadratic	4	14260		4.66	0.0120
Crossproduct	6	7141	1197.6	1.56	0.2272
Total Error	15	11469	764.6		
Lack-of-Fit	10	6113	611.3	0.57	0.7894
Pure Error	5	5356	1071		
Total	29				

TABLE X. Model parameter estimates for the treatments effects of Design I.

Parameter	Estimate	Standard Error	t Value	P
Intercept	773.5	11.3	68.52	<0.001
ETC-D	84	5.6	14.87	<0.001
ETC-C	21.7	5.6	3.85	0.002
ETC-D*ETC-D	15.9	5.3	3.00	0.009
HTC-A	-14.7	5.6	-2.61	0.020
HTC-B	-14	5.6	-2.49	0.025
ETC-C*ETC-C	11.7	5.3	2.22	0.042
HTC-A*ETC-D	-14.7	6.9	-2.12	0.051
HTC-B*HTC-B	8.6	5.3	1.63	0.124
HTC-A*ETC-C	-10.9	6.9	-1.58	0.135
HTC-A*HTC-A	-7.9	5.3	-1.49	0.156
HTC-B*ETC-D	-8.9	6.9	-1.29	0.216
ETC-C*ETC-D	4.8	6.9	0.70	0.497
HTC-B*ETC-C	2.8	6.9	0.41	0.690
HTC-A*HTC-B	-0.2	6.9	-0.03	0.979

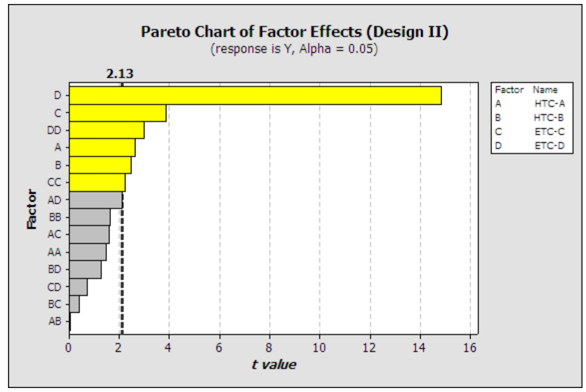

FIGURE 5. Pareto chart of the treatment factor effects showing statistical significance. Factors in yellow are significant at α=0.05 (plot created using Minitab 15 statistical software [14]).

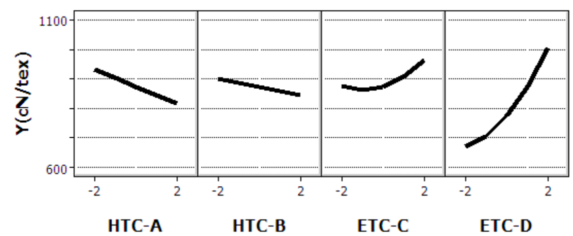

FIGURE 6. Main effect plots for Design II showing the effect of each factor on initial modulus.

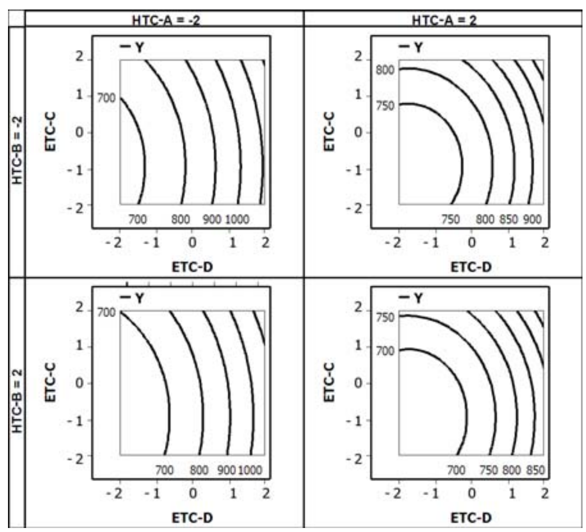

FIGURE 7. Contour plots of predicted values of initial modulus versus coded levels of the HTC factors at high and low levels of the ETC factors for Design II.

Design III: 4-Factor Split-Plot Response Surface Design with Non-Random Center Points

To decrease the number of complete resets required for Design II, a split-plot structure can be utilized by blocking (grouping) the experimental runs that have the same combinations of levels of the two HTC factors. For example, all runs that contain both the low level of HTC-A (-1) and the low level of HTC-B (-1) would fall into the same block. The remaining runs are then assigned to separate blocks in the same manner with each block representing a complete reset of the spin line. The blocking structure of Design III is listed in *Table XI* and illustrated in *Figure 8*.

Rather than combining all six replicated center points into a single block, the center points can be run independently and distributed throughout the design and can be used to provide information about the stability of the process over time in addition to appropriately estimating the experimental error [12]. For example, the center points in runs 26-30 in *Table XI* can be distributed as follows: Run 26 before Reset 1, Run 27 between Reset 2 and 3, Run 28 between Reset 4 and 5, and so forth. Because the center points of the design have the same combination of HTC factor levels as the axial points of the design (Reset 9 in *Table XI)*, one center point (Run 25) can also be run within that block.

TABLE XI. Split-plot blocking structure of Design III.

Run	HTC A	HTC B	ETC C	ETC D	Block A	Block B	Reset
1	-1	-1	-1	-1	1	1	1
2	-1	-1	1	-1	1	1	
3	-1	-1	-1	1	1	1	
4	-1	-1	1	1	1	1	
5	1	-1	-1	-1	2	1	2
6	1	-1	1	-1	2	1	
7	1	-1	-1	1	2	1	
8	1	-1	1	1	2	1	
9	-1	1	-1	-1	3	2	3
10	-1	1	1	-1	3	2	
11	-1	1	-1	1	3	2	
12	-1	1	1	1	3	2	
13	1	1	-1	-1	4	2	4
14	1	1	1	-1	4	2	
15	1	1	-1	1	4	2	
16	1	1	1	1	4	2	
17	-2	0	0	0	5	3	5
18	2	0	0	0	6	3	6
19	0	-2	0	0	7	4	7
20	0	2	0	0	7	5	8
21	0	0	-2	0	8	6	9
22	0	0	2	0	8	6	
23	0	0	0	-2	8	6	
24	0	0	0	2	8	6	
25	0	0	0	0	8	6	
26	0	0	0	0	8	6	10
27	0	0	0	0	8	6	11
28	0	0	0	0	8	6	12
29	0	0	0	0	8	6	13
30	0	0	0	0	8	6	14

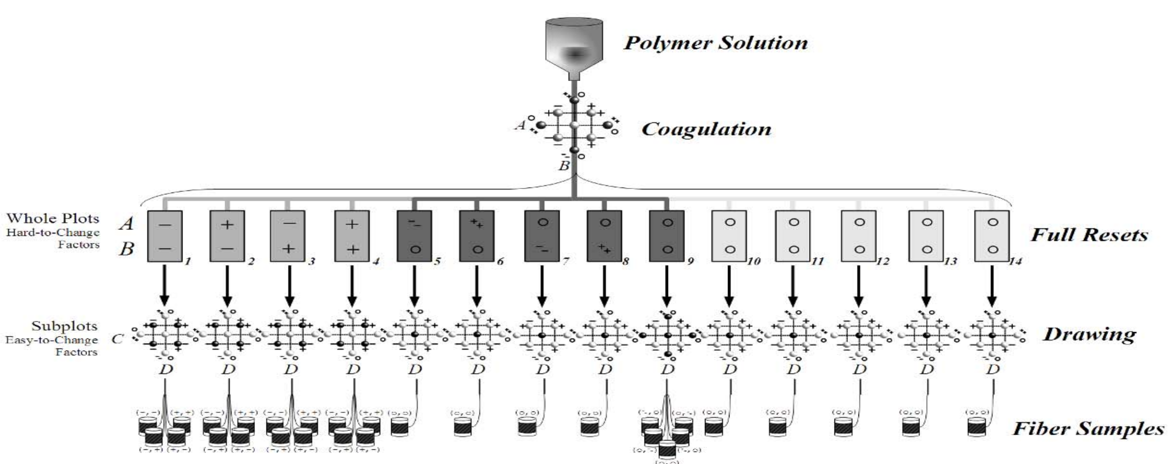

FIGURE 8. Split-plot design structure of Design III showing the whole plots created by the full resets of the HTC coagulation factors and the subplots created by the ETC drawing factors resulting in 30 fiber samples (adapted from Figure 14.7 in Montgomery [1]).

The individual treatment runs in Design III are identical to those in Design II; therefore, the time and amount of polymer calculations are also the same (T_R = 466.4 min (7.8 hours) and M_R = 466.4 mL). However, Design III only requires 14 complete resets, thus the total time lost to changing the HTC factors and resetting the line (T_B) and the total amount of polymer wasted (M_B) are less than the lost time and wasted polymer in Design II (457.8 min and 177.8 mL, respectively). When added to the time and amount of polymer necessary for each run, the total time of the experiment (T_E) is 942.2 min (15.7 hours) and the total amount of polymer required (M_E) is 644.2 mL. The general cost for all experimental runs in Design III is the same as Design II at 30 cost units (Eq. 14) but the cost for all resets is much less at 1400 cost units, begetting the general cost of the experiment much less at 1430 cost units.

Since Design III is in a split-plot arrangement, a mixed-model analysis must be used to determine the treatment effects and the effect of the blocking factor. For this experiment, the *proc mixed* function of SAS software, Version 9.1 [13], was used, and it was found that blocking did not have a significant effect on the experiment as indicated by a covariance parameter estimate of zero. Thus, the treatment effects can be estimated using the same response surface analysis as Design II, which results in identical parameter estimates for the two designs [5].

Summary of Designs

The total time, the cost of the experiment, and amount of polymer required for each design is summarized in *Table XII* and in *Figure 9*. Design III requires the least amount of time and cost to complete and requires only slightly more polymer than Design I. Design III is also capable of fitting a second-order model while Design I is limited to a first-order model. While Design II is also capable of fitting a second-order model, the split-plot structure of Design III reduces the cost of the experiment from Design II by 53%, the total time by 36%, and the amount of polymer required by 24%.

TABLE XII. Design summaries.

Parameter	Design I	Design II	Design III	Units
No. of Runs (***r***)	22	30	30	runs
No. of Resets (***b***)	22	30	14	blocks
Total Time (T_E)	17.7	24.1	15.4	hr
Total Amount of Polymer (M_E)	621.0	847.4	644.2	mL
Total General Cost (C_E)	2222	3030	1430	c.u.
Highest Order Model	1st-order	2nd-order	2nd-order	

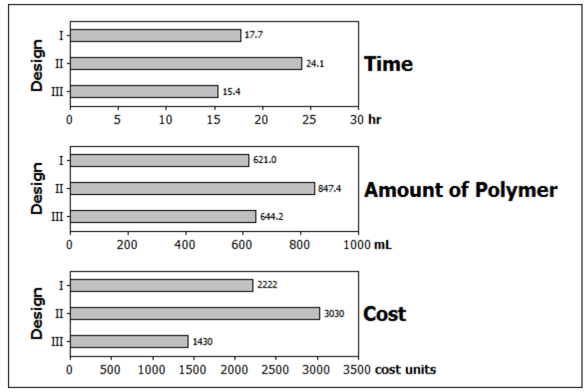

FIGURE 9. Comparison of the total time, amount of polymer, and cost for completing each of three experimental designs.

CONCLUSIONS AND RECCOMMENDATIONS

As evidenced by the wet-spinning example provided, a split-plot response surface design based on hard-to-change factors has many advantages over completely randomized designs for studying lab-scale fiber spinning processes. The improved modeling capabilities of the response surface design combined with the cost reductions of the split-plot structure can be utilized to study lab-scale processes in greater detail and with less time, raw materials, and cost than would otherwise be possible with completely randomized designs. In the example provided, a split-plot response surface design was used to provide a detailed second-order model for the effect of spinning conditions on initial modulus while minimizing the time, cost, and raw material requirements that would have otherwise complicated the implementation of the experiment at the lab scale. In the past, similar lab-scale experiments might have been conducted without completely resetting the factor levels in between each run in order to reduce the cost and time of the experiment. However, the subsequent statistical analysis could result in biased estimates and results since randomization of the design was restricted [5, 7]. Thus a split-plot design based on hard-to-change factors such as the one described in this research provides an ideal solution for experimental design at the lab-scale. While this research focused on the wet-spinning process, other lab-scale textile processes such as melt spinning and yarn formation could also benefit from the split-plotting technique.

ACKNOWLEDGEMENTS
This research was supported by the ERC program of the National Science Foundation under Award Number EEC-9731680 with support from the Clemson University Research Foundation and Ansell Healthcare Products, LLC.

REFERENCES

[1] Montgomery, D. C., *Design and Analysis of Experiments*, 7th ed., John Wiley & Sons: New York, 2009.
[2] Yang, R. D., et al., "The application of factorial experimental design to the processing of polypropylene fibres", *Journal of Materials Science*, 36, 13, 2001.
[3] Hersh, S. P., T. D. H. H. W. K., "The influence of processing variables on the physical properties of a wet-spun modacrylic fiber", *Journal of Applied Polymer Science*, 7, 2, 1963.
[4] Ruodan Yang, R. R. M. A. F. F., "Processing, structure, and mechanical properties of as-spun polypropylene filaments - A systematic approach using factorial design and statistical analysis", *Journal of Applied Polymer Science*, 96, 1, 2005.
[5] Webb, D. F., et al., "Factorial experiments when factor levels are not necessarily reset", *Journal of Quality Technology*, 36, 1, 2004.
[6] Anbari, F. T. and Lucas, J. M., "Designing and running super-efficient experiments: Optimum blocking with one hard-to-change factor", *Journal of Quality Technology*, 40, 1, 2008.
[7] Kowalski, S. M. and Patcner, K. J., "How to recognize a split-plot experiment", *Quality Progress*, 36, 11, 2003.
[8] Vining, G. G., et al., "Response surface designs within a split-plot structure", *Journal of Quality Technology*, 37, 2, 2005.
[9] Ziabicki, A., *Fundamentals of Fibre Formation*, ed., John Wiley & Sons: London, 1976.
[10] Paul, D. R., "Spin orientation during acrylic fiber formation by wet-spinning", *Journal of Applied Polymer Science*, 13, 5, 1969.
[11] Fourné, F., *Synthetic Fibers: Machines and Equipment, Manufacture, Properties*, ed., Hanser Publishers: Munich, 1999.
[12] Myers, R. H. and Montgomery, D. C., *Response Surface Methodology: Process and Product in Optimization Using Designed Experiments*, ed., John Wiley & Sons, Inc. New York, NY, USA: 1995.
[13] *SAS Software Version 9.1*, SAS Institute Inc.: Cary, NC, 2003.
[14] *Minitab 15 Statistical Software*, Minitab, Inc.: State College, PA, 2007.
[15] Cochran, W. G. and Cox, G. M., "Some methods for the study of response surfaces", *Experimental designs*, John Wiley: New York, 1957.

AUTHORS' ADDRESS

Jeffrey C. Moreland
Clemson University
Materials Science and Engineering
511 Westinghouse Road
Pendleton, SC 29625
UNITED STATES

Philip J. Brown, Ph.D.
Clemson University
Materials Science and Engineering
161 Sirrine Hall
Clemson, South Carolina 29634

Julia L. Sharp, Ph.D.
Clemson University
Applied Economics & Statistics
237 Barre Hall
Clemson, South Carolina 29634