Factorial experimental design based on multiple factors for sensors and actuators development

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ABSTRACT – REZUMAT

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This paper presents several use cases for a full factorial experimental design method used in the development of flexible sensors and actuators. The full factorial experimental designs consisted of 4 factors with discrete values (3–4 levels) based on known parameters of the experimental devices. In general, the selected factors can influence other dependent variables. This study aims to investigate the main effects and the interaction effects (antagonistic, synergistic, ceiling) among the different factors and to optimize an experimental design for reducing the consumption (raw materials, water, energy, chemicals) and obtaining the optimal values for surface electrical resistance using a reduced number of experiments. The use of the complete factorial experimental plan and optimization with a minimization function helps to select, from the set of possible experiments, the experiments including the optimal parameters for obtaining the desired result. Therefore, the number of experimental plans and the corresponding amount of resource consumption is reduced (e.g., from 81–256 experiments to 10–20 experiments) while obtaining electroconductive textile electrodes for sensors and actuators.

Keywords: *factorial design, sensors, actuators, textile, conductive, electrical resistance, experiment, optimization*

Proiectare experimentală factorială pe bază de factori multipli pentru dezvoltarea senzorilor şi actuatorilor

Această lucrare prezintă mai multe cazuri de utilizare pentru metoda de proiectare experimentală factorială completă pentru dezvoltarea senzorilor și actuatorilor flexibili. Proiectarea experimentală factorială completă a avut la bază 4 factori cu valori discrete (3–4 niveluri) ale unor parametri cunoscuți pentru dispozitivele experimentale. În general, factorii selectați pot influența alte variabile dependente. Acest studiu își propune să investigheze principalele efecte și interacțiunea efectelor (antagoniste, sinergice, plafon) între diferiții factori și să optimizeze proiectarea experimentală pentru reducerea consumului (materii prime, apă, energie, substanțe chimice) și obținerea unor valori optime pentru rezistenţa electrică de suprafață utilizând un număr redus de experimente. Utilizarea planului experimental factorial complet și optimizarea prin intermediul funcției de minimizare ajută la selectarea, din setul de experimente posibile, a experimentelor care prezintă parametrii optimi pentru obținerea rezultatului dorit. Prin urmare, numărul de planuri experimentale și cantitatea corespunzătoare de resurse consumate sunt reduse (de exemplu, de la 81–256 de experimente la 10–20 de experimente) pentru obţinerea electrozilor textili electroconductivi pentru senzori și actuatori.

Cuvinte-cheie: *proiectarea factorială, senzori, actuatori, textil, conductiv, rezistenţă electrică, experiment, optimizare*

INTRODUCTION

To create smart textile-based sensors or actuators, there are two types of integration in textile products, such as textiles with conventional electronics (e.g., sensors or actuators mounted on PCB boards), based on whether the textile material is a support material or surface electrodes for textile actuators or sensors, which offer easy integration and operate on capacitive, resistive and piezoelectric principles. In these systems, structural integration occurs on small surfaces of a few mm or cm [1–5]. In this work, experimental plans are reported for reducing redundant experiments using static methods (e.g., using full factorial design, optimization or principal component analysis).

In research, factorial experimental design is part of an experimental plan that includes both important and insignificant factors. To develop a factorial design, researchers identify important factors (independent variables) and responses to these factors (dependent variables) for use in the optimization and development of the experimental plan. A full factorial design involves at least 2 factors with different value levels (2 to *n*), and the experimental units cover all possible combinations of these factors having different levels. The factorial experimental design method can be full or fractional, and it is used in numerous studies to reduce the consumption, time and costs allocated for new studies. Full factorial design has been used in numerous investigations, such as temperature for e-textiles [6–8], fabric reinforcement or optimization of carbon electrode parameters [9], parameter optimization of photoelectrocatalytic degradation of a textile dye [10], optimization of carbon electrodes based on commercial activated carbons with differing surface areas and pore dimensions [11, 12], and design of piezoresistive sensors [14–19].

EXPERIMENTAL PART

To develop a full factorial design for sensors, 4 factors (temperature1, temperature2, concentration, time) and a dependent variable (electrical resistance) were considered. Table 1 shows the factors used for experimental plan design in the case of the sensors, where:

- concentration refers to the amount of metal particles in a certain amount of polymer used as the matrix;
- temperature1 (*Temp*$_1$, °C) is the temperature used for crosslinking;
- temperature2 (*Temp*$_2$, °C) is the temperature of the prepared conductive paste;
- time (*T*, min) refers to the time allocated for crosslinking;
- surface resistance (*Rs*, Ω).

Table 1

FACTORS INFORMATION FOR SENSORS

Factor	Levels	Values
Temperature1 (°C)	3	70, 80, 90
Temperature2 (°C)	3	150, 160, 170
Concentration (%)	3	10, 20, 90
Time (min)	3	5, 10, 15

In our development of a full factorial design for an actuator, 4 factors: rotational speed, volume, concentration, and time) specific to the method of thin film deposition by spin coating methods (rotational speed, time) and the parameters specific to the conductive polymer dispersion, such as the volume and concentration of micro/nanoparticles in polymeric dispersions, were used. Thus, 4 variable influence factors and 4 distinct values for each factor were considered, as follows: rotational speed (250, 500, 750, 1000), time (10, 15, 30, 60), concentration (10, 20, 30, 40) and volume (100, 250, 500, 1000). Table 2 presents the factors used for actuator development.

Table 2

FACTORS INFORMATION FOR ACTUATORS

Factor	Levels	Values
Rotational speed (rpm)	4	250, 500, 750, 1000
Volume (µl)	4	100, 250, 500, 1000
Concentration (%)	4	10, 20, 30, 40
Time (s)	4	10, 15, 30, 60

In an experimental plan, using the factorial method redundant variables to reconstruct $np\,x_{ij}$ values from an $Xn{\times}p$ table, through the reduction of rank q, using equation 1:

$$X = u_1 v_1' + u_2 v_2' + \cdots + u_q v_q' + E \qquad (1)$$

where *E* is the residual matrix that can reconstruct from $q(n+p)$ values of the vectors u_α and v_α (α = 1,..., q) the np values of X.

Table 3

FULL FACTORIAL PLAN DESIGN FOR FLEXIBLE SENSORS

Std Order	Run Order	Temp$_1$ (°C)	Temp$_2$ (°C)	Concentration (%)	Time (s)
33	1	80	150	20	15
47	2	80	170	10	10
1	3	70	150	10	5
66	4	90	160	10	15
17	5	70	160	90	10
58	6	90	150	20	5
32	7	80	150	20	10
4	8	70	150	20	5
55	9	90	150	10	5
28	10	80	150	10	5
44	11	80	160	90	10
48	12	80	170	10	15
5	13	70	150	20	10
35	14	80	150	90	10
53	15	80	170	90	10
60	16	90	150	20	15
40	17	80	160	20	5
34	18	80	150	90	5
6	19	70	150	20	15
41	20	80	160	20	10
9	21	70	150	90	15
24	22	70	170	20	15
63	23	90	150	90	15
81	24	90	170	90	15
59	25	90	150	20	10
57	26	90	150	10	15
25	27	70	170	90	5
52	28	80	170	90	5
31	29	80	150	20	5
16	30	70	160	90	5
71	31	90	160	90	10
72	32	90	160	90	15
23	33	70	170	20	10
78	34	90	170	20	15
22	35	70	170	20	5
18	36	70	160	90	15
37	37	80	160	10	5

Note: Temp$_1$ is the temperature used for preparing the electroconductive paste; Temp$_2$ is the temperature used for the crosslinking process; Concentration refers to the metallic micro/nanoparticles from polymeric paste; Time refers to the time allocated for the cross-linking process.

Principal component analysis consists of the algebraic decomposition of a data matrix into a structure of components (factors) having the greatest common variability. Eigen analysis of the correlation and covariance matrix of the principal components for sensors development is presented in table 5, and the eigenvectors for sensors development are presented in table 6. The analysis of principal factors identifies a small number of factors (latent variables) that explain the common variance of the variables (concentration, temperature1, temperature2, time) that subsequently influence the dependent variable (electrical resistance).

Table 7 presents the unrotated matrix of the principal factors and the matrix rotated by the Varimax method for sensors development.

The Eigen analysis of the correlation and covariance matrix of the principal components for actuators' development is presented in table 8, and the eigenvectors for actuators' development are presented in table 9. The analysis of principal factors identifies a small number of factors that explain the common variance of the variables (rotational speed, volume, concentration and time) that subsequently influence the dependent variable (electrical resistance).

Table 10 presents the unrotated matrix of the principal factors and the matrix rotated by the Varimax method for actuators' development.

To graphically represent the correlation between the values of the variables (temperature$_1$, temperature$_2$, concentration and time), we generated a correlogram (figure 1), which compares Pearson correlation coefficients for each pair of variables, indicating properties including the direct/indirect and linear relationship between the variable pair, depending on the corresponding electrical resistance values. Figure 1 shows that between concentration and temperature$_2$ and between time and temperature$_2$, where there is a direct linear relationship because the correlation coefficients are very close to 1 (Correlation_coefficient$_{time,temp2}$ = 0.9; Correlation_coefficient$_{Concentration,temp2}$ = 0.87) for obtaining a textile electrode for flexible sensors with low surface resistance (*Rs* = 20 Ω).

Table 4

FULL FACTORIAL PLAN DESIGN FOR FLEXIBLE SENSORS

Std Order	**Run Order**	**Speed^1 (rpm)**	**Volume^2 (µl)**	**Concentration^3 (%)**	**Time^4 (s)**
10	1	250	100	30	15
158	2	750	250	40	15
215	3	1000	250	20	30
13	4	250	100	40	10
24	5	250	250	20	60
141	6	750	100	40	10
184	7	750	1000	20	60
198	8	1000	100	20	15
17	9	250	250	10	10
232	10	1000	500	20	60
102	11	500	500	20	15
68	12	500	100	10	60
139	13	750	100	30	30
143	14	750	100	40	30
126	15	500	1000	40	15
176	16	750	500	40	60
86	17	500	250	20	15
2	18	250	100	10	15
252	19	1000	1000	30	60
218	20	1000	250	30	15
42	21	250	500	30	15
51	22	250	1000	10	30

Note: Speed represents the rotational speed (rpm) for the spin coater necessary for thin film deposition; Volume represents the volume of the conductive dispersion used to be deposited on the textile surface; Concentration represents the amount of metallic micro/nanoparticles in the polymeric dispersion; Time represents the necessary time for the spin coating process.

Table 5

EIGENANALYSIS OF THE CORRELATION AND COVARIATION MATRICES FOR SENSORS

Variable	**Correlation matrix**				**Covariance matrix**			
Eigenvalue	1	1	1	1	1282.5	67.5	67.5	16.9
Proportion	0.25	0.25	0.25	0.25	0.894	0.047	0.047	0.012
Cumulative	0.25	0.5	0.75	1	0.894	0.941	0.988	1

Table 6

EIGENVECTORS FOR CORRELATION AND COVARIANCE FOR FLEXIBLE SENSORS

Variable	**Correlation**				**Covariance**			
	PC1	**PC2**	**PC3**	**PC4**	**PC1**	**PC2**	**PC3**	**PC4**
Temp$_1$	0	0	0	–1	0	–1	0	0
Temp$_2$	0	0	–1	0	0	0	-1	0
Concentration	–1	0	0	0	–1	0	0	0
Time	0	–1	0	0	0	0	0	–1

Table 7

PRINCIPAL COMPONENT FACTOR ANALYSIS OF THE CORRELATION AND COVARIANCE MATRICES FOR SENSORS

Variable	**Unroted matrix**				**Roted matrix**				**Commu-nality**
	Factor1	**Factor2**	**Factor3**	**Factor4**	**Factor1**	**Factor2**	**Factor3**	**Factor4**	
$Temp_1$	0	0	0	–1	0	0	0	1	1
$Temp_2$	0	0	–1	0	0	0	1	0	1
Concentration	–1	0	0	0	1	0	0	0	1
Time	0	–1	0	0	0	1	0	0	1
Variance	1	1	1	1	1	1	1	1	4
% Var	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	1

Table 8

EIGENANALYSIS OF THE CORRELATION AND COVARIANCE MATRICES FOR ACTUATORS

Variable	**Correlation matrix**				**Covariance matrix**			
Eigenvalue	1	1	1	1	117176	78431	381	125
Proportion	0.25	0.25	0.25	0.25	0.597	0.4	0.002	0.001
Cumulative	0.25	0.5	0.75	1	0.597	0.997	0.999	1

Table 9

EIGENVECTORS FOR CORRELATION AND COVARIANCE FOR ACTUATORS

Variable	**Correlation**				**Covariance**			
	PC1	**PC2**	**PC3**	**PC4**	**PC1**	**PC2**	**PC3**	**PC4**
Rotational speed	0	–1	0	0	0	–1	0	0
Volume	0	0	0	–1	–1	0	0	0
Concentration	0	0	–1	0	0	0	0	–1
Time	–1	0	0	0	0	0	–1	0

Table 10

PRINCIPAL COMPONENT FACTOR ANALYSIS OF THE CORRELATION AND COVARIANCE MATRICES FOR ACTUATORS

Variable	**Unroted matrix**				**Roted matrix (method Varimax)**				**Commu-nality**
	Factor1	**Factor2**	**Factor3**	**Factor4**	**Factor1**	**Factor2**	**Factor3**	**Factor4**	
Rotational Speed	0	–1	0	0	0	1	0	0	1
Volume	0	0	0	–1	0	0	0	1	1
Concentration	0	0	–1	0	0	0	1	0	1
Time	–1	0	0	0	1	0	0	0	1
Variance	1	1	1	1	1	1	1	1	4
% Var	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25	1

Table 11 presents the variance analysis of the independent variables (temperature1, temperature2, concentration, time).

DF represents the number of degrees of freedom. The number of degrees of freedom of the model error (DF) is given by equation 2.

$$DF_{Error} = n - 1 - \left(\sum DF_{factor_i} + \sum DF_{interact_{ij}}\right) \quad (2)$$

where n is the number of observations in the experiment; DF_{factor_i} represents the number of degrees of freedom of factor i; $DF_{interact_{ij}}$ represents the number

Table 11

VARIANCE ANALYSIS			
Source	**DF**	**Adj SS**	**Adj MS**
Model	80	921000000	11511760
Linear	8	126000000	15709203
$Temp_1$	2	33881511	16940756
$Temp_2$	2	39095622	19547811
Concentration	2	19063806	9531903
Time	2	33632683	16816341
2-Way Interactions	24	190000000	7896005
$Temp_1$*$Temp_2$	4	8735544	2183886
$Temp_1$*Concentration	4	15635285	3908821
$Temp_1$*Time	4	20043558	5010890
$Temp_2$*Concentration	4	40458310	10114578
$Temp_2$*Time	4	9734416	2433604
Concentration*Time	4	94897016	23724254
3-Way Interactions	32	2.79E+08	8714303
$Temp_1$*$Temp_2$*Concentration	8	48979158	6122395
$Temp_1$*$Temp_2$*Time	8	60081532	7510191
$Temp_1$*Concentration*Time	8	74161167	9270146
$Temp_2$*Concentration*Time	8	95635833	11954479
4-Way Interactions	16	327000000	20431583
$Temp_1$*$Temp_2$*Concentration*Time	16	327000000	20431583
Error	0	*	*
Total	80	921000000	

Note: Adj SS is the adjusted sum of squares for the model; Adj MS is the square of the adjusted means.

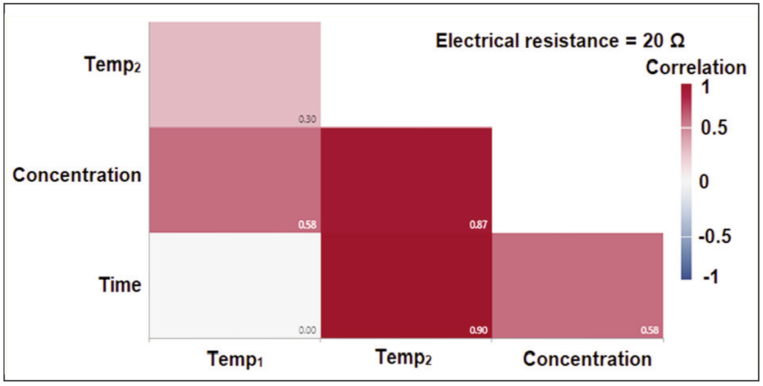

Fig. 1. Correlogram – time, $temp_1$, $temp_2$, concentration depending on electrical resistance (*Rs* = 20 Ω)

of degrees of freedom of the interaction between factor *i* and the *j* factor.

The sum of squares adjusted across the model ($Total_{Adj. SS}$) is presented after the variant analysis and represents the sum of all Adj SS values for all factors, interactions and errors.

RESULTS AND DISCUSSION

Using the full factorial plan responsive to electrical resistance (dependent variable) and applying optimization toward reducing the sensor's electrical surface resistance to 4 Ω or 10 Ω, we used the minimization function ($F(x) = y$, $y = 4$ Ω or 10 Ω) and obtained the results presented in table 12. Figure 2 presents the corresponding optimization graph and shows that the minimum value for electrical resistance (4 Ω) can be obtained if values of $temp_1$= 90°C, $temp_2$ = 150°C, concentration 20% and *time* = 5 minutes are used.

Using the full factorial plan including the response in electrical resistance (dependent variable) and applying optimization toward reducing the actuator's electrical surface resistance to 4 Ω or 10 Ω, we used the minimization function ($F(x) = y$, $y = 4$ Ω or 10 Ω) and obtained the results presented in table 13. Figure 3 presents the corresponding optimization graph and shows that the minimum value for electrical resistance (4 Ω) can be obtained if rotational *speed* = 250 rpm, *volume* = 250 µl, concentration 10% and spin coating time 60 seconds are used.

The optimized solution reduces the number of redundant experiments (e.g., in the case of the conductive textile for actuators, the full factorial plan involves 256 experiments with 4 independent variables, each having 4 levels of values; in the case of sensors, the full factorial plan involves 81 experiments with 4 independent variables each having 3 levels of values), raw material consumption, total costs, and use of utilities (water, energy). However, it is recommended that the textile surface be hydrophobic to ensure a uniform distribution of the conductive dispersion during spin coating, despite that some inconvenience can occur when the textile surface is as flat as a wafer. For example, if the textile surface is hydrophilic and presents polar groups, then the dispersion can be absorbed very quickly before the time allocated for spin coating has ended.

Table 12

OPTIMIZED SOLUTION FOR SENSORS EXPERIMENTS

No.	Temp$_1$	Temp$_2$	Concentration	Time	Resistance fit	Composite desiderability
1	90	150	20	5	4	1
2	90	160	10	5	10	0.9994
3	90	170	90	5	10	0.9994
4	90	170	20	15	10	0.9994
5	90	160	90	10	10	0.9994
6	90	170	90	15	10	0.9994
7	80	160	10	10	10	0.9994
8	90	150	10	5	10	0.9994
9	80	160	20	15	10	0.9994
10	70	150	20	15	10	0.9994

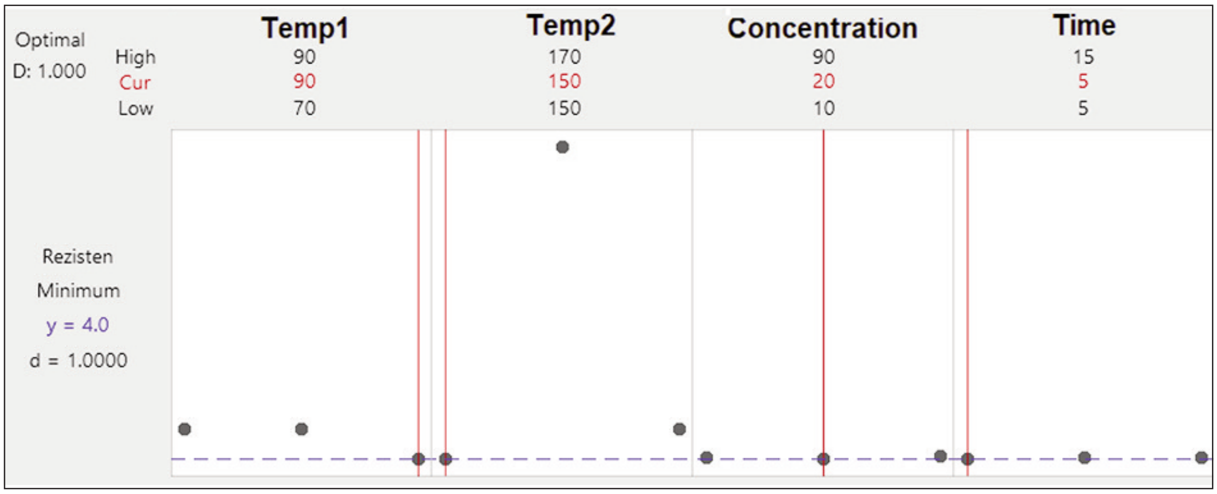

Fig. 2. Optimization graph for the sensor experiments

Table 13

OPTIMIZED SOLUTION FOR ACTUATORS EXPERIMENTS

No.	Speed	Volume	Concentration	Time	Resistance fit	Composite desiderability
1	250	250	10	60	4	1
2	1000	100	20	30	4	1
3	1000	100	10	10	4	1
4	250	100	40	30	4	1
5	1000	500	40	10	10	0.9994
6	250	500	30	30	10	0.9994
7	1000	250	30	30	10	0.9994
8	250	1000	40	60	10	0.9994
9	500	1000	40	10	10	0.9994
10	750	1000	40	30	10	0.9994

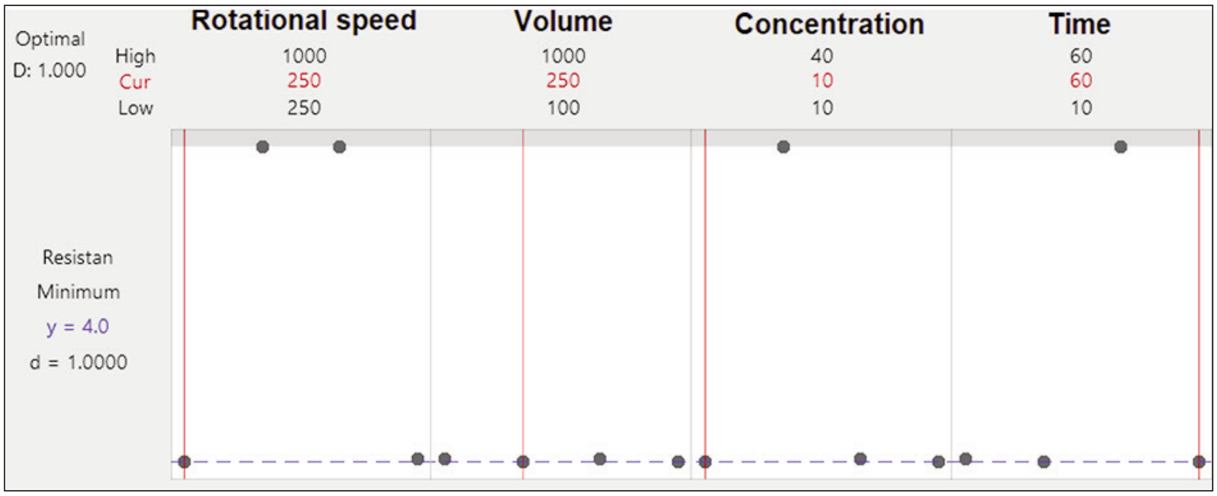

Fig. 3. Optimization graph for actuator experiments

CONCLUSIONS

In conclusion, the principal component analysis may help reduce redundant data, establish the correlations and covariance between variables that are necessary for the prediction of such variable dependencies, discover which variables are latent and establish the correlation and covariance matrix. However, principal component analysis and full factorial design are rarely mentioned in articles considering the development of textile actuators and sensors.

Here, the use of a full factorial experimental design allows us to optimize the number of experiments and to reduce a huge experimental plan (i.e., 256 or 81 experiments), and the correspondingly large consumption of numerous resources (raw materials, chemicals, water, energy), too few experiments (10 experiments) and minimal resources. With these methods, we more quickly obtain the targeted electroconductive fabric to be used as an electrode for sensors or actuators.

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