Using Design of Experiments and Virtual Instrumentation to Evaluate the Tribocharging of Pulverulent Materials in Compressed-Air Devices

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***Abstract*—Tribocharging of pulverulent materials in compressed-air devices is a typical multifactorial process. Quantification of the effects of the factors and of their interactions is a prerequisite for the development of new tribocharging devices for industrial applications. This paper aims at demonstrating the interest of using the design of experiments methodology in association with virtual instrumentation for the study of such processes, in view of their modeling and optimization. A classical 2^3 full-factorial design followed by a composite design were employed for conducting experiments simulating the tribocharging conditions of starch powder. The response function was the charge/mass ratio of the material collected in a modified Faraday cage, at the exit of the tribocharging device, the factors under investigation being the injection pressure, the dillution pressure, and the vortex pressure. The charge measurements were performed using a digital electrometer connected to a personal computer equiped with a data acquisition system. The data were processed by a custom-designed LabView virtual instrument. By using appropriate design of experiments software, it was possible to estimate the effects of these factors and then derive the model of the process as a quadratic polynomial function. This model served at predicting the optimal set point of the process. The results reported in this paper recommend design of experiments methodology as an efficent tool for the optimization of industrial tribocharging devices that might be used in conjunction with electrostatic separators for the selective sorting of various insulating materials contained in mixed pulverulent materials.**

***Index Terms*—Applied electrostatics, design of experiments, electric charge, tribocharging, virtual instrumentation.**

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I. Introduction

In spite of intensive theoretical and experimental research, tribocharging remains one of the less understood and most difficultly controlled electrostatic phenomena [1], [2]. The amount of static electricity generated by tribocharging depends not only on the nature of the materials involved (work function, dielectric constant, electrical conductivity, etc.), the type (sliding, impact, etc.), the duration, and the force of contact, but also on the state of the surfaces (roughness, contaminants, etc.) and the ambient conditions (relative humidity, temperature, etc.). The list of the factors influencing the tribocharging of pulverulent materials also includes the size distribution and shape of the particles.

Neither the few available theoretical models [3], [4] nor the plethora of empirical data [5], [6] can be of great help in the development of a new process based on the use of tribocharging phenomena. Each case is characterized by a specific combination of potentially influential factors, and the only way of evaluating their effects is experimentation. The problem is that the classical experimental method, consisting in measuring the response y for several values of each variable x_i, while fixing the values of the $(n-1)$ other variables, can be prohibitive in terms of both time and costs. In the case of four variables and five experimental values to each of them, the investigator has to carry out $5^4 = 625$ experiments.

Using the design of experiments (DOE) methodology is the only way to reduce the number of experiments to be carried out without influencing the quality of the results [7], [8]. The great innovation of this methodology is that it proposes *a factorial experimentation*, in which the factors investigated vary simultaneously. Simple mathematical processing of DOE data enables a rather accurate evaluation of factor effects and interactions. DOE can also be used for determining the relationship f between the factors x_i, $i = 1, \ldots, n$ affecting a process and the output of that process y. The function f can then be employed for predicting the optimal operating conditions of the process.

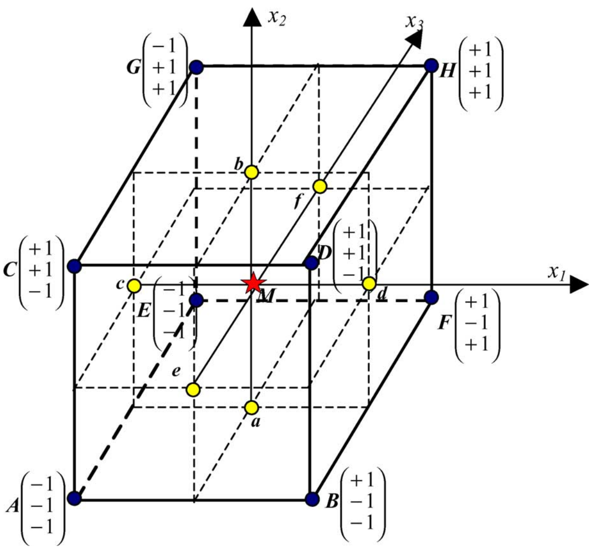

Fig. 1. Representation of the 17 experimental points of the composite design.

Recently, DOE has been successfully employed for optimizing the operation of electrostatic separators [9], [10]. Recommendations could be formulated for the robust design of such processes [11].

The aim of the present paper is to demonstrate that DOE can be an effective tool for modeling and optimization of a class of tribocharging processes. The main difficulty to surpass is the accurate characterization of process output: the charge/mass ratio of processed materials. One solution might be the use of virtual instrumentation [12].

The study is focused on the tribocharging of starch powder in compressed-air devices similar to those employed in electrostatic painting. Nevertheless, its conclusions could be of interest for a wide range of industrial applications, including the tribo-electrostatic separation of powder mixtures in mining or food industry.

II. Design of Experiments Methodology

When the objective is screening of the various factors that might influence the outcome of a process, DOE recommends the use of two-level full-factorial designs [7], [13]. In this kind of experiment, each factor at each level is evaluated at an equal number of other factor-level combinations. In the case of three factors (x_1, x_2, x_3), the experiment is conducted in accordance with the so-called 2^3 factorial design (points $A \ldots H$ in Fig. 1). The mathematical model derived from such a design is a first-order polynomial function y that usually takes into consideration all the two-factor interactions

$$y = f(x_i) = a_0 + \sum a_i x_i + \sum a_{i,j} x_i x_j, \quad i = 1 \div 3, \quad j = 1 \div 3, \quad i \neq j. \tag{1}$$

The mathematical model contains $p = 7$ coefficients, which, in the most simple case of eight experiments in points $A \ldots H$, are calculated as follows:

$$a_0 = [y(A) + y(B) + \ldots + y(H)]/8 \tag{2}$$

$$a_i = [y(A)x_i(A) + y(B)x_i(B) + \ldots + y(H)x_i(H)]/8, \quad \text{for } i = 1 \div 3 \tag{3}$$

$$a_{i,j} = [y(A)x_i(A)x_j(A) + y(B)x_i(B)x_j(B) + \ldots + y(H)x_i(H)x_j(H)]/8, \quad \text{for } i = 1 \div 3, \quad j = 1 \div 3, \quad i \neq j. \tag{4}$$

A straightforward way to validate this linear-interaction model is to calculate the value of the response y in the central point of the experimental domain (point M in Fig. 1), and compare it to the experimental value measured in the same point.

If computed and measured values differ significantly, the first-order model cannot be validated, and a quadratic model should be adopted

$$y = f(x_i) = a_0 + \sum a_i x_i + \sum a_{ii} x_i^2 + \sum a_{i,j} x_i x_j, \quad i = 1 \div 3, \quad j = 1 \div 3, \quad i \neq j. \tag{5}$$

In order to obtain such a quadratic model, the 2^3 factorial design with center points is augmented with a group of "star points" ($a \ldots f$ in Fig. 1). Such a composite design was adopted for the present study.

The statistical significance of the coefficients a_i and $a_{i,j}$ can be evaluated by calculating the *residues* e_i, i.e., the difference between the experimental value and the one predicted by the model, and estimating the variance

$$s^2 = \frac{1}{n-p} \sum e_i^2 \tag{6}$$

where n is the number of experiments and p the number of coefficients of the model. A coefficient a_i of the model is statistically significant if it satisfies the Student's *t* test

$$t_i = \frac{|a_i|}{s_i} > t_{\text{crit}} \tag{7}$$

with t_{crit} given in tables as a function of the degrees of freedom $(n - p)$, and

$$s_i^2 = \frac{s^2}{n}. \tag{8}$$

In the present study, an in-house design of experiments software was employed for validating the models using the Fischer's test [8] for evaluating their predictive ability.

III. Experimental Procedure

The experimental setup consists of a tribocharging device [14] provided with means to control the charge by adjusting the air pressure in different circuits (Fig. 2). The injection pressure p_{inj} determines the particle speed through the tribogun and the energy of particle-wall impacts; the dilution pressure p_{dil} is used to modify the concentration of powder in the transported air, and hence, the number of particle-wall contacts; the vortex pressure p_{vor} controls the turbulence of the motion.

The limits of the experimental domain were established based on the following constraints: $p_{\text{inj}} \leq 2.8$ bar, $p_{\text{inj}} > p_{\text{dil}} > p_{\text{vor}}$. Thus: 2.2 bar $\leq p_{\text{inj}} \leq 2.8$ bar; 1.4 bar $\leq p_{\text{dil}} \leq 2$; 0.8 bar

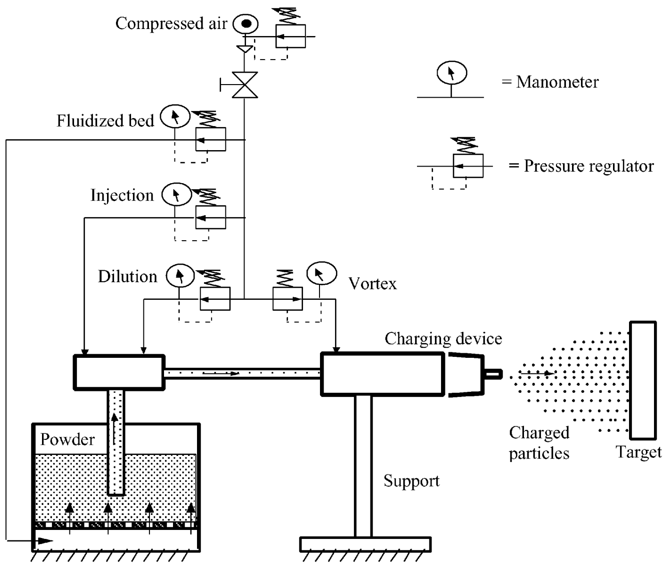

Fig. 2. Schematic representation of the powder tribocharging installation.

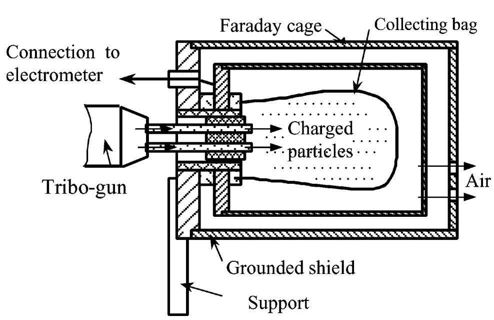

Fig. 3. Custom-designed Faraday pail for powder charge measurements.

$\leq p_{\text{inj}} \leq 1.2$ bar. The ambient temperature was $19.5 \pm 0.5°$C, while the relative humidity of ambient air was $37.5 \pm 1.5\%$.

The charged powder (starch, characteristic size: 0.02 mm) was collected in a modified Faraday pail (Fig. 3), connected to an electrometer (model 6514, Keithley Instruments).

The charge measurement data acquired during up to 30 s of steady-state operation of the tribogun were processed by a virtual instrument (VI) developed in the LabView environment [15]. The VI commanded the grounding of the electrometer input every 3 s. In this way, it displayed the charge accumulated in the modified Faraday pail within 3 s intervals (Fig. 4). These data were then employed for the calculation of the charge/mass ratio.

The two-step experimental procedure consisted of a 2^3 full-factorial design, aimed at screening the effects of the three factors under study, followed by a star design. The resultant composite design made possible the modeling and the optimization, using the MODDE 5.0 software [13].

IV. Results and Discussion

A. The Linear-Interaction Model

The results of 2×8 runs of the 2^3 factorial design experiment are given in the first 16 lines of Table I. The coefficients

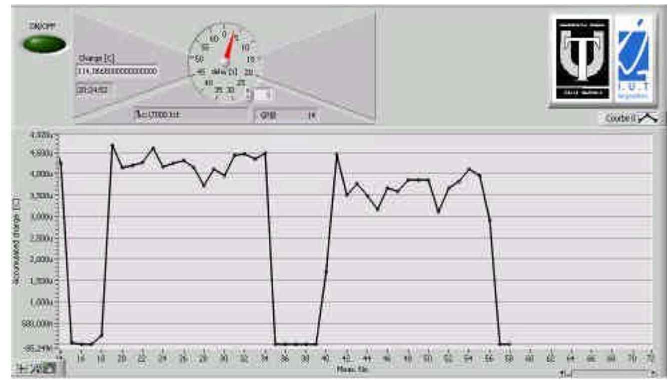

Fig. 4. Charge measurement chart displayed by the custom-designed VI for two points of the experimental design.

TABLE I
RESULTS OF THE COMPOSITE FACTORIAL DESIGN

Run n°.	Factors			Results			REPONSE
	p_{inj} [bar]	p_{dil} [bar]	p_{vox} [bar]	Q [μC]	M [g]	Q/M [μC/g]	y [μC/g]
1(*A*)	2.2	1.4	0.8	25.1	81.1	0.310	0.313
2(*A*)	2.2	1.4	0.8	30.8	97.8	0.315	
3(*B*)	2.8	1.4	0.8	38.9	90.9	0.427	0.425
4(*B*)	2.8	1.4	0.8	44.5	105.5	0.422	
5(*C*)	2.2	2.0	0.8	30.5	79	0.386	0.386
6(*C*)	2.2	2.0	0.8	33.9	87.9	0.386	
7(*D*)	2.8	2.0	0.8	45.2	121.5	0.372	0.375
8(*D*)	2.8	2.0	0.8	46.0	121.8	0.378	
9(*E*)	2.2	1.4	1.2	39.6	95.1	0.416	0.418
10(*E*)	2.2	1.4	1.2	36.2	86.5	0.419	
11(*F*)	2.8	1.4	1.2	59.1	104.9	0.563	0.564
12(*F*)	2.8	1.4	1.2	65.6	116.4	0.564	
13(*G*)	2.2	2.0	1.2	37.0	69.7	0.531	0.533
14(*G*)	2.2	2.0	1.2	49.1	91.9	0.534	
15(*H*)	2.8	2.0	1.2	63.9	126.8	0.504	0.502
16(*H*)	2.8	2.0	1.2	62.9	126.2	0.499	
17(*a*)	2.2	1.7	1.0	42.5	77.5	0.549	0.499
18(*a*)	2.2	1.7	1.0	29.8	66.4	0.448	
19(*b*)	2.8	1.7	1.0	38.3	74.5	0.514	0.513
20(*b*)	2.8	1.7	1.0	43.6	85.1	0.512	
21(*c*)	2.5	1.4	1.0	38.6	70.7	0.546	0.548
22(*c*)	2.5	1.4	1.0	37.4	68.2	0.549	
23(*d*)	2.5	2.0	1.0	32.0	41.9	0.763	0.65
24(*d*)	2.5	2.0	1.0	41.4	75	0.552	
25(*e*)	2.5	1.7	0.8	39.1	83.1	0.470	0.473
26(*e*)	2.5	1.7	0.8	35.1	73.9	0.475	
27(*f*)	2.5	1.7	1.2	46.7	75.5	0.619	0.613
28(*f*)	2.5	1.7	1.2	41.2	68	0.606	
29(*M*)	2.5	1.7	1.0	51.1	102.3	0.500	0.502
30(*M*)	2.5	1.7	1.0	62.3	123.5	0.504	
31(*M*)	2.5	1.7	1.0	49.5	97.6	0.507	0.506
32(*M*)	2.5	1.7	1.0	49.9	99	0.504	
33(*M*)	2.5	1.7	1.0	38.3	76.2	0.502	0.505
34(*M*)	2.5	1.7	1.0	54.9	108.3	0.507	

of the linear-interaction model were computed with (2)–(4), with $y(A), y(B), \ldots y(H)$ being the average values of the response in the eight points of the experimental design. Thus, the charge/mass ratio y can be expressed by the following polynomial function:

$$y = 0.439 + 0.027p_{\text{inj}} + 0.010p_{\text{dil}} + 0.065p_{\text{vox}} - 0.038p_{\text{inj}}p_{\text{dil}} + 0.002p_{\text{inj}}p_{\text{vox}} + 0.004p_{\text{dil}}p_{\text{vox}}. \quad (9)$$

As expected, the charge/mass ratio y increases with p_{inj}, p_{dil}, and p_{vor}. Though the coefficient of p_{dil} is smaller than the others, the influence of this variable upon the output y cannot be

TABLE II
EVALUATION OF THE EFFECTS USING STUDENT'S t TEST

Variable	Coefficient	t_i	Significance
Constant	$a_0 = 0.532$	t_0=137.65 > 2.064	Significant
p_{inj}	$a_1 = 0.028$	t_1=7.122 > 2.064	Significant
p_{dil}	$a_2 = 0.008$	t_2= 2.068 > 2.064	Significant
p_{vor}	$a_3 = 0.066$	t_3=17.048 > 2.064	Significant
$p_{inj} * p_{inj}$	a_{11} = -0.071	t_4=18.25 > 2.064	Significant
$p_{dil} * p_{dil}$	a_{22} = -0.005	t_5=1.251 < 2.064	Non Significant
$p_{vox} * p_{vor}$	a_{33} = -0.013	t_6=3.254 > 2.064	Significant
$p_{inj} * p_{dil}$	I_{12} = -0.038	t_7=9.694 >2.064	Significant
$p_{inj} * p_{vor}$	I_{13} = 0.002	t_8=0.452 < 2.064	Non Significant
$p_{dil} * p_{vor}$	I_{23} = 0.004	t_9=0.937 < 2.064	Non Significant

neglected, as it has a strong interaction with p_{inj}, as shown by the respective coefficient in (9).

The charge/mass ratio in the center of the experimental domain predicted by the linear-interaction model is 0.439 μC/g. This value is significantly different from the average of the experimental values measured for the same point: 0.504 μC/g. This justifies the need for a composite design, so as to enable the derivation of a quadratic model.

B. The Quadratic Model

In addition to the 2×8 experiments $A \ldots H$ of the factorial design, 2×6 other experiments were carried out, corresponding to the six points $a \ldots f$ forming a star in Fig. 1, as well as 2×3 experiments in the central point M. The results of the 34 runs carried out according to this composite experimental design are presented in Table I. The statistical significance of the coefficients of the quadratic model

$$\begin{aligned} y = 0.532 &+ 0.028p_{\mathrm{inj}} + 0.008p_{\mathrm{dil}} + 0.066p_{\mathrm{vor}} \\ &- 0.071p_{\mathrm{inj}}p_{\mathrm{inj}} - 0.005p_{\mathrm{dil}}p_{\mathrm{dil}} - 0.013p_{\mathrm{vor}}p_{\mathrm{vor}} \\ &- 0.038p_{\mathrm{inj}}p_{\mathrm{dil}} - 0.002p_{\mathrm{inj}}p_{\mathrm{vor}} - 0.004p_{\mathrm{vor}}p_{\mathrm{dil}} \end{aligned} \quad (10)$$

was evaluated with the Student's t test, using (6)–(8), for $n = 34$, and $p = 10$ (Table II). According to this test, the interaction between the vortex and dilution pressures, and the interaction between the injection and vortex pressures are not significant.

The analysis of variance using the Fischer's test [8] validated the quadratic model: $F_{\mathrm{obs}} = 39.238 > F_{\mathrm{cr}} = 2.3$. Two statistical parameters were evaluated, using the standard procedure described in [13, p. 237]: the goodness of fit $R^2 = 0.9364$, which is a measure of how well the regression model can fit the raw data, and the goodness of prediction $Q^2 = 0.8934$. The predictive ability of the model can be further improved by removing the nonsignificant coefficients. In that case, R^2 slightly diminishes to 0.9344, but Q^2 increases to 0.9046, which is an excellent result.

The aspect of the predicted response curves, computed with (10) and represented in Fig. 5, indicates that the optimum operation is attained at $p_{\mathrm{inj}} \approx 2.5$ bar, $p_{\mathrm{dil}} \approx 2$ bar, and $p_{\mathrm{vor}} \approx 1.2$. These values are confirmed by computations performed with MODDE 5.0 software [13] (Fig. 6).

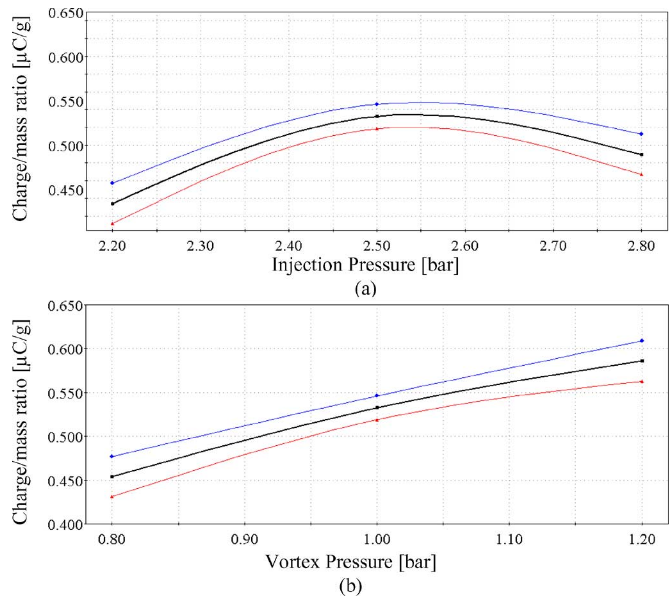

Fig. 5. Plot of predicted response and the 95% confidence interval, as function of the injection pressure, for (a) $p_{\mathrm{dil}} = 1.7$ bar and $p_{\mathrm{vor}} = 1$ bar, and as a function of the vortex pressure, for (b) $p_{\mathrm{inj}} = 2.5$ bar and $p_{\mathrm{dil}} = 1.7$ bar.

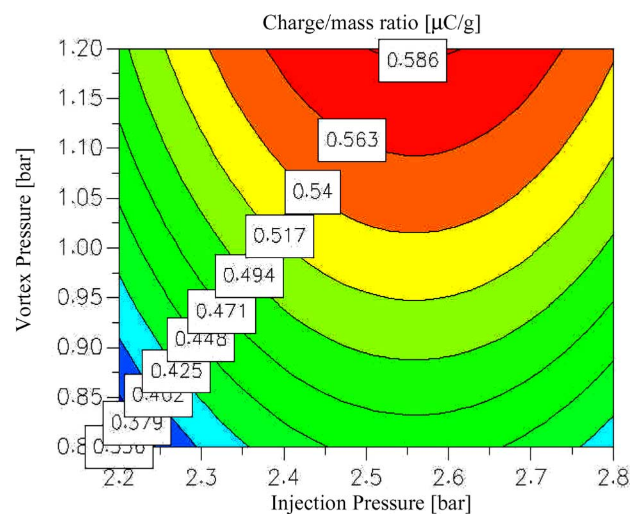

Fig. 6. Contour plot of the response predicted by the quadratic model: variation of the charge/mass ratio when the injection and vortex pressures vary over the range defined by the experimental design, at $p_{\mathrm{dil}} = 1.7$ bar.

V. CONCLUSION

Tribocharging of insulating powders in compressed-air devices is a multiple-factor process that can be effectively modeled and optimized by using the DOE methodology.

Of the three factors considered in this study, the vortex pressure p_{vor} is the most important, as indicated by the coefficients of the quadratic model. Indeed, by enhancing the turbulance of particle movement through the tribocharging device, the incresed vortex pressure p_{vor} multiplies the number of particle-wall collisions, and hence, the charge imparted to the processed powders. As the coefficients of p_{dil} and p_{dil} are smaller than the others in the quadratic model of the process, it might be concluded that the dillution pressure p_{dil} affects to a less extent the charge/mass ratio at the output of the tribocharging device. However, a strong interraction exists between the injection and the dillution pressures. Therefore, the optimum operation of the device imposes a rigorous control of all three input variables.

The experimental procedure described in the paper enabled the prediction of the optimum operating conditions. A step further in the application of the DOE methodology would be the study of the robustness of this optimum for small changes in the values of the control variables. An adddional challenge for further research would be the robust design of the tribocharging process, which is known to be highly sensitive to any alteration in powder hygrometry and in the environmental conditions.

The availability of user-friendly software tools simplifies the task of the investigator, who is no longer constrained in the application of this methodology by the complexity of statistical analysis of the experimental data. With virtual instruments facilitating data acquisition and processing, DOE is likely to be employed on a wider scale for the optimization of various electrostatic processes that make use of tribocharging phenomena, such as the separation of mixed powders in mining or food industry.

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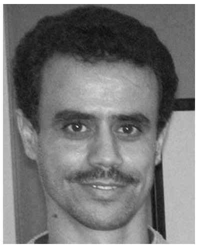

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