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DESIGN OF EXPERIMENTS IN PLANNING METALLURGICAL TESTS

50

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CNPq CETEM

GOVERNO DO BRASIL

DESIGN OF EXPERIMENTS IN PLANNING METALLURGICAL TESTS

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SUMMARY:

RESUMO

Este trabalho apresenta algumas noções básicas do planejamento estatístico de experimentos e faz uma revisão da aplicação desta técnica à metalurgia, com as referências respectivas.

ABSTRACT

The experimental design of experiments, as applied to metallurgy, is discussed and examples of applications are presented.

1. INTRODUCTION

Researchers are supposedly paid to undertake measurements and to extract the significant data out of those measurements. From these, conclusions are taken and decisions must be made.

However, one thing is to be paid for and another very different task is to come to the decision; this is so due to the very fact that decisions involve risks and unless you have a strong feeling that whatever decision you are coming to is the right one, you are not going to endanger your prestige !

But, hard life, you must to! Decisions must be taken and, in fact, need to, at every moment of our life.

The parameters affecting a decision are not always straight forward; in fact, they are never so. Experience and common sense are always brought to the scene of a decision in such a way that risks are minimized (or, at least, thought to be !).

Notwithstanding, common sense, again, is very difficult to rank. What seems to be common sense for one individual, may not be so to another.

Let us take the notion of measurement: this has been taken from common sense and largely utilized by science and engineering and it has been defined[1] as the assignment of numbers to represent properties according to arbitrary rules which are based on scientific laws.

In order to measure something two consecutive steps are to be taken:

a. sampling and subsampling some aggregate of material;

b. measuring physical and/or chemical properties on individuals taken from the samples.

Since both a. and b. may produce variations, these cumulate into an overall variation in the final result.

Assigning a measure to a property is an operation which when under control, associates each value of the property to a corresponding value of the measured value.

Controlling such an operation requires a knowledge of the environmental conditions under which the property is being measured.

It has been shown[2] that the values of a measurement for a particular value of a property can be partitioned into subpopulations, each corresponding to situations in which environmental conditions are under different states of local control.

What does significant data means ?

Measurements, as discussed, are vital to assess values for a given property. However, are these measurements significant ?

It is possible to postulate[3] four possible components of variance which cumulate into the variance that is needed to assess a statistical significance to the measured data:

$$ST_T^2 = S_S^2 + S_L^2 + S_{D(L)}^2 + S_{R(D)}^2$$

where:

ST_T^2 = is the variance of the acceptance plan;

S_S^2 = is the variance of the aggregate of material;

S_L^2 = is the variance of laboratories (or factories);

$S_{D(L)}^2$ = is the variance of tests made on different days within laboratories (or factories);

$S_{R(D)}^2$ = is the variance of tests replicated within days and laboratories (or factories).

All these variances represent random variations that are encountered when materials are properly sampled and tested in laboratories (factories) everywhere.

The comparison between accepted variances is what makes a measure significant, that is, significant is a value that does not arise by mere chance !

2. DESIGNING EXPERIMENTS

In the laboratory, pilot-plant or industry, engineers and researchers seek relationships among inputs and outputs. These relationships, once established, will help to run the process better and in a more comprehensive way.

In the design department[4], engineers analyse performance, cost and safety data to help them make decisions about a new plant. In the administration office, engineers look at trends in suply, demand, price and cost to decide how to run the business.

Designing industrial experiments is a quite different task than designing scientific and agricultural experiments. This, of course, does not mean that the statistical schemes applied to scientific and agricultural experiments might not be used, as well, to industrial experiments. In fact they are so.

It means, however, that the environmental constraints related to industry, agriculture and science are, by their own natures, different, i.e., the speed in which the several experimental units (treatments) respond to the influence of the acting variables (factors) in agriculture is slower than that of the industry. On the other hand the degree of control (cleaniness, for instance) of scientific experiments is greater than that of industrial and agricultural experiments.

An industrial environment offers a considerable variation in many conditions of the industrial experiment: raw materials varying in quality, operators varying from shift to shift, undesirable peaks of energy inputs, machinery performance variations throughout the working shift, etc.

Experimental designs in such instances enables to separate uncontrolled variations from the real variations due to the affecting variables. Minimizing the systematic errors influence upon the response is accomplished applying the techniques of **randomization** and **blocking**.

Blocking, plus replication of the treatments enhances **precision**, the statistically α error. The **accuracy** of a measurement, the β error, is linked to the α error (precision) and the size (N) of the taken sample. This linkage is statistically shown by the **power curves**.

Student, or **t - test**, Fischer, or **F - test**, are powerful tests, and largely applied in analysing data coming from the experiment, assuming the Central-Limit Theorem underlies such a data.

However, randomization, as fully discussed by Youden[5], may involve, in industry, awkward additional operations and although *'often specified as an indispensable requirement in experimental design, it is required only when the order or position of the experimental unit influences the performance of the unit'*.

Full replication of the experiment, as well, may be uneconomical, and techniques of fraccionizing it are available.

Excellent introductory accounts of experimental designs are presented in articles such those of Smallwood[6], Szonyi[7], Hahn[8] and Hendrix[9], as well in books as Duckworth[10] and Deming&Morgan[11] among several others, not to mention the classical text on the subject, Davies[12]. An annotated bibliography up to 1980 was tabulated by Hahn[13].

Several designs are available to the metallurgist or chemical metallurgist and to choose of one specific design against any other is a matter of what the researcher is looking for. It is worth to emphasize that the problems related to designs are inseparable from those of their analysis. In fact a given chosen sample scheme implies a certain technique of statistical analysis.

Designing and analysing an experimental plan are the essence of any research undertake to establish influences and effects of a set of variables on chosen responses. However, as stated by Box & Hunter[14]: *'Of the two, design and analysis, the former is undoubtetly of greater importance. The damage of poor design is irreparable; no matter how ingenious the analysis, little information can be salvaged from poorly planned data. On the other hand, if the design is sound, then even quick and dirty methods of analysis can yield a great deal of information.'*.

Designs to scan variables (factors) may be found in the literature: Placket-Burmann[15], Latin-Squares[16], Greco-Latin-Squares[17] are used depending upon the envisaged target. These designs are mostly utilized when the informations regarding the single variables are the main objectives, and no **interactions** (effects due to the interplay between variables) are sought.

Other fraccional factorials[18] are utilized when the main factor effects as well some of the lower order interactions (two or three variables effects) are sought.

Largelly used are factorial designs, specially the two level factorial designs.

Factorial designs may be **symetrical** (when the number of the levels is the same for all variables) and **assymetrical** (when otherwise). A presentation of the matematics involving factorial designs was given by Bose & Srivastava[19].

Classes of factorial designs that are very useful are those employing two and three levels for each of the variables, either **complete** or **incomplete** (fractional). A quite useful class of three level designs for the study of quantitative variables was developed by Box

& Behnken[20].

Latin Cubes and Hyper-Greco-Latin-Cubes[21] have been used extensively in the search for compositions of new multicomponent material and the development of technologies for their production.

Response Surface Designs are special multivariable designs for quantitative independent variables (temperature, pressure, etc.). Prediction equations are developed using the least-square approach[22].

3. STATISTICAL DESIGN OF EXPERIMENTS IN METALLURGY

Several examples of applications of statistically designed experiments in metallurgical testing are available in the literature.

Although this section does not intend to give a comprehensive survey of the subject, its purpose, however, is to present some examples of applications to guide the interested reader.

Palazzi[23] presented an account of the employment of statistical methods in industrial and metallurgical research. As well, Palazzi[24] discussed a four variables incomplete factorial on the evaluation of combined carbon as affected by metallurgical variables; Hamaker[25] presented an experiment involving the investigation of electrophoretic deposition of aluminium oxide on nickel rods (3 x 5); Hornak&Wittenberger[26] provided a study of several dessulfuring agents running a 2^4 singly replicated factorial experiment, further commented by Cuthbert[27]; Duckworth[28] conducted a series of lectures at the British Iron & Steel Research Association which were edited in a book form, presenting exercises and examples applied to metallurgical studies; Boyard[29] discussed the 2^4 factorial design applied to the flotation of lead and zinc ores from Touissit, France, and further investigated a 2^{7-1} factorial[30]; Dorenfeld[31] concentrated a copper porphyry ore through a five variable factorial design; Stone et al.[32] presented a response surface design on the extraction of silica from quartz by digestion in sodium hydroxide solutions; Plaksin et al.[33] had determined the optimum flotation conditions of a copper-lead flotation tailings utilizing a technique exposed by Box&Wilson[34]; Balberyszski&Villas Bôas[35] applied a quarter replicate of a 2^7 factorial to study the effect of organic additives on zinc electrowinning; Honeywell[36] developed a Plackett-Burman experimental plan to study the effects of six variables on the column flotation behaviour of uranium ores. Colombo[37] presented the effect of tapioca flour on the anionic flotation of gangue from iron ores, through a response surface methodology; Spironello[38] discussed the variables affecting electric-furnace smelting applying 2^3 and 2^{7-4} factorial plans; Granovski[39] investigated a hydrometallurgical route separate scandium from titanium, via selective sorption from sulphuric acid solutions, applying a 2^{4-1} fractional factorial and optimization procedures (step ascendend); Orofino Pinto&Villas Bôas[40] described the direct leaching of chromite ores applying a 2^4 factorial design; Granato&Villas Bôas[41] utilized a 2^4 full factorial plan to verify the effects of operational variables on gold electrorefining.

Hopkins et al.[42] described two experimental plans, the basis of both being a 2^{5-1} fractional factorial design; the first design was a central composite design, including the 16 runs and 10 axial runs; the second plan was constructed from the 2^{5-1} design and 10 centrepoint, when studying the critical factors influencing uranium precipitation by hydrogen peroxide.

For those whose objective is to study the behaviour of **mixtures**, where the sought answer is a function of the relative proportions of the constituent components and not of the total amount of the mixture *per se*, examples are found in the litterature, although not necessarily directly linked to metallurgical mixtures. Notwithstanding the ideas, conception and propositions do apply of course, for everyday problems regarding mixtures in the metallurgical industries.

Thus, Snee & Marquardt[43] present Simplex Screening Designs for mixtures studies; also Drader & Lawrence [44] [45] report mixture designs for three factors and four factors, and Snee[46] presents techniques for the analysis of mixture data.

An study of fly ash-rubber mixtures, measuring the skid resistance and durability was presented by Capp & Makovsky[47], through a four factor, five level factorial design.

When modelling a complex process, a non-linear system situation normally arises, and in such cases a careful planning of the experiment is of utmost importance. For instance the kinetic model discrimination of the water-gas shift reaction over iron-oxide catalysts as presented by Kim[48].

Of great interest for those interested in getting into the basic mechanisms of the system under study are the papers by Box[49], Box & Youle[50] and the already mentioned Box & Hunter[14].

Chemical analysis are a part of metallurgical testing procedure. Therefore, for those concerned to such a problem, say need to assure water quality of effluents in metallurgical plants, Wilson[51] presents an approach to ensure results from several laboratories of adequate accuracy; as well, when the economics of chemical analysis is what concerns, in establishing the costs of routine analytical tests as weighed against losses due to shipping material, the paper by Davies[52] is a necessary reference.

Evolutionary operation (E.V.O.P.) is very much used in process control and optimization on a continuous basis and designed to be operated in the presence of error. The Patiño Mining Corporation, at Chibougmau, developed in its copper concentrator and E.V.O.P. Program reported by Eggert[53].

Also related readings are Box[54], Coutie[55], Baasel[56] and Hahn[57].

4. FINAL REMARKS

It is hoped that this paper brought to the attention of the metallurgical engineer several examples of data aquisition and analysis, according to statistically planned designs.

Such techniques minimizes systematic errors, bring up to the researcher's attention the experimental error, and allow the interpretation of single variables effects as well as the interactions effects between these same variables, thus permitting the synergetic nature of the phenomena to be assessed.

As stated by Box & Bisgaard[58], *'Experimental design is not a new idea. It would be new, however, if management in this country were to embrace it as standard practice.'*. As well *'the simultaneous study of many factors and their interactions can provide design engineers with more data in far less time than conventional experimental methods. Consequently, experimental design has the potential to accelerate research and the generation of improved products.'*.

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48 - Caracterização tecnológica de caulim para à indústria de papel;

49 - Amostragem de Minérios.

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