Tuning of an empirical mathematical model for particle size classification using hydroclassification

Pedro Miguel Semeano Ferreira

Instituto Superior Técnico, Universidade de Lisboa, 2019

Abstract

Particle size classification is a subject with many years of study. Since mid-20th century and with the growth of mining industry, classifiers became a theme with great interest of research and development.

In an industrial environment it's difficult to make experiments, especially because of the demands of production but also due to variations of operation conditions. Simulations gains here special importance because it allows to predict responses without being experimented. Unless there is a small simulation plant to make the experiments, they must be performed by laboratorial kits.

The aim is to demonstrate the entire process, from laboratorial tests to data processing, statistical analysis and the creation of mathematical empirical relationships for the main parameters of evaluating the performance of a hydrocyclone, such as water recovery in underflow, cut size, parameters of perfection and operational conditions.

Although the model needs a larger data base for the water recovery in underflow and for the cut size, the multiple correlation between the experimental data and the estimated values was 0.72. The differentiating factor in the results was the diameter relationship between vortex finder and apex, especially in the d_{50c}. The water recovery in the underflow, with values around 50%, it's influenced in the pulp feed pressure.

1. Introduction

After research on the previous work about the subject, it was found that the most used empirical models are still the Lynch and Rao model (1975), Plitt model (1976, 1980), and more recently the Nageswararao model (1995) with application on the *JKTech* software. It was also found some models that try to describe a phenomenon that appears in this paper and it is very common in literature - the fish hook effect - which is related with a higher recovery in the small calibers part of the partition curve. However, they are not widely used because the results are dubious.

These models are built with a significant database of experiments and have a lot of variables, which sometimes are difficult to measure, like viscosity. They are widely used for scale-up and simulation, although the databases didn't have hydrocyclones with small diameter <2", so it's applicability in small hydrocyclones it's limited.

This project aims to provide a full methodology for plan and designing of experiments and data analysis.

The methodology followed for plan and designing of experiments was the Response Surface Methodology (RSM) which can be found in Montgomery, 2009.

The data analysis started with mass balance and the formulation of a non-linear problem which allows to achieve consistent data, in terms of recovery of solids in underflow and size distribution of products. Simultaneously, partition curves were adjust and the parameters of partition functions were define to assess the performance of the hydrocyclone kit in each laboratory test, as well as after all the tests being completed.

The next phase was to perform a correlation analysis between the estimated parameters (dependent variables) and the operation conditions (independent variables).

Subsequent to the multiple regression analysis to create the empirical models, they are also subjected to validation, which was performed by the multiple determination coefficient calculated by the scatterplot between measured values and estimated values for the same parameter.

3. Metodology

As was mentioned before, the methodology followed was the RSM. This is a complex methodology for plan and designing of experiments. MSR is a collection of statistical and mathematical techniques useful to develop, improve and optimizing processes, new products and existing products. Its major applications are at industrial level, particularly in situations where there's plenty of variables that can potentially influence some measure of the process performance or quality. This performance measure is called the response. Input variables are called independent variables and are subject to the control of the engineer or scientist, at least in tests or experimental trials (Montgomery et al, 2009).

The application sequence of the MSR begins with the screening or phase zero, where first tests are made to inquiry which variables are most influential on the response surface and to eliminate those that are insignificant. In this work, this part was not performed and it was initially decided that the independent variables to be studied are: the injection pressure, the percentage of solids by weight of the feed pulp and the relationship between the vortex finder and apex diameters.

The following phase - phase one - contemplates the preliminary tests that are performed to determine the levels of the independent variables to be used, for example the injection pressure at 0.5, 1 and 1.5 bar, defining the extreme values of operability. Having defined the extreme values of the independent variables that were chosen, follows the construction of the test plan. The test plan has to contain the number of tests to be performed and the operating conditions of each test. The definition of the number of tests to be performed depends on the number of independent variables and the number of values to be used per variable.

$$N = L^{k} \qquad (1)$$

N represents the number of tests, L represents the number of levels per variable and k represents the number of variables.

3.1. Apparatus

In this work we used a hydrocycloning kit from *R.Mozley Ltd.*, which consists of an injection pressure gauge, hydrocyclone feed valve, bypass valve, single pump, a sump, the hydrocyclone and the overflow (Fig.1). For the two inch hydrocyclone that was used, two vortex finder with different diameters (14.3 and 11.1mm) were available, the apex was in solidarity with the hydrocyclone.

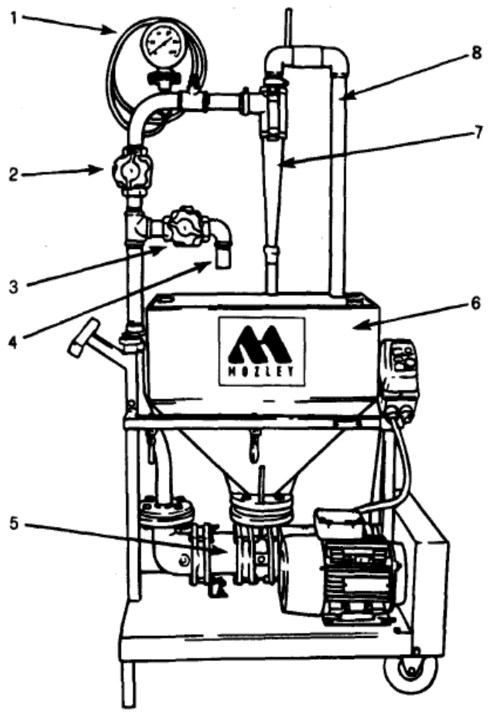

Figure 1-Hydrocycloning kit (1) pressure gauge (2) hydrocyclone supply valve (3) bypass valve (4) bypass outlet pipe (5) pump (6) sump (7) hydrocyclone (8) overflow pipe. Adapted from Vallebuona, 1995

3.2. Design of experiments

It was initially defined that the number of independent variables to be studied would be three: the injection pressure, percentage of solids by weight and the relationship between the vortex finder and apex diameters. Since the hydrocycloning kit that was used contained two vortex finders available with different diameters and the apex was not removable, it was only possible to make two combinations of diameter associations between the vortex finder and the apex. Thus it was decided that the other variables would also have two levels, one higher and one lower. Applying formula (1), the number of tests (N) is eight, where L, the number of levels per variable is two and k, the number of variables is 3.

To calculate the variance of the experimental error, it was defined that four further tests would be made in which the operating conditions were equal, in this case the injection pressures and the percentage of solids by weight with intermediate values but fixed between the maximum and minimum values of the first eight tests and the relationship between the vortex finder and apex diameters as no intermediate vortex finder was maintained with the minimum ratio.

Table 1 summarizes the operational conditions of the tests performed with a view to tuning the mathematical model.

Table 1-Table 1 - Test Plan, all dimensions are in millimeters

Test	**Dependent variables**				**Independent variables**				
	D_c	**D_i**	**h**	**D_u**	**D_o**	**D_o/D_u**	**ΔP (bar)**	**%Sp**	**D_F**
E1	50.8	32.9	300	9.4	14.3	1.52	0.5	16.67	5
E2	50.8	32.9	300	9.4	14.3	1.52	0.5	28.57	2.5
E3	50.8	32.9	300	9.4	14.3	1.52	1	16.67	5
E4	50.8	32.9	300	9.4	14.3	1.52	1	28.57	2.5
E5	50.8	32.9	300	9.4	11.1	1.18	0.5	16.67	5
E6	50.8	32.9	300	9.4	11.1	1.18	0.5	28.57	2.5
E7	50.8	32.9	300	9.4	11.1	1.18	1	16.67	5
E8	50.8	32.9	300	9.4	11.1	1.18	1	28.57	2.5
E9	50.8	32.9	300	9.4	11.1	1.18	0.7	23.08	3.3
E10	50.8	32.9	300	9.4	11.1	1.18	0.7	23.08	3.3
E11	50.8	32.9	300	9.4	11.1	1.18	0.7	23.08	3.3
E12	50.8	32.9	300	9.4	11.1	1.18	0.7	23.08	3.3

3.3. Material of test

The material used was a silica sand, with relative density equal to 2.6 g / cm3. The sample that was used to perform the tests had to be milled in a ball mill because it was too coarse to form the feed to a 2” diameter hydrocyclone.

A batch of maximum particle size after grinding was set up and served to perform all tests below 224 μm.

3.4. Laboratorial procedure

In each test the hydrocyclone feed pulp was made in the sump (Fig.1) by combining the amount of water and solids according to the defined test plan. Sampling of the overflow and underflow products was done simultaneously with 20L buckets, and the duration of the sampling was the time it took to empty the sump. A product that was called waste was also collected which was the material that was not sampled in any of the overflow or underflow products and that came out of the pump by two drains for mass balances. Each sample of the overflow, underflow and waste products was filtered through a pressure filter. The water and the wet sample resulting from the solid-liquid separation performed by the filter were weighed, after which the samples were placed in the oven at 105 °.

After being dried, the samples were weighed and separated with the *Jones* divider. They were sampled to perform particle size analysis with a laser diffractometric particle size analyzer, model *Cilas920L*. The amount that the *Cilas920L* equipment needs to perform a particle size analysis is very small, about 1 gram, constituting a subsample of the initial sample. In preliminary tests, particle size analyzes were performed on the same sample to evaluate the differences that could arise in the result due to sampling and measurement errors by the *Cilas920L* equipment, verifying that the differences were not significant.

4. Results and discussion

Table 2 shows the data collected for the solids and water mass balances in each of the tests, thus constituting measured values without any correction target.

Table 2-Mass balances with measured values of all tests performed

Test	Water volumetric balance (ml)				Solids mass balance (g)					
	Overflow	Underflow	Leftover	Total	Overflow	Underflow	Leftover	Total	Losses	Losses(%)
1	4047.0	4575.4	841.0	9463.4	135.7	1625.4	189.3	1950.5	49.5	2.5
2	3587.2	3973.9	846.0	8407.2	283.4	2871.5	375.6	3530.6	469.4	11.7
3	5321.4	2518.7	886.7	8726.8	214.9	1584.2	154.7	1953.8	46.2	2.3
4	5012.9	2337.0	2251.7	9601.6	473.6	3017.8	437.3	3928.6	71.4	1.8
5	4174.2	4562.1	1212.5	9948.9	132.3	1617.6	227.0	1977.0	23.0	1.2
6	4445.4	4288.6	1207.6	9941.6	304.0	3217.1	460.6	3981.6	18.4	0.5
7	4466.8	4354.7	1152.5	9973.9	119.8	1650.2	217.7	1987.7	12.3	0.6
8	4800.5	4042.6	1123.4	9966.5	285.6	3247.0	430.8	3963.4	36.7	0.9
9	4661.4	4338.1	1106.7	10106.2	190.3	2472.6	320.6	2983.5	16.5	0.6
10	4500.6	4406.7	1239.5	10146.7	197.8	2420.6	356.4	2974.8	25.2	0.8
11	4420.4	4148.4	1359.0	9927.8	213.0	2380.8	377.5	2971.3	28.7	1.0
12	4605.7	4213.4	1257.6	10076.7	209.0	2397.4	370.3	2976.7	23.3	0.8

These data are used to assess, quantify losses, calculate underflow solids recoveries, water flow distribution and product dilutions in each test.

In general, mass balances do not show very high errors of less than 2.5% for solids except test 2 where losses were greater than 10%. Regarding water balances, the largest losses observed were also in test 2. It was found that the errors were decreasing throughout the tests, demonstrating an increasing refinement in the sampling procedure and sample treatment.

The values of the adjusted mass fractions or percentages of each particle size class are the solution of a nonlinear optimization problem subject to restrictive conditions and which aims to minimize the sum of the squared residues between the particle size fractions and underflow, overflow and feed measured and estimated dilutions (2).

$$Min\ SQR = \sum_{i=1}^{n} \left(\frac{f_i - \hat{f}_i}{\sigma_f}\right)^2 + \left(\frac{u_i - \hat{u}_i}{\sigma_u}\right)^2 + \left(\frac{o_i - \hat{o}_i}{\sigma_o}\right)^2 + \left(\frac{D_f - \widehat{D}_f}{\sigma_{D_f}}\right)^2 + \left(\frac{D_u - \widehat{D}_u}{\sigma_{D_u}}\right)^2 + \left(\frac{D_o - \widehat{D}_o}{\sigma_{D_o}}\right)^2 \qquad i = 1,2,\dots,n \qquad (2)$$

$$\sum_{i=1}^{n} \hat{f}_i = 100$$

$$\sum_{i=1}^{n} \hat{u}_i = 100$$

$$\sum_{i=1}^{n} \hat{o}_i = 100$$

$$\hat{f}_i \geq 0$$

$$\hat{u}_i \geq 0$$

$$\hat{o}_i \geq 0$$

$$\hat{f}_i - \left(\hat{R}_U * \hat{u}_i\right) - \left(1 - \hat{R}_U\right) * \hat{o}_i = 0$$

$$\widehat{D}_f - \left(\hat{R}_U * \widehat{D}_u\right) - \left(1 - \hat{R}_U\right) * \widehat{D}_o = 0$$

$$0 \leq \hat{R}_U \leq 1$$

The purpose of the mass balances is to obtain the best possible estimate of the weight recovery of solids in underflow and the particle size compositions of the products adjusted so that the difference between the measured and estimated particle size values and the product dilutions is minimal.

After the step of building consistent data, the work consists in adjustments of partition numbers to known models of partition curves, like Rosin-Rammler and Lynch and Rao, also known as exponential sum. This involved the estimation of each model parameters for all the tests performed. Table 3 resumes all the results obtained.

Table 3-Summary of operating conditions and responses obtained in each test

Fatorial Plan							Responses					
Test	DA	DE	DT	ΔP (bar)	Do/Du	Sampling time(s)	Rf(Plitt) (%)	D50c_Plitt(μm)	D50c(μm)=D80OF*1.25	SI	m	α
E1(E4)	5	2.8	29.8	0.5	1.52	32	51.8	11.6	8.5	1.50	4.4	6.4
E2(E5)	2.5	1.4	12.7	0.5	1.52	33	60.6	12.9	9.3	1.49	3.81	5.44
E3(E6)	5	1.6	24.8	1	1.52	14	33.2	12.0	8.7	1.45	4.48	6.50
E4(E8)	2.5	0.8	10.6	1	1.52	18	32.0	12.1	9.2	1.50	4.05	5.80
E5(New)	5	2.8	31.6	0.5	1.18	26	64.5	11.3	8.4	1.55	4.09	5.97
E6(New)	2.5	1.3	14.6	0.5	1.18	31	46.4	11.8	8.6	1.46	4.56	6.70
E7(New)	5	2.6	37.3	1	1.18	13	47.1	9.9	7.1	1.66	3.44	5.00
E8(New)	2.5	1.2	16.8	1	1.18	21	60.0	9.4	7.1	1.60	3.49	5.12
E9	3.3	1.8	24.5	0.7	1.18	24	50.0	10.3	7.6	1.63	3.63	5.30
E10	3.3	1.8	22.8	0.7	1.18	26	50.4	11.2	8.1	1.56	4.06	5.95
E11	3.3	1.7	20.8	0.7	1.18	25	51.0	10.1	7.4	1.60	3.80	5.58
E12	3.3	1.8	22.0	0.7	1.18	22	46.4	9.7	6.6	1.45	5.02	7.46
						Var	4.37	0.38	0.41	0.01	0.39	0.92
						SD	2.09	0.62	0.64	0.08	0.62	0.96

$DA=D_F$, $DE=D_U=DT=D_O$

As already shown in table 2, for the measured mass balances, test 2 had the highest losses and this is reflected as in d_{50c}, obtaining an anomalous value of 12.9 μm. To analyze the effect of dilution on feed we can compare the tests E1 (E4) and E2 (E5), in which the vortex finder/apex ratio is fixed as well as the pressure.

As would be expected with a lower dilution, i.e. with a higher percentage by weight of solids, the *Rf* would be higher and this is confirmed as in the E1 (E4) assay the dilution is 5 and in the E2 (E5) assay equals to 2.5, whereby the values of *Rf* are 51.8% and 60.6% respectively. Nevertheless, an opposite relationship is observed in the E5 (New) and E6 (New) assays where the lower dilution yields a higher *Rf* value, although the vortex finder/apex ratio is lower than in the E1 (E4), E2 assays. (E5).

The test sets E1 (E2), E2 (E5) and E3 (E6), E4 (E8) are tests in which the vortex finder/apex ratios were maintained, but dilutions and pressures were manipulated. Ignoring the dilution effect and considering only the pressure effect, there is a clear difference in the *Rf* results between the first set of tests E1 (E2), E2 (E5) where the pressure was lower (equals to 0.5 bar) and the second test set E3 (E6), E4 (E8) where the pressure was greater than 1 bar.

Summarily, the values show low variability, except for *Rf*. The differences are so small that it is difficult to ascertain whether they were the result of manipulation of the variables or experimental errors, because there was no repetition of tests. The E2 test has an anomalous value because it is higher compared to the others, but in theory it is the test where operating conditions are more favorable to obtain a higher d_{50c}, with the highest vortex finder/apex ratio, the lowest pressure, injection and the lowest dilution.

Correlation analysis was performed to identify the pairs of values which has correlation between each other and if they are significant.

Table 4-Spearman and p-value correlation value matrix

	Rf	**d_{50c}(µm)**	**SI**	***m***	***α***
D_F	(0.07,0.82)	(-0.15,0.65)	(0.12,0.71)	(0.12,0.71)	(0.15,0.65)
DP(bar)	(-0.52,0.08)	(-0.33,0.3)	(0.27,0.4)	(-0.41,0.18)	(-0.38,0.22)
D_o/D_u	(-0.15,0.63)	(0.77,0.004)	(-0.49,0.11)	(0.21,0.52)	(0.15,0.63)

Table 4 shows Spearman and p-value correlation values between pairs of dependent variables (*Rf*, d_{50c}, SI, *m* and α) and independent variables (D_F, DP and D_o/D_u). The first value in parentheses refers Spearman's correlation coefficient value and the second is p-value.

It can be concluded that the pairs (DP, *Rf*), (DP, *m*) and (D_o/D_u, SI) have a moderate correlation and with evidence that the correlation is significant is weak or null. The pair (DoDu, d50c) has a strong correlation and there is very strong evidence to say that the correlation between them is significant. The correlations between (DP, *Rf*), (D_o/D_u, SI) and between (DP, *m*) are negative and between (D_o/D_u, d_{50c}) is positive.

The proposed relationships between the independent (xi) and dependent (yi) variables have the following general formula (3):

$$y = k0.x1^{k1}.x2^{k2}.x3^{k3} \qquad (3)$$

$$Rf = 48.12 D_o/D_u{}^{-0.54} \Delta P^{\,-0.37} D_F{}^{0.02}, \qquad (4)$$

$$d50c = 9.62 D_o/D_u{}^{0.59} \Delta P^{\,-0.12} D_F{}^{-0.05}, \qquad (5)$$

$$m = 3.6 D_o/D_u{}^{0.15} \Delta P^{\,-0.12} D_F{}^{0.04}, \qquad (6)$$

$$SI = 1.6 D_o/D_u{}^{-0.22} \Delta P^{\,0.05} D_F{}^{0.02}, \qquad (7)$$

$$\propto = 5.26 D_o/D_u{}^{0.09} \Delta P^{\,-0.13} D_F{}^{0.04}\,, \qquad (8)$$

5. Conclusions

The variability of the results is reduced in terms of the dependent variables studied, possibly reflecting the low amplitude experimentation of the operating conditions, which in turn reflected the limitations of the kit. The single pump did not allow to work with pulps with solids percentages greater than 30% and the pressure gauge did not allow stable readings greater than 1 bar, so also the application domain of the empirical models created is also reduced.

Given the small diameters (10-12 μm) operated by the hydrocycloning kit, the main use of cyclones of this type should be for desliming, i.e. removing part of the fine fraction. This makes sense in some processes with the aim of decreasing the percentage of solids by weight or because such a fine fraction has no economic interest.

The value of d_{50c} is essentially determined by the D_o/D_u ratio and drainage coefficients, Rf, of the order of 50%, sensitive to the feed pulp injection pressure. The high values of this coefficient determine that an effective desliming with high percentage elimination (> = 80%) of the infra 10 micrometer fraction is only attainable with successive repulping and hydrocycloning of the respective thickeners. The imperfection of separation operated is essentially independent of the operating conditions tested.

6. Future work

As future work, it would be very interesting to extend the domain of operating conditions and apply the same methodology to compare the empirical models created and the correlation between variables and models.

Improve the experimental arrangement by introducing an independent pulping tank according to the arrangement adopted by Plitt and additionally the use of a sample with a different density or a sample composed of minerals/materials of different densities.

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