

The application of sampling theory in bauxite protocols

Teoria da amostragem aplicada aos protocolos de bauxita

Daniel Armelim Bortoleto

Department of Mining and Petroleum Engineering, University of São Paulo, Brazil bortoda@usp.br

Ana Carolina Chieregati

Doutor Professor

Department of Mining and Petroleum Engineering,
University of São Paulo, Brazil
ana.chieregati@gmail.com

Antônio Henrique Rietra Pereira

Alcoa World Alumina, Juruti Mine, Brazil antonio.h.pereira@alcoa.com.br

Raiza Cavalcante Oliveira

Alcoa World Alumina, Juruti Mine, Brazil raiza.oliveira@alcoa.com.br

Abstract

The applications of Gy's formula are appropriate for calculating variances of the fundamental sampling error (FSE) at any stage of the sampling protocol and before samples are collected as well. However, the formula can be inaccurate because general factors are used to estimate the ore characteristic. In order to allow the calculation of fundamental sampling error and the minimum representative sample masses without using Gy's factors, there are experiments to calibrate the sampling parameters, namely: the heterogeneity test (HT); the sampling tree experiment (STE) and the segregation free analysis (SFA). The present work describes the experimental procedure for the three calibration methods using bauxite and shows the correlation between them.

Keywords: sampling protocols; fundamental sampling error; bauxite.

Resumo

A fórmula de Gy é apropriada para calcular a variância do erro fundamental de amostragem (FSE), em qualquer estágio do protocolo de amostragem e, mesmo, antes de a amostra ser coletada. Entretanto a fórmula pode ser inacurada por utilizar fatores aproximados para todos os tipos de minérios. Para calcular a variância do erro fundamental de amostragem e as massas mínimas das amostras sem usar os fatores de Gy, existem experimentos para calibrar os parâmetros de amostragem, a saber: teste de heterogeneidade (HT); experimento da árvore (STE) e análise de segregação livre (SFA). O presente trabalho descreve os procedimentos experimentais para a bauxita, utilizando esses três métodos de calibração, e apresenta a correlação entre eles.

Palavras chave: Protocolos de amostragem, erro fundamental de amostragem, bauxita.

1. Introduction

Sampling theory of broken material ores cannot be mentioned without a direct reference to Gy's fundamental contributions (Gy, 1998). According to Minnnitt

(2007) apud François-Bongarçon (1998), the fundamental sampling error (FSE) is the smallest achievable residual average error, because of the physical and chemical composition as well as the particle size distribution. FSE can be determined by Gy's formula, applicable to all kinds of ore (Equation 1):

$$s^2_{FSE} = c f g l d_N^3 (\frac{1}{M_s} - \frac{1}{M_L})$$
 (1)

Where Ms is the mass of selected sample, ML is the mass of the original lot, c is the mineralogical factor, f is the shape

factor, g is the granulometric factor, l is the liberation factor and dN is the "top size". Furthermore, the formula allows the calculation of the minimum sample mass to represent a lot for a maximum variance. The experiments developed to estimate the sampling constants K and alpha of Equation 2 are described as

follows, where K is a specific constant to a given ore type at a given grade, and

alpha, is the exponent of original Gy's cubic formula

$$s^2_{FSE} = K d_N^{\alpha} (\frac{1}{M_S} - \frac{1}{M_L})$$
 (2)

2. Methodology

Heterogeneity test (HT)

Initially, a 500 kg sample representing the typical aluminium ore was selected at the mine, then crushed and screened in four size fractions: "-38.1 mm +25.4 mm"; "-25.4 mm +12.7 mm";

"-12.7 mm +6.3 mm"; "-6.3 mm +1.2 mm". For each size fraction, 50 groups of at least 50 fragments each, collected one by one at random, were selected. Once samples were weighted and analyzed, the

mass Mq and grade aq for each group of fragments, as well as the average masses Mq and weighted average grades aQ were calculated according to Equation 3 (Koyama et al., 2010).

$$a_{Q} = \frac{1}{M_{Q}} \sum a_{q} M_{q}$$
(3)

The estimated constant factor of constitutional heterogeneity, EST IHL, was calculated according to Equation

4, for each of the four size fractions. These factors generate a regression line calculated using the four resulting

points, which allows estimating the parameters K and alpha of Equation 2 (Pitard, 1993).

95% passing 0.1 mm, composes the fourth

nominal size fraction. This procedure gen-

erates four portions of more or less equal mass, about 15 kg. Using a riffle splitter,

each of these portions is now split into 32

sub-samples per fraction. All samples are

weighted and chemically analysed. Ac-

cording to Minnitt et al. (2007), the form

of Gy's equation can be changed by taking

the logarithms of both sides, transposing

to Equation 5.

(6)

EST IH_L=
$$g \sum \frac{(a_q-a_Q)^2}{a_Q^2} \cdot \frac{M_q^2}{M_Q}$$
 (4)

The heterogeneity test allows the experimental calibration of the sam-

pling constants K and alpha, which must be calibrated for each particular

ore at a particular grade.

Sampling tree experiment (STE)

The sampling tree experiment proposed by François-Bongarçon (Minnitt (2007) apud François-Bongarçon 1995 and 1998) follows a detailed analysis of the application of Gy's formula to determine the fundamental sampling error. A representative 60 kg sample was collected at the mine. The primary crushing stage using a jaw crusher reduces the ore nominal size to a uniform 95% passing 25.4 mm. One quarter of the total lot is split out

and forms the first nominal size fraction. The remainder of the ore is then crushed to 95% passing in 6.3 mm and the lot is then split into three equal portions. One of these portions is set aside and constitutes the second nominal size fraction. The remaining two fractions are recombined, crushed to 95% passing 2.0 mm and split into two portions of equal mass. One portion is set aside as the third nominal size fraction and the remaining one, crushed to

$$ln(s_{FSE}^2 M_S) = \alpha ln(d_N) + ln(K)$$
 (5)

Segregation free analysis (SFA)

Both the heterogeneity test and the sample tree experiment suffer from weaknesses that cast suspicion on the derived values for K and alpha. Minnitt et al. (2011) proposed a new test called segregation free analysis (SFA). The nominal (average) fragment size, dN, for a fragment passing between two screens

$$\mathbf{d_N} = \sqrt[3]{\frac{\mathrm{USS^3 + LSS^3}}{2}}$$

The material, 300 kg of crushed bauxite collected from many cuts in the sampling station, was classified at 14 size fractions: "-38.1 mm +25.4mm", "-25.4 mm +12.7mm"; "-12.7 mm +6.3 mm"; "-6.3 mm +1.4 mm"; "-1.4 mm

+0.84 mm"; "-0.84 mm +0.60 mm"; "-0.60 mm +0.5 mm"; "-0.5 mm +0.3 mm"; "-0.3 mm +0.23 mm"; "-0.23 mm +0.15 mm"; "-0.15 mm +0.10 mm"; "-0.10 mm +0.07 mm"; "-0.07 mm +0.05 mm"; "-0.05 mm +0.03 mm".

Each size fraction was split using a riffle splitter into a series of 32 samples and sent to the chemical laboratory. The variance of the sample series associated with each fragment size was calculated and plotted to produce a straight line

which can be used to calculate K, alpha and liberation size, d_{i} . Provided that the screen sizes are chosen such that the ratio $r = d_{MAX}/d_{MIN}$ is reasonably consistent, all points representing different d_{MAX} values on the calibration curve will

represent the formula for the relative variance given by Equation 7.

tion between the liberated, calculable

variance, to the non-liberated one given

by Equation 8.

$$s^2_{FSE} = c f g' 1 d^3 (\frac{1}{M_S} - \frac{1}{M_L})$$
 (7)

The granulometric factor g is replaced here by g', where g' = g'(r) is read off the curve of granulometric factor g' vs ratio $r = d_{MAX}/d_{MIN}$, for closely screened materials.

The liberation factor is the transi-

$$l \!\!=\!\! \left(\frac{d_l}{d_{MAX}}\right)^{^{(3-\alpha)}} \tag{8}$$

Equation 9 results from substituting Equation 8 in Equation 7 and

taking the log on both sides. The linear regression built using the 14 resulting

$$n(S_{ral}^2 M_s) = \alpha ln(d_{MAY}) + ln(K)$$
(9)

points gives the sampling parameters K and alpha.

$ln(S^2_{rel}M_S) = \alpha ln(d_{M\Delta X}) + ln(K)$

3. Results and discussion

Heterogeneity test (HT)

EST IHL was calculated using Equation 4. Table 1 shows the results

of EST IHL for each fragment size

(shown in cm). The granulometric

factor for calibrated material is 0.55 (Gy, 1998).

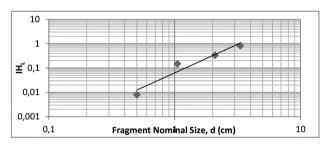
Correlation between IH, and the fragment

Table 1 EST IH, results for corresponding nominal

3.32 2.09 1.05 0.5 d_N **EST IH** 0.823 0.337 0.149 0.008

Figure 1 shows the correlation between the nominal size and EST IH, The equation of the power trendline $(y=c*x^b)$ built for the four fragment sizes gives the sampling parameters K (c of the trendline equation) and alpha

(b of the trendline equation).



According to Figure 1, the estimates for K and alpha are 0.0638 and 2.34 respectively.

Sampling tree experiment (STE)

The alumina content and the sample masses were submitted to a statistical treatment as described by Minnitt et al. (2007) and are presented in Table 2.

Figure 1

nominal size

Serie	Total Mass	Average Grade	CV	Relative VAR	Stnd. Rel VAR	Ln(d _N)	Ln(♂² × Ms)
1	477.3	48.17	0.0254	0.0006	0.0312	0.9322	2.7005
2	460.48	47.86	0.0126	0.0002	0.0076	-0.4541	1.2503
3	459.96	47.62	0.0195	0.0004	0.0181	-1.6094	2.1219
4	407.03	48.06	0.0067	0	0.0022	-4.5469	-0.1191

Table 2 STE statistical results

Where relative variance is CV² (coefficient of variation), standard

relative variance (σ^2) is the product between average grade and relative variance, and M_a is the sample average mass. Four fragment sizes were used to build

the regression line (Figure 2) according

to Equation 5.

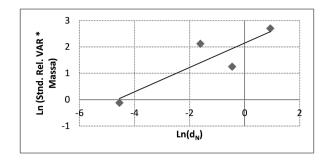


Figure 2 K and alpha calibration curve for a 47.8% Al_2O_3 ore.

The exponential function of the intercept on the y-axis provides an

estimate of K. According to the sampling tree experiment, the estimates

for *K* and alpha are 8.54 and 0.46 respectively.

Segregation free analysis (SFA)

The alumina content and the sample masses for the 14 size fractions were submitted to a statistical treatment as

described by Minnitt et al. (2011) and are presented in Table 3. The relative variance is CV² (coefficient of variation), standard

relative variance (o²) is the product between average grade and relative variance and Ms is the sample average mass.

Serie	Total Mass	Average Grade	CV	Relative VAR	Stnd. Rel VAR	Ln(dN)	$Ln(\sigma^2 \times M_s)$
1	360.07	50.98	0.04	0.0019	0.097	0.932	3.556
2	168.19	51.17	0.04	0.0013	0.064	0.239	2.381
3	176.61	48.77	0.02	0.0004	0.019	-0.454	1.197
4	11w3.37	50.51	0.01	0.0001	0.007	-1.966	-0.223
5	59.80	47.74	0.01	0.0001	0.005	-2.465	-1.215
6	27.58	49.51	0.01	0.0001	0.004	-2.813	-2.132
7	43.29	49.66	0.01	0.0001	0.006	-2.996	-1.300
8	26.34	50.32	0.02	0.0003	0.017	-3.507	-0.803
9	38.11	49.78	0.01	0.0002	0.008	-3.854	-1.213
10	40.08	50.48	0.01	0.0002	0.008	-4.200	-1.097
11	46.21	53.14	0.01	0.0001	0.003	-4.547	-2.010
12	34.32	50.83	0.01	0.0002	0.011	-4.893	-0.942
13	40.82	50.53	0.01	0.0001	0.007	-5.240	-1.184
14	39.64	50.03	0.01	0.0000	0.002	-5.573	-2.686

Table 3 SFA statistical results

The 14 fragment sizes were used

to build the regression line on Figure 3, according Equation 9.

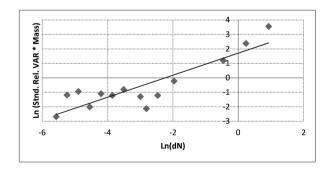


Figure 3 K and alpha calibration curve for a 50.2% Al₂O₃ ore

According to SFA, the estimates for

K and alpha are 5.49 and 0.76 respectively.

Comparison between sampling protocols using HT, STE and SFA

Table 4 shows the values of Gy's factors (Pitard, 1993) for Juruti's bauxite according to Equation 1.

Factor	С	f	g	di	Al_2O_3	λΜ	λG	IH
Values	3.6	0.5	0.25	0.00036	48.1%	3.98	2.65	0.0085 d ^{2.5}

Table 5 shows the sampling and sample preparation protocol for Juruti's mine and the relative standard deviation

of the fundamental sampling error (last 4 columns, in %), s_{FSE} , of each stage using Gy's factors (Gy,1998) as well as the

Tal	ole 4
Fac	ctors used in Gy's relative variance
equ	uation

results of HT, STE, SFA.

Step	M _L (g)	MS (g)	d _N (cm)	s _{FSE} GY	s _{FSE} HT	s _{FSE} STE	s _{FSE} SFA
Primary Samping	1.5E+10	15000	5	0.5628	1.3461	3.4618	3.5328
Crushing	15000	15000	2.54	0.0000	0.0000	0.0000	0.0000
Primary Quartering	15000	2500	2.54	0.5397	1.3632	6.6183	6.1023
Grinding	2500	2500	0.2	0.0000	0.0000	0.0000	0.0000
Secondary Quartery	2500	200	0.2	0.0836	0.2592	13.6578	8.6044
Pulverization	200	200	0.015	0	0	0	0
TOTAL		·		0.784 %	1.933 %	15.567 %	11.125 %

Considering Equation 2, a maximum relative standard deviation of the fundamental error of 3% and a high value

for the initial mass M_L , the minimum sample mass to represent the primary sample at the mine, where $d_{\rm max}$ is 5 cm,

Table 5
Sampling protocol comparing the three heterogeneity experiments

can be calculated. The last line of table 6 shows the same calculations for a $\rm d_{MAX}$ of 1 cm.

$d_{MAX}(cm)$ 5	5	5	5
$M_{s}(g)$ 530	3.063	19.895	20.728
d _{MAX} (cm) 1	1	1	1
$M_s(g)$ 9	71	9.486	6.099

Table 6
Minimum sample masses (Ms) for the primary sample at the mine

4. Conclusions

This paper presented a comparison of different methods to estimate the variance of the fundamental sampling error, minimum sample masses and the sampling parameters by different methods. An appropriate sampling protocol for any type of ore requires experimental test work in order to determine the deposit's heterogeneity. The heterogeneity test (HT) has been used for several decades and has proved to be an excellent method to calculate the minimum representative sample masses and to optimize sampling protocols during the beginning of mining projects and during operation as well. However, the new proposed tests show significant differences

which should be taken into account. The comparison between the results of STE and SFA shows the relationship between the fundamental sampling error and the grouping and segregation error. SFA was developed aiming to eliminate the residual grouping and segregation variance which biases the calibration curve and should present smaller variances than STE. However, for higher diameters, SFA presents higher values of s2SFE, suggesting that the fundamental sampling error makes the most important role for coarser particles. Based on the results, the company decided to adopt the segregation free analysis for defining its sampling protocols due to two major

reasons: (1) the SFA proved to reduce the grouping and segregation error and (2) the SFA presented higher minimum sample masses than the heterogeneity test, making this method safer than the HT with respect to the representativeness of samples. The results showed that optimizing sampling protocols is not a simple task and more than one experimental test should be performed to confirm what the minimum sample masses should be. This paper is a study for bauxite and may be not applicable to other types of ore. The authors highly recommend other companies to compare the different approaches and define the most suitable for their operations.

5. Acknowledgments

The authors acknowledge Alcoa World Alumina and University of São Paulo for their support in conducting the research and producing this paper.

6. References

GY, P.M. *Sampling for analytical purposes*. West Sussex, England: JohnWiley & Sons, 1998. (Translated by A.G. Royle).

PITARD, F.F. *Pierre Gy's sampling theory and sampling practice*. (2. ed.). CRC Press, Boca Raton, 1993.

KOYAMA, I.K., CHIEREGATI, A.C., ESTON, S.M. Teste de heterogeneidade como método de otimização de protocolos de amostragem. Brasil Mineral, v. 27,

- p. 63-68, 2010.
- MINNITT, R.C.A., RICE, P.M., SPANGENBERG C.. Part 2: Experimental calibration of sampling parameters K and alpha for Gy's formula by the sampling tree method. The Journal of *The Southern African Institute of Mining and Metallurgy*, p. 513-518, 2007.
- MINNITT, R.C.A., FRANÇOIS-BONGARÇON, D., PITARD, F.F. 2011. Segregation Free Analysis for calibrationg the constants K and α for use in Gy's formula. In: *WORLD CONFERENCE ON SAMPLING AND BLENDING*, 5. Gecamin, Santiago, p.133-150.
- MINNITT, R.C.A. APUD FRANÇOIS-BONGARÇON, D. Gy's Formula: Conclusions of a new phase of research. *Proceedings Australian Association of Geoscientists*. v.22, 1998.

sing, v. 29, n. 3-4, p. 149-174, 1990.

Received: 08 September 2013 - Accepted: 23 March 2013.