A multidimensional analysis and modelling of flotation process for selected Polish lithological copper ore types

Tomasz Niedoba¹, Agnieszka Surowiak¹, and Paulina Pięta^{1,*}

¹AGH - University of Science and Technology, Faculty of Mining and Geoengineering, Department of Environmental Engineering and Mineral Processing, Cracow, Poland

Abstract. The flotation of copper ore is a complex technological process that depends on many parameters. Therefore, it is necessary to take into account the complexity of this phenomenon by choosing a multidimensional data analysis. The paper presents the results of modelling and analysis of beneficiation process of sandstone copper ore. Considering the implementation of multidimensional statistical methods it was necessary to carry out a multi-level experiment, which included 4 parameters (size fraction, collector type and dosage, flotation time). The main aim of the paper was the preparation of flotation process models for the recovery and the content of the metal in products. A MANOVA was implemented to explore the relationship between dependent (β , ϑ , ε , η) and independent (d, t, cd, ct) variables. The design of models was based on linear and nonlinear regression. The results of the variation analysis indicated the high significance of all parameters for the process. The average degree of matching of linear models to experimental data was set at 49% and 33% for copper content in the concentrate and tailings and 47% for the recovery of copper minerals in the both. The results confirms the complexity and stochasticity of the Polish copper ore flotation.

1 Introduction

Flotation is a method of enrichment used to distribute grains, which differ in their surface properties. During the process the bubble-particle aggregates are formed and they are upraised to flotation froth. However, copper minerals do not exhibit natural floatability, which forces certain substances to modify their hydrophobicity.

The main factors that determine the final result in terms of high yield and content of the minerals in the concentrate can be divided into 3 groups: feed properties, flotation device characteristics and enrichment methods. An accurate analysis of these three aspects led to a set of more than100 different factors which simultaneously affect the final effects of the separation and they interact with one another. [Bazin, 2001; Aldrich, 2002; Drzymała, 2007; Brożek & Młynarczykowska, 2009].

What causes an additional difficulty of Polish copper ores beneficiation is their complex structure and the presence of three lithological types: dolomite, sandstone and shale, also varied internally. These lithological fractions appear simultaneously in most parts of the deposit, but in varying proportions. However, their exploitation does not currently allow to be selective. In addition, each type has different types and sizes of copper minerals. Other characteristics that differentiate the individual lithological fractions are the enrichment and copper content of the feed. Sandstone type exhibits the greatest floatability and the lowest content of copper minerals. The reverse situation is for the shale type, which is by far the hardest to enrich, but it is characterized by the highest average content of copper. On the other hand, dolomite copper ore has indirect properties in terms of the discussed features [Laskowski & Łuszczkiewicz, 1989; Kijewski & Jarosz, 2007; Kłapciński & Peryt, 2007; Nieć & Piestrzyński, 2007].

The complex structure of the phenomenon induces to choose multidimensional methods of data analysis that match the nature of the process and the properties of the feed. The use of universal, multi-parameter statistical methods allows a full analysis of empirical data, although their multiplicity may give rise to some interpretation difficulties. However, the implementation of mathematical statistics in the consideration of the flotation process and other issues related to the mineral processing is fully justified [Tumidajski, 2010; Tumidajski et al., 2012; Duchnowska et al., 2015].

2 Aims and experiment description

The aim of the study was analyzing and modelling the effects of the sandstone copper ore flotation in a Denver flotation machine in terms of yield and content of metals in concentrates and tailings in multi-parameter approach. The application of multidimensional methods of data analysis enabled a full examination of the complex flotation process and the determination of functional

© The Authors, published by EDP Sciences. This is an open access article distributed under the terms of the Creative Commons Attribution License 4.0 (http://creativecommons.org/licenses/by/4.0/).

^{*}Corresponding author: <u>ppieta@agh.edu.pl</u>

dependency between selected separation indices and parameters.

The starting point for further consideration of the sandstone copper ore beneficiation was the results of laboratory tests that allowed the achievement of the objectives. Implementations of multivariate statistical methods and estimation of the process model were necessary to perform a multi-level experiment which assumed four analysed parameters (grain size, type of collector reagent, the dose of the reagent collecting the flotation time).

The material was taken from the technological line of the Copper Ore Preparation Plant Polkowice KGHM Polska Miedź SA. Sandstone copper ore was separated from the sample and subjected to further preparatory work. One of the aims of the analysis was to investigate the granulation influence on effects of copper ore flotation.Therefore, as a result of appropriate comminution and sizing processes, the following size fractions were separated: 125-200, 100-125, 71-100, 40-71, 20-40, 0-20 [μ m], which constituted feed material for laboratory flotation tests in a Denver flotation machine. The average content of copper in individual size fractions were characterized in Tab. 1.

Experiments were carried out to maintain a constant concentration flotation slurry 200 g/dm3, an aerating and a rotor speed. Two types of flotation collector were used during the tests and they were added in two dosages:100 g/Mg and 150 g/Mg. They were an Hostaflot and a sodium ethyl xanthate aqueous solution. Nasfroth in dosage 50 [g/Mg] was used as a frother during the test. Before starting the single flotation test, sample was mixed in a flotation cell for 3 min, followed by addition of flotation reagents for the next 7 min. Samples of the froth concentrate were collected after 1, 2, 4, 6, 9, 12, 17, 22, 30 minutes. The flotation concentrates and tailings were dried and weighed and subjected to X-ray fluorescence to determine the copper content. According to the experimental plan, 24 major flotation tests were performed, which were repeated four times. The data set collected in this way made it possible to draw up a mathematical description of the process and to carry out the analysis. For each experiment, the following process evaluation indicators were determined: copper content in concentrate β (1), copper content in tailings (2), copper recovery in concentrate ε (3), copper recovery in tailings η (4), which are determined by the following formulas :

$$\beta_i = \frac{\sum_{i=1}^n \gamma_i \cdot \lambda_i}{\sum_{i=1}^n \gamma_i} \tag{1}$$

$$\vartheta_i = \frac{\sum_{i=1}^n \gamma_i \cdot \lambda_i}{1 - \sum_{i=1}^n \gamma_i} \tag{2}$$

$$\varepsilon_i = \frac{\beta_i}{\alpha} \sum_{i=1}^n \gamma_i \tag{3}$$

$$\eta_i = \frac{\vartheta_i}{\alpha} \sum_{i=1}^n \gamma_i \tag{4}$$

where: $\alpha - a$ copper content in the feed, $\gamma_i = \frac{m_i}{\sum_{i=1}^n m_i} - a$ yield of a product [%], $\lambda_i - \text{content of copper in a product,}$ $m_i - \text{an amount of a product.}$
 Table 1. The average copper content in size fractions.

Size fraction [µm]	Copper content [%]
0-20	1.50
20-40	1.51
40-71	1.10
71-100	0.42
100-125	0.30
125-200	0.21

Conducting tests in accordance with the requirements to the maintenance of representativeness of the data collection is time-consuming and requires considerable workload. The analyses, tests, or coefficients decreased in their simplicity compared to a 1 or 2-parameters experiments. For these reasons, multi-level experiments are rarely used in optimization, but they have undeniable benefits.

3 Calculations methods

The analysis and evaluation of the impact of the studied process parameters on the quality and composition of flotation products were based on the methods from the family of variance analysis. The multivariate analysis of variance (MANOVA) is a method that allows numerical analysis of at least two dependent variables while controlling any number of independent variables. Depending on the aim, it can be carried out as a one- and multivariate scheme. In this case, the multivariate vector of dependent variables Yi (5) is characterized by the following variables: $Y_1 = \beta_{Cu}$, $Y_2 = \vartheta_{Cu}$, $Y_3 = \varepsilon_{Cu}$, $Y_4 = \eta_{Cu}$. However, in the analysis, the vectors of mean values of the variables μ_i (6), where i=1, 2, ..., N for different systems of factors and their levels (6) were compared[Stanisz, 2007; Niedoba, 2013; Niedoba et al., 2016a, Niedoba et al., 2016b]. The relevance of the main effects among the investigated factors was considered for significance level equalled to 95%, which means that the model with a P-value lower than 0.05 could be considered.

$$Y_{i} = \begin{bmatrix} \beta_{iCu} \\ \vartheta_{iCu} \\ \varepsilon_{iCu} \\ \eta_{iCu} \end{bmatrix}$$
(5)

$$\mu_{i} = \begin{bmatrix} \mu_{\beta i} \\ \mu_{\vartheta i} \\ \mu_{\varepsilon i} \\ \mu_{\eta i} \end{bmatrix}$$
(6)

A linear (7) and linearized (8) model allowed to determine quantitative relationships between the measured parameters of the process $(x_1=d, x_2=t, x_3=c_t, x_4=c_d)$ and response variable (y_i) . Equations (7) and (8) describe the general regression equations that form the basis for modelling.

$$Y_i = \beta_0 + \beta_1 \cdot x_1 + \beta_2 \cdot x_2 + \beta_3 \cdot x_3 + \beta_4 \cdot x_4 + \varepsilon_i \quad (7)$$

$$Y_i = \beta_0 + \beta_1 \cdot \ln(x_1) + \beta_2 \cdot \ln(x_2) + \beta_3 \cdot \ln(x_3) + \beta_4 \cdot \ln(x_4) + \varepsilon_i$$
(8)

where: d - size fraction,

- t- flotation time,
- c_t collector type,
- c_d collector dosage.
- β_i model parameter,
- ε_i standard estimation error.

In order to assess the accurateness of matching the regression functions to the experimental data, coefficients of determination R^2 were used. It takes values from the range <0.1>.When the values of R2 are close to 1, the model images an appropriate explanation of the variability of experimental values to the predicted values [Jajuga, 1993; Güler & Akdemir, 2012, Masiya & Nheta, 2014; Nakhaei et al., 2015].

4 Results and discussion

The basis for the application of the MANOVA is preservation of the assumptions concerning the representativeness of the dataset and sampling, the normality of distributions of the dependent variables, and the homogeneity of the intergroup variance. However, by choosing the appropriate statistics, it is possible to refrain from adhering to these conditions in situations of minor deviations from the assumptions [Maxwell & Delaney, 2004]. Shapiro-Wilk normality and Brown-Forsyth homogeneity of variance tests were performed for the analyzed dataset, which in a few groups indicated a moderate deviation from normality. Relying on this finding Pillai's trace (9) was used in the MANOVA [Stanisz, 2007]:

$$V = \sum_{i=1}^{q} \frac{\lambda_i}{1+\lambda_i} \tag{9}$$

where: q – number of non-zero own values λ_i of matrix $I+BW^{-1}$,

 λ_i – own value of matrix *BW*⁻¹,

- **B** matrix of intergroups of sums of squares and mixed products,
- W matrix of intersection sums of squares and

mixed products.

Based on the results of the multivariate significance test (Tab. 2) it can be concluded that the combined effect of all tested independent variables (flotation time, dose and type of collector, feed granulation) on the effectiveness of the flotation process is significant. A onedimensional analysis of variance (Tab. 3) confirmed these conclusionsin most cases. For single analyses only the lack of significant impact of the type of collector on the fianal effect was noted, as shown in Fig.2.

In order to analyze the effects of individual parameters on the flotation enrichment results, the mean standardized values of the separation rating indicators at different levels of independent variables are also shown in Fig. 1-4.

 Table 2. The results of multivariate tests of significance using Pillai's trace.

Variable	V	F	р
Flotation time t	0.621	34.732	0.000
Collector type ct	0.252	119.069	0.000
Collector dosage cd	0.435	272.115	0.000
Size fraction d	0.793	76.472	0.000

 Table 3. The results of one-dimensional tests of significance (Fisher-Snedecor test).

Variables	Copper in tail	Copper recovery in tailings η		Copper content in tailings ५	
v al lables	F	р	F	р	
Flotation time t	54.448	0.000	5.483	1.10-5	
Collector type ct	311.918	0.000	<u>2.498</u>	<u>0.114</u>	
Collector dosage cd	164.200	0.000	52.878	0.000	
Size fraction d	100.039	0.000	108.903	0.000	
Variablas	Copper in conce	recovery ntrate ε	Copper in conce	content ntrate β	
Variables	Copper in conce	recovery ntrate ε Ρ	Copper in conce F	content ntrate β P	
Variables Flotation time t	Copper in conce F 54.451	recovery entrate ε p 0.000	Copper in conce F 54.451	content ntrate β p 0.000	
Variables Flotation time t Collector type ct	Copper 1 in conce F 54.451 311.934	recovery ntrate ε P 0.000 0.000	Copper in conce F 54.451 311.934	content ntrate β P 0.000 0.000	
Variables Flotation time t Collector type ct Collector dosage cd	Copper lin conce F 54.451 311.934 164.203	recovery ntrate ɛ P 0.000 0.000 0.000	Copper in conce F 54.451 311.934 164.203	content ntrate β P 0.000 0.000 0.000	

^{*}p>0.05 no significant influence of the independent variable on the dependent variable



Fig. 1. Standardized average value of the dependent variable for the two dosages of the collector.



Fig. 2. Standardized average value of the dependent variable for the two types of the collector (1-sodium ethyl xanthate aqueous solution, 2-Hostaflot).



Fig. 3. Standardized average value of the dependent variable on time.



Fig. 4. Standardized average value of the dependent variable on size fractions.

It was found that the best effects of flotation were obtained for 40-71 and 20-40 $[\mu m]$ size fractions. The use of xanthate mixtures clearly increased the metal recovery and contentin the froth product compared to the second collector. Higher copper recovery was also obtained with

the introduction of an increased dosage (150 g/Mg) of the collector, which naturally degraded its quality.



Fig. 5. Experimental and theoretical values for copper recovery in concentrate.

The construction of the model describing dependencies between the variables was based on linear (7) and nonlinear regression (8). Linear regression equations for copper recovery in the concentrate ε (10) and tailings η (11) included four independent variables that had a significant effect on the results of the flotation tests and the lack of mutual correlation. Fig. 5-6 show the regression function with the experimental points for the copper recovery in the concentrate. Their placement indicates the random nature of the process and therefore the difficulty in defining the model for ε and η . In both cases, dependent variables were explained by models only in 47%, hence confirming its poor match.

$$\varepsilon = 0,84446 + 0,0056 \cdot t - 0,10921 \cdot c_t + 0,00158 \cdot c_d - 1,2178 \cdot d \pm 0.112$$
(10)

 $\eta = 0.15556 - 0.0056 \cdot t + 0.10921 \cdot c_t - 0.00158 \cdot c_d + 1.2178 \cdot d \pm 0.112$ (11)



Fig. 6. Experimental and theoretical values for copper recovery in tailings.

For the other two parameters (β, ϑ) it was necessary to introduce linearized models, which, despite the shape of the empirical cloud, did not work well for copper recovery in both products. The linearized regression equation of the general form given by the formula (8) was obtained by making an elementary substitution of $d_i = \ln(d_i)$, $t_i = \ln(t_i)$, $c_{t_i} = \ln(c_{t_i})$, $c_{d_i} = \ln(c_{d_i})$. In this way, semilogarithmic functions were outlined, in which the curvilinear relation was reduced to a simpler linear form. According to the results of one-dimensional variance analysis, the copper content in concentrate model (12) included all the tested parameters, and the influence of collector type on separation results was omitted in the model of copper content in tailings (13). Based on the graphs of experimental and theoretical values (Fig. 7-8), significant differences can be noted for β and ϑ variables. The values of the determination coefficient R^2 confirms the conclusion. Evaluation of fit of all models is presented in Table 4.

 $\beta = 5.35713 + 0.21758 \cdot ln(d) - 0.42454 \cdot ln(t)$ $-0.28347 \cdot ln(c_t) - 1.46706 \cdot ln(c_d) \pm 0.59207 \quad (12)$

 $\vartheta = 0.02715 - 0.00526 \cdot ln(d) + 0.0013 \cdot ln(t)$ $-0.00795 \cdot ln(c_d) \pm 0.00746$ (13)



Fig. 7. Experimental and theoretical values for copper content in concentrate.



Fig. 8. Experimental and theoretical values for copper content in tailings.

Table 4. The coefficient of determination R ² for models.

Dependent variables	Adjusted R ²
Copper content in concentrate	0.492
Copper recovery in concentrate	0.469
Copper content in tailings	0.336
Copper recovery in tailings	0.469

5 Conclusions

The paper presents the results of a multidimensional analysis and modelling of copper content and recovery in products of sandstone copper ore originating from Polkowice Copper Preparation Plant. The study concerned the influence of four controlled parameters (feed granulation, flotation time, dosage and type of collector) on the final effect of the process (recovery and content of copper in separation products).

Based on MANOVA results at a 95% confidence level, it was found that all investigated factors significantly affected the flotation of copper ore. Only the one-dimensional variance analysis performed for each dependent variable indicated some minor deviations that were included in the models.

Models of copper recovery in concentrate and tailings were generated by the linear regression method. The function of the relationship between the parameters involved all the variables, and the degree of model match to the experimental data in both cases was equal to 47%. In order to model the copper content in the concentrate and tailing, it was necessary to introduce nonlinear The accomplishment of methods. elementary substitutions $x_i = \ln(x_i)$ enabled the preparation of semilogarithmic functions, in which the curvilinear relations were reduced to a simpler linear form. According to the results of the one-dimensional analysis of variance in the model of copper content in the concentrate, all investigated process factors were taken into account. On the other hand, the copper content in the tailing did not account for the impact of the collector type on the separation results. In all cases, the degree of explanation of the phenomenon by the models was unsatisfactory, not exceeding 50%. In addition, the distribution of empirical points indicated the random character of the copper ore flotation process, and therefore significant difficulties in determining the regression function. Flotation, as the main operation of enrichment of Polish copper ore, is characterized by its complex structure and randomization. Modelling such processes is extremely difficult, and in some cases even impossible.

The paper is the effect of the scientific project no. 15.11.100.021.

References

- 1. C. Aldrich, Process Metallurgy 12 (2002)
- C. Bazin, M. Proulx, Int. J. Miner. Process. 61, 1-12 (2001)
- 3. M. Brożek, A. Młynarczykowska, *Kinetics of flotation*. (IGSMiE PAN, Cracow, 2009) [in Polish]
- J. Drzymała Mineral Processing. Foundations of theory and practice of minerallurgy. (Oficyna Wydawnicza Politechniki Wrocławskiej, Wroclaw, 2007) [in Polish]
- M. Duchnowska, E. Kasińska-Pilut, A. Bakalarz, K. Konieczny, P. Kowalczuk, A. Łuszczkiewicz, CUPRUM, 2(75), 97-108 (2015) [in Polish]
- 6. T. GÜLER, U. AKDEMIR Trans. Nonferrous Met. Soc. China, **22**, 199-205 (2012).
- 7. K. Jajuga, *Multidimensional statistical analysis*. (PWN, Warsaw, 1993) [in Polish]
- P. Kijewski, J. Jarosz, Monograph KGHM PM SA II.2.24 *Mineral properties*. (KGHM CUPRUM Sp. z o.o. CBR, Wroclaw, 244-245, 2007) [in Polish]
- J. Kłapciński, T.M. Peryt, Monograph KGHM PM SA II.2.1*The geological structure of the Fore-Sudetic Mnocline* (KGHM CUPRUM Sp. z o.o. CBR, Wroclaw, 69-77 2007) [in Polish]
- J. Laskowski, A. Łuszczkiewicz, Mineral prcessing. Enrichment of mineral resources. (Oficyna Wydawnicza Politechniki Wrocławskiej, Wroclaw, 1989) [in Polish]
- T. Masiya, W. Nheta, Flotation of nickel-copper sulphide ore: optimisation of process parameters using Taguchi method. (Proceedings of the International Conference on Mining, Material and Metallurgical Engineering, Prague, Czech Republic, 113, 1-11, 2014)

- 12. S.E. Maxwell, H.D. Delaney, *Designing experiments* and analyzing data. A model comparison Perspective, 2-nd ed. (Lawrence Erlbaum Associates, 2014)
- F. Nakhaei, M. Irannajad, A. Sam, A. Jamalzadeh, Physicochemical. Problem. Mi., 52(1), 252-267 (2015)
- M. Nieć, A. Piestrzyński, Monograph KGHM PM SA II. 2.19*Mineralisation*, (KGHM CUPRUM Sp. z o.o. CBR, Wroclaw, 167-197, 2007) [in Polish]
- 15. T. Niedoba, *Multidimensional characteristics of* random variables in description of grained materials and their separation processes (Mineral Resources Management, Cracow 2013)
- T. Niedoba, A. Surowiak, P. Pięta, J. Pol. Miner. Eng. Soc., 17(1), 15-22 (2016a)
- T. Niedoba, P. Pięta, A. Surowiak, D. Jamróz, Multidimensional statistical and visualization methods in description of grained materials. (MEC2016, E3S Web of Conferences, 8, 01036, 2016b)
- A. Piestrzyński Monograph KGHM PM SA II.2.19 Mineralisation. (KGHM CUPRUM Sp. z o.o. CBR, Wroclaw, 167-197, 2007) [in Polish]
- 19. A. Stanisz, *The accessible statistics course using STATISTICA PL on examples from medicine. Volume 2. Linear and nonlinear models* (StatSoft, Cracow, 2007) [in Polish]
- 20. T. Tumidajski, MRM, 23(6), 111-123 (2010).
- T. Tumidajski, T. Niedoba, D. Saramak, AGH Journal of Mining and Geoengineering, 36 (4), 167-177 (2012)