The comparison of laboratory and industrial copper ore upgrading indices during introduction of new flotation reagents

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Abstract. This article draws attention to the need for the introduction of a laboratory testing methodology for new flotation reagents prior to the implementation of them for industrial use which would be more effective than the one currently used. Laboratory and industrial indicators of copper ore beneficiation during the testing of new flotation reagents are compared herein. For this purpose, two-stage laboratory tests and industrial trails were carried out on three types of collector mixtures, the results of which were used to analyse the technological indicators achieved. It has been concluded that the applied laboratory testing methodology requires a new approach that allows for a more precise correlation of variables which will allow the results of industrial trial to be predicted with greater probability than at present.

1. Introduction

The regulation on the manufacture and use of chemicals (REACH) introduced by the European Union has led to certain restrictions on the placing on the market of chemicals which could be used as flotation collectors. For this reason, it is more difficult than ever to apply on an industrial scale collection reagents which have not previously been used. This is mainly due to legal restrictions on the production of reagent in a quantity that would allow for the verification of laboratory test results under industrial conditions. Therefore, it is advisable to develop a laboratory methodology that reflects industrial conditions to a greater extent than it did before, and thus will allow effective implementation of new collecting reagents in industrial practice, eliminating the risk of production losses.

Despite the fact that a variety of work has been carried out so far to develop an effective method for prediction of beneficiation results on an industrial scale on the basis of data from laboratory experiments [1, 2, 3, 4], there is still no effective tool for achieving a higher degree of correlation of industrial research results on the basis of results obtained during laboratory tests.

All the methods, which have been presented in the literature as well as developed for the needs of processing plants, boil down mainly to conduct research in simple and small-scale systems. The results obtained are chemical analyses based on which balances of experiments are prepared in the form of tables, with the determination of upgrading indices (product yields and grades and metal recoveries). Therefore, the analysis of the impact of a reagent on flotation performance is created mainly on numerical chemical analysis data as well as on graphical interpretation. On the basis of the analysis of the obtained results, a decision is made concerning the carrying out of industrial scale trial.

The current actions, based on what has already been achieved by researchers, aim at developing an effective method for testing new reagents under laboratory conditions that will make possible the prediction of industrial results. In order to meet the expectations of technology optimization works have been started towards the development of a methodology that would allow for a decision of launching industrial scale trial on the basis of laboratory tests results with minimizing the risk of production losses. The issue of searching for new reagents for the industrial process is an open question. New chemical compounds whose application may improve the efficiency of the
technological process are constantly being sought, which makes it necessary to conduct research in this area.

The number and complexity of different factors present in industrial conditions make the issue of precise and approvable results prediction extremely difficult. In existing plant technology lines the ore beneficitation process is disturbed by natural fluctuation of ore physicochemical properties influencing on process efficiency such as: chemical and mineral composition, degree of minerals liberation, grain size, contains of compounds disturbing process. Moreover the technology regime causes changes in throughput, materials flow rates, flotation reagents distributions, the scale of slurry recirculation and water to solids ratio having impact on slurry density. Additionally, gradually aging of machinery, servicing and maintenance breaks, unplanned shutdowns and processing water chemistry are also significant for process efficiency. All listed factors have independent impact on ore processing results, some of them, but not all, have the numerical values and this is the reason for difficulties in developing an universal formula for results scale-up.

2. The analysis of the influence of feed grade fluctuation on the results of laboratory tests

A comprehensive analysis concerning the existing methodology of laboratory tests of flotation reagents does not allow for defining guidelines for its implementation to forecast of industrial results. Therefore, the factors that may influence the increase in the degree of achievement of industrial results based on laboratory tests were analysed. Several of them were selected, the most important of which are the quality of the output constituting a feed suitable for processing, the content of individual lithological fractions and the quality of mineralization.

First of all, the influence of the variability of ore grade on the results was analysed. The results of laboratory test carried out simultaneously during the industrial trial, compared with previous laboratory test on the basis of which it the decision concerning the carrying out of industrial trial was made, will show whether the variability of feed over time has a significant impact on the prediction of industrial trial results.

Introduction of a new type of reagent for industrial practice at KGHM was an example to research of influence of feed variability on effects resulting from the scale-up of results. In order to verify this relationship, laboratory tests with different mixtures of flotation reagents (named A, B, C) were carried out and then repeated in parallel with ongoing industrial trails.

Laboratory experiments were carried out on material from one of the concentrator plant. Material for the study was collected in two periods: 3 months before the planned industrial test and during the industrial test. In both cases, the methodology of laboratory flotation was identical. The experiments were carried out with a Mechanobr laboratory flotation machine, with a cell of 1 dm³ capacity. The flotation tests included laboratory flotations with selected mixtures. The flotation time has been set at 30 minutes. Fractional flotations were carried out, three froth products were collected (K1 = 5 min, K2 = 10 min, K3 = 30 min) and remaining tailing (OK). Collectors were dosed in the amount of 100 g/Mg, whereas frother in the amount of 60 g/Mg. Collectors were dosed in two stages: the first dose (60% of the total reagent) was added during agitation and the next 40% was added after 5 minutes of flotation. The frother was dosed in one step. The flotation products were dried, weighed and prepared to chemical analyses for Cu content. In order to reduce the systematic errors, the flotation experiment was conducted for each mixture twice, determining Cu in each of the products.

The results of flotation experiments are presented in Tables 1 to 6. On the basis of the data obtained, the Fuerstenau curves ($\varepsilon_r \equiv f(\varepsilon)$) were plotted, the magnifications of which are shown in Fig. 1a ÷ 3a and the Halbich curves ($\varepsilon \equiv f(\beta)$), the fragments of which are shown in Fig. 1b ÷ 3b. Figures 1 ÷ 3 also show the point of industrial upgrading, in order to illustrate the differences between the results of laboratory and industrial scale.
2.1. Results of flotation conducted with mixture A

**Table 1.** Results of laboratory flotation prior to the industrial trial

| Product | Flotation time | Product yield $\gamma$[g] | Product yield $\gamma$[%] | Cu content $\lambda$[%] | Cumulat. yield $\Sigma\gamma$[%] | Cumulat. Cu content $\Sigma\text{Cu}$[%] | Cu recovery, [%] |
|---------|---------------|---------------------------|--------------------------|------------------------|-----------------------------|--------------------------------|-----------------|
| K1      | 5 min         | 81.26                     | 17.09                    | 8.69                   | 17.09                       | 8.69                           | 81.21           |
| K2      | 15 min        | 75.06                     | 15.79                    | 1.38                   | 32.87                       | 5.18                           | 93.16           |
| K3      | 30 min        | 34.58                     | 7.27                     | 0.48                   | 40.15                       | 4.33                           | 95.09           |
| OK      |               | 284.6                     | 59.85                    | 0.15                   | 100.00                      | 1.83                           | 100.00          |
| $\Sigma$ |             | 475.5                     | 100.00                   | 1.83                   | 100                          | 1.83                           |                 |

**Table 2.** Results of laboratory flotation carried out during the industrial trial

| Product | Flotation time | Product yield $\gamma$[g] | Product yield $\gamma$[%] | Cu content $\lambda$[%] | Cumulat. yield $\Sigma\gamma$[%] | Cumulat. Cu content $\Sigma\text{Cu}$[%] | Cu recovery, [%] |
|---------|---------------|---------------------------|--------------------------|------------------------|-----------------------------|--------------------------------|-----------------|
| K1      | 5 min         | 86.04                     | 16.67                    | 8.62                   | 16.67                       | 8.621                          | 83.50           |
| K2      | 15 min        | 69.37                     | 13.44                    | 1.14                   | 30.10                       | 5.281                          | 92.40           |
| K3      | 30 min        | 30.16                     | 5.84                     | 0.49                   | 35.94                       | 4.502                          | 94.04           |
| OK      |               | 330.7                     | 64.06                    | 0.16                   | 100.00                      | 1.721                          | 100.00          |
| $\Sigma$ |             | 516.27                    | 100.00                   | 1.72                   | 100                          | 1.72                           |                 |

(a) [Graph showing prior and during industrial trial with point of industrial upgrading marked]

(b) [Graph showing prior and during industrial trial with point of industrial upgrading marked]
Figure 1. Laboratory upgrading curves: Fuerstenau (a) and Halbich (b) for flotations conducted in the presence of mixture A

2.2. Results of flotation conducted with mixture B

Table 3. Results of laboratory flotation prior to the industrial trial

| Product | Flotation time | Product yield γ[g] | Product yield γ[%] | Cu content λ[%] | Cumulat. yield Σγ[%] | Cumulat. Cu content ΣCu[%] | Cu recovery, [%] |
|---------|---------------|---------------------|-------------------|---------------|---------------------|---------------------------|-----------------|
| K1      | 5 min         | 80.71               | 16.95             | 8.85          | 16.95               | 8.85                      | 82.77           |
| K2      | 15 min        | 74.5                | 15.64             | 1.13          | 35.39               | 5.15                      | 92.57           |
| K3      | 30 min        | 37.34               | 7.84              | 0.54          | 40.43               | 4.25                      | 94.90           |
| OK      |               | 283.69              | 59.57             | 0.16          | 100.00              | 1.81                      | 100.00          |
| Σ       |               | 476.24              | 100.00            | 1.81          | 100.00              | 1.81                      |                 |

Table 4. Results of laboratory flotation carried out during the industrial trial

| Product | Flotation time | Product yield γ[g] | Product yield γ[%] | Cu content λ[%] | Cumulat. yield Σγ[%] | Cumulat. Cu content ΣCu[%] | Cu recovery, [%] |
|---------|---------------|---------------------|-------------------|---------------|---------------------|---------------------------|-----------------|
| K1      | 5 min         | 74.45               | 17.69             | 8.09          | 17.69               | 8.091                     | 84.79           |
| K2      | 15 min        | 61.36               | 14.58             | 0.83          | 35.28               | 4.808                     | 91.91           |
| K3      | 30 min        | 34.78               | 8.27              | 0.50          | 40.54               | 3.930                     | 94.37           |
| OK      |               | 250.17              | 59.46             | 0.16          | 100.00              | 1.689                     | 100.00          |
| Σ       |               | 420.76              | 100.00            | 1.69          | 100.00              | 1.69                      |                 |
Figure 2. Laboratory upgrading curves: Fuerstenau (a) and Halbich (b) for flotations conducted in the presence of mixture B.

2.3. Results of flotation conducted with mixture C

Table 5. Results of laboratory flotation prior to the industrial trial

| Product | Flotation time | Product yield $\gamma$[g] | Product yield $\gamma$[%] | Cu content $\lambda$[%] | Cumulat. yield $\Sigma\gamma$[%] | Cumulat. Cu content $\Sigma\gamma$[%] | Cu recovery, [%] |
|---------|----------------|---------------------------|--------------------------|------------------------|---------------------------------|----------------------------------|-----------------|
| K1      | 5 min          | 83.56                     | 17.47                    | 8.82                   | 17.47                           | 8.82                             | 84.12           |
| K2      | 15 min         | 76.57                     | 16.01                    | 1.02                   | 33.49                           | 5.09                             | 93.08           |
| K3      | 30 min         | 34.87                     | 7.29                     | 0.52                   | 40.78                           | 4.27                             | 95.15           |
| OK      |                | 283.18                    | 59.22                    | 0.15                   | 100.00                          | 1.83                             | 100.00          |
| $\Sigma$|                | 478.18                    | 100.00                   | 1.83                   | 100.00                          | 1.83                             |                 |

Table 6. Results of laboratory flotation carried out during the industrial trial

| Product | Flotation time | Product yield $\gamma$[g] | Product yield $\gamma$[%] | Cu content $\lambda$[%] | Cumulat. yield $\Sigma\gamma$[%] | Cumulat. Cu content $\Sigma\gamma$[%] | Cu recovery, [%] |
|---------|----------------|---------------------------|--------------------------|------------------------|---------------------------------|----------------------------------|-----------------|
| K1      | 5 min          | 78.71                     | 15.70                    | 8.66                   | 15.70                           | 8.664                            | 85.19           |
| K2      | 15 min         | 68.02                     | 13.57                    | 0.81                   | 29.27                           | 5.021                            | 92.04           |
| K3      | 30 min         | 38.16                     | 7.61                     | 0.51                   | 36.88                           | 4.090                            | 94.47           |
| OK      |                | 316.38                    | 63.12                    | 0.14                   | 100.00                          | 1.597                            | 100.00          |
| $\Sigma$|                | 501.27                    | 100.00                   | 1.60                   | 100.00                          | 1.60                             |                 |
The results of laboratory tests carried out with mixtures of reagents A, B and C before the industrial trial compared with results during industrial trial indicate that the process with a given mixture in a specific period of time was similar, as evidenced by similar upgrading curves. This means that the observed changes in Cu content in the feed have practically no influence on the beneficiation results. In addition, the process efficiency when using different reagent mixtures and specific test periods was characterised by similar indices. The analysis of results obtained in both periods of laboratory tests indicates that the best effects were obtained when using a mixture of reagents A in comparison to other mixtures. On the basis of the conducted research it may be stated that the variability of feed grade as a decisive parameter in the prediction of industrial results does not have a significant impact as long as period of research (3 months).

3. The impact of scaling-up of experiments on results

Differences in upgrading indices between laboratory tests and industrial trials were analysed in next stage of the research. Industrial trials were conducted with the same reagent mixtures (A, B, C) in one of the KGHM production plant according to a regular procedure. During the trial carried out, the dose of the collecting reagent was determined in accordance with the operating instruction specified for this technological circuit. The effect of the applied collector mixtures on the copper recovery was determined. Although all the trial periods were conducted on the same technology line, with a comparable technological regime with the only variable parameter, e.g. the use of different mixtures of collectors, it was impossible to retain the same experimental conditions. Due to process disturbances occurring in industrial conditions, such as fluctuations of throughput and feed grade, each working shift was treated as a separate piece of data. This allowed for the comparison the both the feed parameters and the upgrading results versus time, as well as the determination of the confidence intervals of the selectivity index $a$ on the basis of its variability.

The following equation [5, 6] was used to determine the industrial upgrading curves in the $\varepsilon_r = f(\varepsilon)$ coordinates:

$$
\varepsilon_r(a') = a' \frac{(100 - \varepsilon)}{(a' - \varepsilon)} - 0.07 \left( a' \frac{(100 - \varepsilon)}{(a' - \varepsilon)} - 95.5 \right)^2 \left( 100 - a' \frac{(100 - \varepsilon)}{(a' - \varepsilon)} \right)
$$

where:
- $a'$ is numerically equal to the value of $a$,
- 0.07 - empirical constant of the equation,
- 95.5 - means the point of maximum curvature of the industrial upgrading curve occurring at $\varepsilon_r = 95.5\%$.

The results of industrial upgrading results during trials together with the results of laboratory experiments (before and during industrial trial) are presented on the Fuerstenau upgrading curves, $\varepsilon_r = f(\varepsilon)$ on Fig. 4 ÷ 6.
**Figure 4.** Enlarged part of Fuerstenau curves for flotation with mixture A.

**Figure 5.** Enlarged part of Fuerstenau curves for flotation with mixture B.
Figure 6. Enlarged part of Fuerstenau curves for flotation with mixture C

The industrial upgrading curves in each of the analysed cases (Fig. 4 ÷ 6) show that the point of the greatest convexity, i.e. the point when more useless than useful components start to get into the concentrate, take similar values. Hence it is reasonable to claim that the feed had similar enrichment properties and that the differences between the laboratory curves of the two research periods and the industrial curve for all three mixtures are similar. The swarms of industrial upgrading points indicate some regularities of industrial enrichment in each of the mixtures discussed. The evidence for this is the oscillation their positions around a constant value at approximately $\varepsilon_r = 95.5\%$.

It should be noticed that the variability of enrichment results under industrial conditions is significantly greater than the differences determined in laboratory tests. Moreover, the effects obtained in laboratory conditions were not confirmed in the industrial conditions. In order to analyse the reasons for this phenomenon, Table 7 summarizes the Cu content in the feed and the values of selectivity index $a$, which describes the efficiency of the enrichment process regardless of the content of the useful component in the feed.

Table 7. Summary of selectivity indices of Cu for laboratory flotation and industrial trial.

| Mixture  | Laboratory flotations | Industrial trials |
|----------|-----------------------|-------------------|
|          | $Cu$ content in the feed; $a$ [%] | selectivity index $a$ | $Cu$ content in the feed; $a$ [%] | selectivity index $a$ |
| A        | 1.83                   | 103.43            | 1.60                   | 100.57            |
| Coefficient of variation; $\nu$ [%] | 4.05                   | 0.17              |                      | ±0.10             |
| Confidence interval, $1-\alpha = 0.95$ |                      |                   | ±0.10             |
| B        | 1.81                   | 103.74            | 1.67                   | 100.58            |
| Coefficient of variation; $\nu$ [%] | 4.61                   | 0.14              |                      |                   |
The analysis shows that the differences in enrichment results are poorly correlated with the variability of Cu content in the feed. Additionally, it is confirmed that the variance of shift Cu content in the feed, measured by the coefficient of variation, is at a low and constant level of about 5%. There are no clear correlations and influence of individual parameters on each other. The factors determining the differences in the curves (Fig. 4 ÷ 6) should result from the tested reagent mixtures. The relations between the parameters obtained under industrial and laboratory conditions should be the same for all three cases. This would make it easier to determine the scale-up factors and thus to predict the industrial enrichment parameters.

It was expected that the determined laboratory and industrial enrichment indices, while eliminating feed variability, would allow the determination of the scale-up factor. The lack of correlation between laboratory and industrial parameters indicates that the determination of a simple dependence, enabling the prediction of industrial enrichment parameters, requires more comprehensive approach than the one which has been applied so far. Therefore, the number and variability of enrichment parameters indicate the necessity of searching for the influence on the scaling-up such parameters as: the variability of process with related variability of throughput and grinding times, flotation time as well as differences in mineralogical properties of ore and degree of mineral liberation, pH, Eh and other important process variables [6]. All of these factors and the influence of parameters related to the operation of the processing plant (density of pulp, reagent dosing accuracy, wear of machine working parts) may be the reason why the effect of introducing new reagents is very often unnoticeable or even negative in industrial conditions.

Therefore, the problem of the absence of correlation between the results of laboratory tests and industrial research should be seen in the current methodology of laboratory tests. Such conclusions were also reached by Łuszczkiewicz and others [5]. In their report, they pointed out that the currently practised methodology of laboratory scale research differs significantly from the flotation conditions present in processing plants (backflows, accumulation of disturbing components, continuity of the process, the recycling of industrial water with some impurities, etc.). It appears necessary to develop a methodology for laboratory testing of new flotation reagents taking into account these factors. Therefore, the issue of so-called "scaling-up of laboratory research" requires thorough research and analyses.

The new attitude to prediction of industrial plant upgrading results based on previously executed laboratory experiments should also contain an implementation of gradually scaling up process from laboratory, pilot plant, semi-industrial and finally existing plant technology line. Each step should be observed in order to formulate algorithms of prediction results from obtained results. Such approach should point critical factors having the most significant influence on correctness and precision of scaled-up results. Moreover some attempts should be undertaken to modify laboratory and pilot scale methodology to develop optimised procedure and practise enabling to predict results with satisfactory quality and with approvable costs.

4. Summary

The presented results of both laboratory and industrial scale studies on new mixtures of collecting reagents (A, B, C) indicate that the positive results obtained in laboratory tests are not sufficient to make a decision regarding the introduction of a new reagent for use without unnecessary risk. The reasons for this phenomenon are probably due to the simplification of the current laboratory testing
practices, which do not take into account the complexity of the industrial conditions. Taking into account a limited amount of data from laboratory flotations carried out in an open system (yields with concentrate and tailing grade) as well as Cu content in the feed are the most common data, it should be assumed that the applied methodology is also susceptible to measurement errors of analysis, i.e. determination of masses and contents. Even the execution of multiple flotations with the same parameters does not significantly increase the accuracy of the results.

Further work on finding correlations between laboratory and industrial scale results should be based on a more extensive scheme of laboratory flotation. In addition, it is necessary to take into account previous studies in which the authors suggest that the result of copper enrichment in concentrate may be influenced by other ore minerals (galena, sphalerite, pyrite) as well as base rock minerals [7, 8]. Therefore, when scaling-up the results, it is advisable to investigate the relationship between the main sulphide minerals of copper, associated metals and base rock minerals. The introduction of mineralogical studies may provide a number of information on the interactions between scale-up of individual minerals and process performance after the introduction of new flotation reagents. This methodology should provide a number of necessary information to enable the decision to introduce a new reagent with minimal risk of loses. It should be emphasized that the direction of research may contribute to the increase of production effects and to the reduction of irreversible losses of copper and other useful metals in tailing.

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