A new methodology of floatability tests on Polish copper ores based on grain size-density fractions

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Abstract Ores difficult to enrich require the appropriate methodology of tests on enrichment for the preparation of ore for flotation tests and precise mineralogical analytics. A feature which should differentiate the separated ore samples for flotation tests is not only the size of grains, but also their density. These features reflect the characteristics of real industrial streams directed for flotation. The methodology for separating narrow grain classes by means of sieves applied to date did not guarantee meeting these separation conditions. The article presents a new methodology for examining the copper ore enrichment based on, instead of testing samples in grain classes, testing grain size-density fractions. To conduct these tests, the hydraulic classification in the field of centrifugal force which ensures the separation of grain size-density fractions varied in terms of granulation and density was used. Samples prepared in this way were subjected to flotation tests.

1. Introduction
Flotation is the main method for enriching the majority of metal ores in the world. It is applied for fine-grained mixtures. It uses differences in wettability of mineral grains' surface [1, 2]. Many factors affect the flotation effectiveness, the most important ones include: the size and shape of enriched grains, their mineralogical composition, surface properties, a type and amount of dosed flotation reagents, suspension parameters.

One of widely discussed issues related to the flotation process effectiveness is the effect of the grain composition on its results. Numerous scientific studies raise the issue of the granulation of the feed directed for the flotation process [3-7]. Many researchers indicate that the best results are obtained for the granulation of 20-100μm, while below this value, there is a big problem with the flotation selectivity.

In the case of difficult to enrich non-ferrous metal ores, in which useful minerals are very finely sprinkled and dispersed in gangue, one of the most important directions of technology development are therefore new solutions concerning ores' milling processes to the granulation from the range of single micrometers, but predominantly the development of conditions for the effective flotation with such granulation [7].

Polish copper ore is difficult to enrich, polycrystalline, lithologically variable where separate lithological types differ in enrichment characteristics, additionally, it contains much organic carbon which limits recoveries and the quality of concentrate; it is also fine crystalline, and impregnations and
Concretions occur even in micrometer-sized particles [9]. It is these finest grains that significantly contaminate concentrates and generate losses in tailings.

The technique of preparing samples for flotation tests is very important in order to correctly locate grains difficult to float. In industrial conditions, ore for flotation is prepared in hydraulic classification processes in hydrocyclones, not on screens. Therefore, the feature which should differentiate the separated samples of ore for flotation tests is not only the size of grains, but also their density. The methodology for separating narrow grain classes by means of sieves applied to date did not guarantee meeting these separation conditions.

In turn, the precise qualitative and quantitative mineralogical analysis of minerals' recognition, mineral liberation, type of concretions with gangue, useful minerals granulation which provides representative results is indispensable to correctly characterize grains difficult to float. Currently, there are many techniques available which enable to obtain such a detailed characteristics of these grains. These methods include the quantitative electron mineralogical scanner, QEMSCAN, and the mineral liberation analyzer, MLA [9,10].

2. Materials and methods

The new methodology of feeds preparation for flotation tests in the hydraulic classification process conducted in the field of centrifugal force is proposed in the paper. This was done in the way ensuring the separation of particle size-density fractions not only in terms of granulation, but also according to density. So far, the methodology of separating narrow particle fractions on sieves did not guarantee maintaining these two separation arguments.

For the purpose of distinguishing products obtained by means of both methods a new term of particle size-density fraction was introduced. This is a population of particles of certain equivalent Stokes diameter related to both size and density. It is obtained by means of flow or gravity classification methods. The argument of separation of the feed into size-density fractions will therefore be composed of three factors: density, particle size and resistance of the medium.

Samples of copper ore in different lithological types representing: sandstone, dolomite and shale were subjected to tests. After the initial comminution of each type to the granulation <10 mm, products were secured in vacuum in order to limit the process of minerals' surface oxidation. Directly before each cycle of flotation tests, samples secured in this way were prepared according to the diagram presented in figure 1.

Laboratory tests of ore enrichment in size-density fractions were conducted on ground ore to the granulation of about <160 µm (P99% =160 µm). The diagram of samples preparation was identical for every lithological type. Samples were ground in a standard Bond mill filled with 14.7 kg of milling balls in 80 minutes.

Ore size-density fractions differing in granulation and density were prepared for flotation tests in a multihydrocyclone classifier. From every lithological type, four size-density fractions (HCl÷HClV), which were then subjected to flotation tests, were obtained. The non-classified representative sample (feed) was also floated in a wide grain class 0÷160µm. For the conducted flotation, chemical analyses were conducted for all products: concentrates K1÷K5 and the cell product (tailings).

Flotation tests were conducted in a laboratory machine of the Denver D12 type as 30-minute-long fractionated flotations. The flotation chamber volume was equal to V = 1.5 dm³, slurry density was \(D_S = 1180 \text{ g/dm}^3\). A mixture of xanthates in the amount of 100 g/Mg was applied as a collector, while Corflot added in the amount of 30 g/Mg was the foaming reagent. The feed pH value varied at the level of about 7.7-7.9. Conditions of the process (reagent regime, hydrodynamic parameters) were identical for all flotation tests. Flotation concentrates were collected in time of: 1, 3, 6, 12 and 13 minutes (respectively: K1, K2, K3, K4 and K5). In total, 15 flotation tests were conducted.
3. Analysis of results
Samples directed for the flotation process: lithological types in a wide grain class and size-density fractions of separate copper ore lithological types were subjected to granulometric analyses by the method of laser diffraction. Analyses were conducted in an Analysette 22 device - a laser particle analyzer.

The detailed results of analyses are presented in table 1 and in a form of curves of the grain composition (figures 2-4). The granulation range of floated feeds: of both the wide class and size-density fraction varies depending on the lithological type, which is caused by the different grindability.

Figures 2÷4 present curves of the grain composition of non-classified lithological types and size-density fractions separated from them.

![Diagram of samples preparation for flotation tests](image-url)
Table 1. Averaged results of feeds' granulation for the flotation process.

| Name of sample | $d_{10}, \mu m$ | $d_{50}, \mu m$ | $d_{90}, \mu m$ |
|----------------|----------------|----------------|----------------|
| S_F            | 2.2            | 27.8           | 124.5          |
| S_HCI          | 12.6           | 90.9           | 173.6          |
| S_HCII         | 1.8            | 21             | 55.4           |
| S_HCIII        | 1.2            | 8.9            | 27.8           |
| S_HCIV         | 1.1            | 5.6            | 19             |
| D_F            | 1.3            | 11.4           | 37.7           |
| D_HCI          | 1.6            | 24.3           | 68.8           |
| D_HCII         | 1.3            | 14.3           | 39.1           |
| D_HCIII        | 1.1            | 8.4            | 25.8           |
| D_HCIV         | 1              | 4.8            | 17.4           |
| Sh_F           | 1.8            | 16.6           | 53.4           |
| Sh_HCI         | 2.4            | 35.5           | 103.9          |
| Sh_HCII        | 1.7            | 17.6           | 46.6           |
| Sh_HCIII       | 1.3            | 10.1           | 28.9           |
| Sh_HCIV        | 1.1            | 6              | 20.1           |

where: F - the feed for the multihydrocyclone classifier; S, D, Sh – copper ore lithological type, respectively: sandstone, dolomite, shale; HCl ÷ HCIV – size-density fractions from the multihydrocyclone classifier.

Figure 2. Curves of the grain composition for the feed and separated size-density fractions for sandstone ore.

Figure 3. Curves of the grain composition for the feed and separated size-density fractions for dolomite ore.
Basing on granulometric analyses results, it can be stated that the widest range of granulation is represented by the sandstone ore, while the finest one is the dolomite ore. Grinding conditions were identical in each lithological type; however, individual lithological types differ in grindability and this causes these differences. Products of the multihydrocyclone classifier differ among types in two first size-density fractions: HCl and HCII, which is related to the feed granulation for the classification. Classes: HClIII and HClIV have, on the other hand, characteristic grains which are very close to each other in terms of size, grain d90 in all three types is equal to accordingly 27-29 and 9-10 µm. It is caused by the operation of the classifier which in the first two products separates thicker grains providing products III and IV stable in terms of granulation. Size-density fractions III and IV are ultra-fine classes, separation of which would be extremely difficult in a traditional way, on sieves.

Results of flotation tests of feeds non-classified and size-density fractions for separate copper ores' lithological types in a form of Fuerstenau upgrading curves, kinetics curves (figures 5-7) and in a form of technological indexes are presented in table 2.

**Figure 4.** Curves of the grain composition for the feed and separated size-density fractions for shale ore.

**Figure 5.** The comparison of results of flotation enrichment of sandstone copper ore for the non-classified and separated size-density fractions.
Figure 6. The comparison of results of flotation enrichment of dolomite copper ore for the non-classified feed and separated size-density fractions.

Figure 7. The comparison of results of flotation enrichment of shale copper ore for the non-classified feed and separated size-density fractions.

Table 2. presents the most important qualitative and quantitative indexes obtained in conducted experiments.

| Name of sample | $\alpha$ | $\beta_1$ | $\beta_c$ | $\nu$ | $\epsilon_{c1}$ | $\epsilon_c$ | $\gamma_{c1}$ | $\gamma_c$ | $d_{90}$ µm |
|----------------|--------|----------|-----------|------|----------------|-------------|--------------|---------|-------------|
| S_F            | 0.81   | 12.83    | 3.3       | 0.051| 51.84          | 4.85        | 3.26         | 23.23   | 124.5       |
| S_HCII         | 0.49   | 19.38    | 4.94      | 0.037| 49.51          | 6.89        | 1.25         | 9.19    | 173.6       |
| S_HCIII        | 1.24   | 3.48     | 2.5       | 0.135| 18.7           | 5.78        | 6.69         | 46.77   | 55.4        |
| S_HCIV         | 1.84   | 3.56     | 2.93      | 0.724| 8.32           | 19.39       | 4.31         | 50.66   | 19          |
| D_F            | 0.85   | 4.34     | 1.39      | 0.073| 49.95          | 3.49        | 9.84         | 59.19   | 37.7        |
| D_HCII         | 1.22   | 8.72     | 2.38      | 0.064| 80.95          | 2.63        | 11.32        | 49.97   | 68.8        |
| D_HCIII        | 0.83   | 6.36     | 1.27      | 0.06 | 22.75          | 2.63        | 2.97         | 63.63   | 39.1        |
| D_HCIV         | 0.69   | 3.21     | 1.04      | 0.07 | 18.68          | 3.64        | 4.03         | 64.05   | 25.8        |
| Sh_F           | 0.29   | 2.14     | 0.52      | 0.138| 7.75           | 28.53       | 1.03         | 39.83   | 17.4        |
| Sh_HCII        | 1.22   | 2.1      | 1.56      | 1.16 | 0.74           | 81.81       | 0.43         | 14.19   | 53.4        |
| Sh_HCIII       | 1.44   | 3.57     | 2.86      | 0.87 | 8.1            | 43.56       | 3.26         | 28.38   | 103.9       |
| Sh_HCIV        | 1.13   | 1.75     | 1.66      | 0.42 | 0.75           | 15.41       | 0.49         | 57.97   | 46.6        |
| Sh_HCIII       | 0.99   | 1.57     | 1.34      | 0.55 | 14.87          | 24.78       | 9.38         | 55.51   | 28.9        |
| Sh_HCIV        | 0.87   | 1.61     | 1.1       | 0.64 | 0.94           | 37.67       | 0.51         | 49.24   | 20.1        |
where:
\(\alpha, \beta_1, \beta_2, \nu\) - content of the Cu, respectively, in the: feed, first fraction of concentrate, whole concentrate, tailings,
\(\varepsilon_{c1}\) - recovery of the Cu in the first fraction of concentrate,
\(\varepsilon_t\) - losses of the Cu in tailings,
\(\gamma_{c1}, \gamma_c\) - yield of the, respectively: first fraction of the concentrate, whole concentrate.

In the case of sandstone ore, the best results were obtained for the coarsest class (S_HCI) and slightly worse for the non-classified feed (S_F). A substantial decrease in floatability is observed in the remaining size-density fractions, despite the increasing content of Cu in the feed.

Results of flotation tests of dolomite ore indicate high cleanness of obtained tailings, low contents of Cu in tailings were obtained in each size-density fraction. Only in the finest size-density fraction (D_HCIV), the value \(\nu\) was equal to 0.134% (the lowest one from among all types in this class), while in the remaining four classes, this content was at the level of 0.06-0.07%.

Distinctly the weakest results were obtained for shale ore. For size-density fraction (Sh_HCI), losses of the useful component in tailings were equal to over 43%, with the Cu content in tailings at the level of \(\nu=0.87\%\). Determined qualitative-quantitative parameters for this lithological type of ore indicate large problems with floatability. No satisfactory results were obtained in the conducted tests.

4. Conclusions
The article proposes the new methodology for testing the enrichment of Polish copper ore based on, instead of testing samples in grain classes, testing grain size-density fractions.

The use of a multihydrocyclone classifier enables to prepare and examine the ore enrichment with very narrow ranges of granulation and density. The hydraulic classification in the field of centrifugal force ensures separation of products varied in terms of not only granulation, but also density. These features reflect the characteristics of real industrial streams directed for flotation.

Additionally, thanks to the possibility provided by the device - separation of size-density fractions with specified ranges of granulation and density, it is possible to observe concentration of copper-bearing minerals in separate classification products. Density is related in an obvious way to the mineralogical composition of grains. In the case of carbonate and shale ore, a decrease in the Cu content along with a decrease in granulation is observed; the opposite happens in the case of sandstone ore, in which the copper content in the coarsest size-density fraction is significantly the smallest (0.49%), while the the greatest amount of copper-bearing minerals is accumulated in the finest size-density fraction (1.84% Cu).

The obtained results of tests on size-density fractions confirmed a high effectiveness of flotation for size-density fraction No. 1 (the coarsest grained one and deprived of extremely fine grains) and a gradual decrease in the effectiveness of flotation along with the share of finest grains in size-density fractions. Size-density fractions III and IV (d90 <30µm) were characterized by a sudden decrease in the effectiveness of flotation.

The separation of ore into products with very narrow ranges of granulation and density and the evaluation of density of such size-density fractions provide many opportunities to identify the causes of their difficult enrichment. To that end, further tests on the causes of state of things are necessary.

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