E3S Web of Conferences 18, 01025 (2017) DOI:10.1051/e3sconf/20171801025
MEC2017

Influence of liberation of sulphide minerals on flotation of sedimentary copper ore

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Abstract. Ore liberation is one of the most important parameter in mineral processing, especially in flotation. To separate the valuable components from gangue minerals, it is necessary to liberate one from others. It is achieve primarily through crushing and grinding. These stages are one of the most expensive of mineral production. It is important to determine the adequate mineral liberation which would result in huge savings in the overall cost of flotation plant. The aim of the paper was the analysis of the influence of milling time on the laboratory flotation of the copper ore from stratiform Polish deposit. Three different milling time of copper ore in laboratory ball mill was applied. The flotation results were presented as the recovery-recovery and grade-recovery upgrading curves. The liberation of sulphides and the particle size of sulphides in flotation product were analysed and compared.

1 Introduction

The success of the separation process is related to the size and liberation of feed particles [1-3]. The definition of liberation varies from author to author [4]. The feed can consist of a single mineral called 'free particles', or it may consist of two or more minerals named 'locked particles'. Locked particles can be binary, ternary, quaternary etc., as they consist of two, three, four or more minerals [5]. Liberation of particles is achieved mainly through crushing and grinding of the ore. These stages usually consume 30–50% of overall energy costs of mining operation, but it can rise even to 70% for hard and/or finely intergrown ores. Achieving 'sufficient' liberation can bring huge savings in costs of mineral production [6-8].

Flotation process strictly depends on the particle size. According to Pease et al [9], the finer particles tend to be more liberated, different than coarse particles. Nevertheless, the recovery is reduced for finer and coarser particles. Gaudin [5] as a first extensively described the liberation issue associated with comminution by two means: liberation by size reduction and liberation by detachment. According to the paper, the less abundant mineral is not fully liberated unless the particles are finer than the grain size. Moreover, the more abundant phase is always better liberated than the less abundant phase.

The liberation of the ore is dependent on the mineralogy and texture [4,5,10]. Therefore, the upgrading results of flotation stream depend on the texture of the ore and the particle size to which the processing ore is crushed or milled. In Fig. 1 is shown, how particle composition defines the theoretical grade recovery curve [11]. It is important, that this type of curve do not take account of the process kinetics and mechanical entrainment [10]. According to Cropp et al [11] the losses in tailings are caused mainly by fine and liberated valuable minerals and locked minerals. The reasons of reducing the grade of concentrate are gangue composites, entrained and activated gangue, and deleterious element distribution in various size fractions.

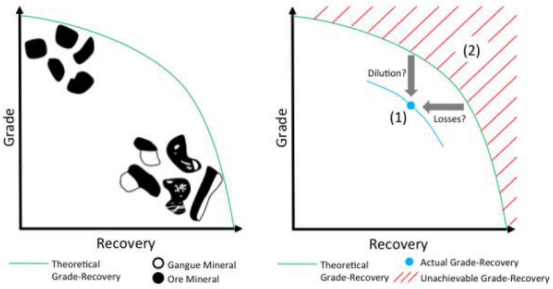

Fig. 1. A schematic of the theoretical grade recovery curve with typical particle images included (left) and the position of the actual grade/recovery relative to the theoretical potential (right); the area '1' – the upgrading can be improved by operational changes; the area '2' – grinding and classification is necessary to improve the upgrading results (based on Cropp et al [11]).

Inadequate liberation is often the main reason of the poor performance. In the literature is a lot of examples, where process mineralogy surveys identified and resolved many problems in the process. The mineralogical and liberation analysis can result in addition of a regrind mill in the circuit or an application of the metallurgical operations in the upgrading stream [10].

The modern mineral liberation measurement techniques can be separated into two main types: two and three dimensional. Two dimensional technique is used in the automated scanning electron microscopy with energy

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dispersive X-ray detectors (SEM/EDS), which popularity and application have been raising since 70s of the twentieth century. Nowadays automated image analysis instruments widely available are QEMSCAN, MLA or EPMA [10,12]. Three dimensional techniques relate to the non intrusive measurements of the whole ore particle (e.g. 3D tomography).

This paper focuses on the influence of liberation of sulphide minerals in flotation of Polish copper ore from the Lubin-Glogow Basin (LGOM, SW Poland), the biggest copper deposit in Europe [13]. To prepare the flotation feed, different milling time of ore was applied. Mineralogical analysis of all upgrading products using QEMSCAN system were conducted. The influence of liberation of copper sulphide minerals in narrow particle size on the upgrading selectivity was analysed by plotting separation curves.

2 Experimental

2.1. Materials and methods

A sample of sandstone copper ore collected from Rudna mining area was investigated. The chemical and mineral composition of flotation feed is presented in Table 1. The sample contains 1.72% of copper. The flotation feed contains 59% quartz, 21% carbonates and 10% clay minerals and micas. The content of all sulphides is about 2.8% (1.3% chalcocite, 0.8% bornite, 0.2% chalcopyrite and 0.3% pyrite). Additionally, the flotation feed contained 0.1% covellite, 0.05% galena and 0.04% sphalerite.

The ore was dry-crushed in the jaw crusher to obtain particles fraction below 1 mm. A sample of 800 g in the presence of 500 cm^3 of industrial water was wet-ground in a laboratory ball mill. The grinding media (stainless balls) filled about 40% of the mill capacity. Three different milling time of copper ore was applied: 60, 80 and 100 minutes.

Table 1. Chemical and mineral composition of flotation feed.

Element/Mineral	Content, %
Cu	1.72
Chalcocite	1.35
Bornite	0.76
Chalcopyrite	0.21
Covellite	0.14
Pyrite	0.26
Galena	0.05
Sphalerite	0.04
Quartz	58.78
Ca, Mg carbonates	21.39
Clay minerals+micas	10.00
Others	5.30

The flotation methodology is presented in Fig. 2. All experiments were conducted in a Denver D12 laboratory flotation machine equipped with 2.5 and 1.5 dm^3 flotation cells. The air flow rate was regulated during each flotation test using a rotameter. Industrial mixture of sodium ethyl and isobutyl xanthate and sodium O,O-diethyl dithiophosphate in proportion 7:3 and the dose of 80 g/Mg was used as a collector. An aqueous solution of Nasfroth was utilized as a frother at a dosage of 20 g/t in all flotation tests. Both reagents were prepared directly before flotation test. After the experimental part, all the products were dried and weighted. A QEMSCAN system was used to determine mineralogy of feed samples and flotation products. The content of copper was determined by using a spectrometric method.

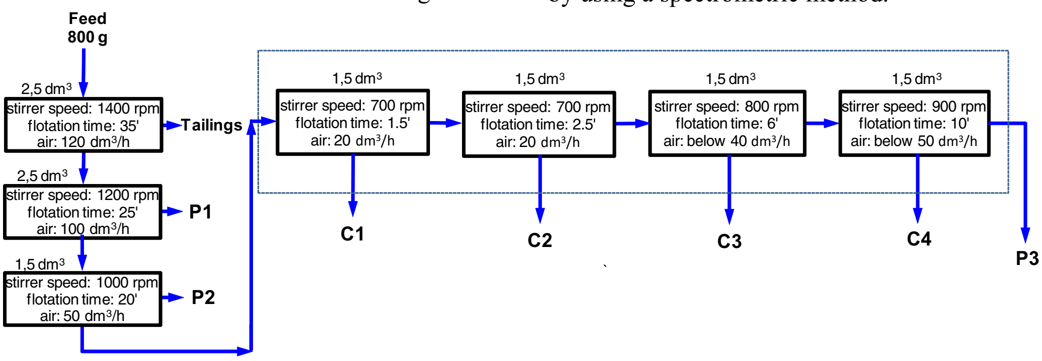

Fig. 2. The scheme of experimental methodology.

2.2 Results

2.2.1 Upgrading results

The results of flotation were evaluated by the mass balance of components and products of separation as well as by plotting recovery-recovery (the Fuerstenau upgrading curve, Fig. 3) and grade-recovery separation curves (Fig. 4).

As it is presented in Figs. 3 and 4, the best and similar results of upgrading of copper sulphides were obtained after 80 and 100 minutes of milling. In these tests the best upgrading selectivity of copper sulphides was observed. It can be assumed that the milling time equals to 80 minutes is sufficient to obtain the highest upgrading selectivity of the examined copper ore.

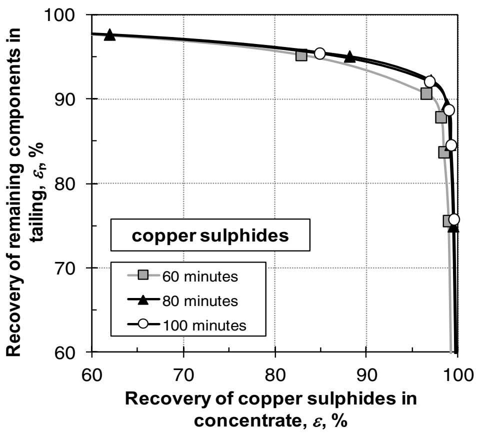

Fig. 3. A recovery of copper sulphides in concentrate vs. cumulative recovery of remaining components in tailing obtained after 60, 80 and 100 minutes of milling.

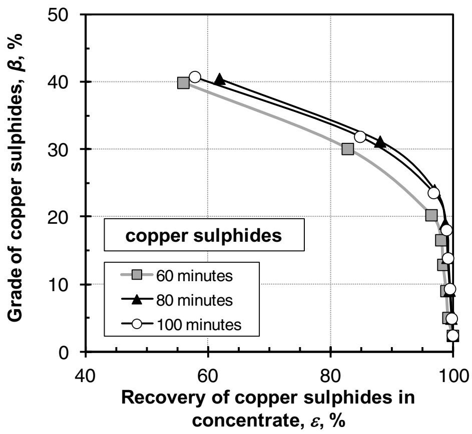

Fig. 4. Copper sulphide grade vs. recovery obtained after 60, 80 and 100 minutes of milling.

2.2.2 Analysis of liberation results

The comparison of the grain composition and liberation of sulphides in the flotation feed is presented in Fig. 5 and 6.

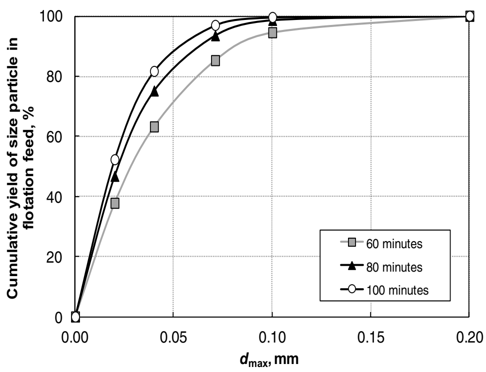

Fig. 5. Particle size distribution in flotation feeds.

As expected, the particle size decreases with increasing milling time (Fig. 5). After 60, 80 and 100 minutes of milling, P_{80} parameter equals to 52 um, 46 um and 38 um, respectively.

In table 2 the liberation of sulphides in the feed after milling is presented. The results of liberation was splited on four ranges: 0–20% (locked particles), 20–50% and 50–70% (impregnations) and 70–100% (free particles). It is important to mark that these data were calculated for sulphides of copper (chalcocite, chalcopyrite, bornite, covellite and tennantite), zinc (sphalerite), lead (galena) and iron (pyrite).

As expected, the liberation of sulphides becomes better with the milling time of the feed decreasing (Table 2). It is noticeable in each grain fraction. The highest of fully liberated particles are observed for particles size in the range of 0–20 um. It is important to note that quite small contents of impregnations are observed in each fraction in feed.

The relationship between liberation of sulphides and milling time is presented in Fig. 6. Similar results of liberation in flotation feeds after milling in 80 and 100 minutes were obtained. A significant differences are observed in liberation of sulphides in feed after 60 minutes and 80 or 100 minutes. It indicates that milling time equals to 80 minutes is sufficient to obtain the maximum of liberation of sulphide minerals in examined copper ore. However, it is important to notice, that this observation concerns in the treatment the ore in a wide size distribution. The liberation of sulphides in narrow grain fractions in flotation feed were analysed (Figs. 7-8).

Table 2. Liberation of sulphides in flotation feed.

Particle size, um	Liberation, %	Milling time of the feed, minutes		
		60	80	100
0–20	0–20	1.77	1.46	1.66
	20–50	3.95	3.38	3.86
	50–70	2.82	2.50	2.80
	70–100	35.88	41.66	45.69
20–40	0–20	1.89	1.62	1.68
	20–50	2.05	1.75	1.73
	50–70	1.61	1.54	1.43
	70–100	21.25	23.34	24.97
40–71	0–20	2.08	1.45	1.17
	20–50	1.85	1.29	1.17
	50–70	1.13	1.04	0.65
	70–100	15.17	14.09	10.88
71–100	0–20	0.92	0.33	0.13
	20–50	0.57	0.34	0.18
	50–70	0.46	0.30	0.18
	70–100	4.36	3.33	1.45
>100	0–20	0.45	0.09	0.02
	20–50	0.10	0.00	0.00
	50–70	0.12	0.00	0.00
	70–100	1.57	0.49	0.35

DOI:10.1051/e3sconf/20171801025

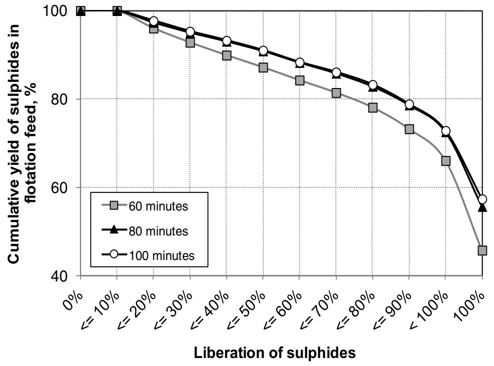

Fig. 6. Liberation of sulphide minerals in flotation feeds.

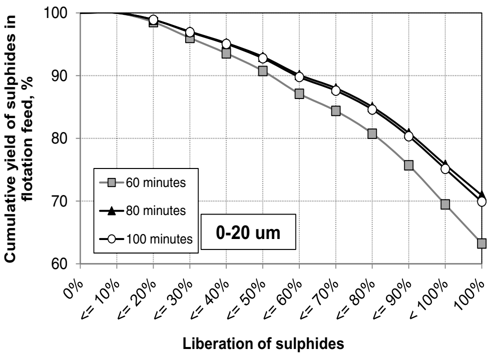

Fig. 7. Liberation of sulphide particles in the size range of 0–20 um in flotation feed.

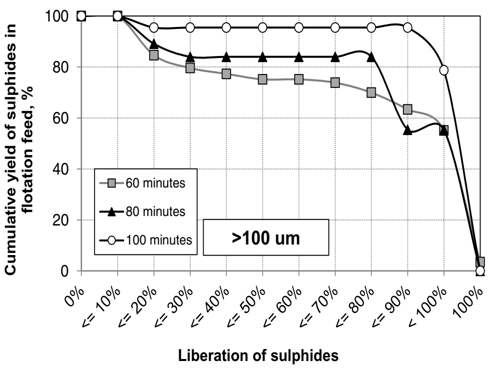

Fig. 8. Liberation of sulphide particles in the size range of >100 um in flotation feed.

As it has been shown, significantly worse liberation after 60 minutes of milling of each grain fraction was observed. It is presented in Fig. 7, that the comminution of sulphides to the size minus 20 um does not provide the good liberation of the fines. Very similar results were observed for sulphide particles in the range 20 to 40 um. Similar liberation for sulphides in the size range of 0–71 um after 80 and 100 minutes of milling was obtained. Probably, the reason of weak liberation of fines is very small dimensions of sulphides, which initially appears in the ore. It can be supposed that the grinding of narrow grain fractions separately can result in better liberation of sulphides, also for fines.

Noticeable differences between 80 and 100 minutes of milling are observed for particle size ranging from 71 to 100 um and >100 um. For the grain fraction >100 um, the liberation of sulphides increases with the milling time (Fig. 8).

The influence of liberation of sulphides was evaluated by plotting grade-recovery and recovery-recovery (α-non-sensitive) upgrading curves. The analysis was conducted for narrow particle size fractions and the liberation degree of sulphides. Only the part of figures is shown in the paper. The curves are presented in Figs. 9-21.

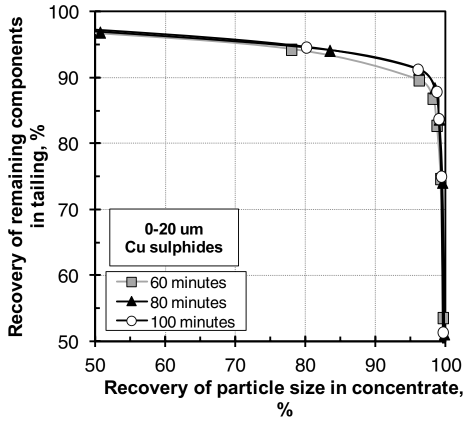

Fig. 9. A recovery of copper sulphides in concentrate vs. recovery of remaining components in tailing in size range of 0–20 um obtained after 60, 80 and 100 minutes of milling.

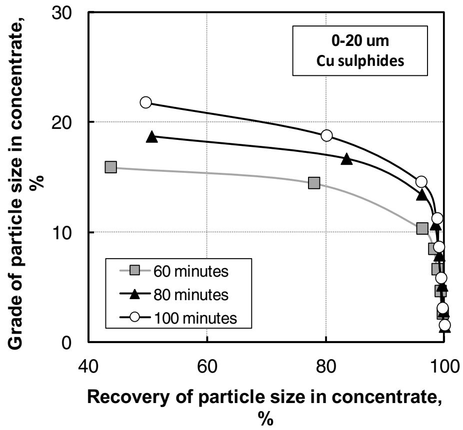

Fig. 10. Copper sulphides grade vs. recovery in size range of 0–20 um obtained after 60, 80 and 100 minutes of milling.

As it is presented in Figs. 9-10, the lowest upgrading selectivity and grade of the finest particles in concentrate is observed in flotation test in which 60 minutes of milling was applied. Similar performance was obtained for the flotation experiments after longer milling time (80 and 100 minutes). Comparable results were obtained for sulphides in the size range of 20–40 um. Since the each flotation feed contains the highest content of grain fraction between 0 and 40 um size (Fig. 3), lower upgrading selectivity of these sulphides size can result in overall poorer upgrading of copper.

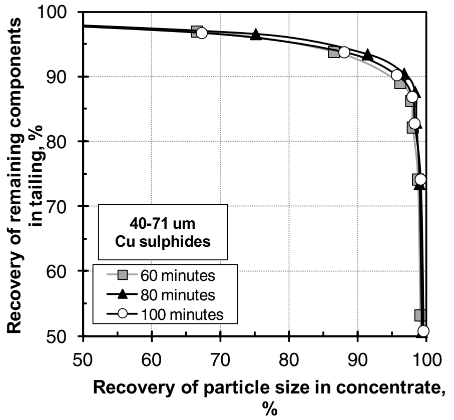

Fig. 11. A recovery of copper sulphides in concentrate vs. recovery of remaining components in tailing in size range of 40–71 um obtained after 60, 80 and 100 minutes of milling.

For the particles greater than 40 um, the differences between three curves become more significant with extending of the milling time (Fig. 11). Similar results for the particles size ranging from 71 to 100 um were obtained. The best upgrading selectivity for copper sulphides was observed after milling time equals to 80 minutes. A bit worse flotation efficiency after 100 minutes of milling was obtained. The lowest selectivity is observed for the shortest milling time. For the sulphide grains greater than 100 um the observation has changed (Fig. 12). It is quite interesting that even for coarse-grain fraction (+100 um) the best upgrading selectivity is obtained for the flotation feed after 100 minutes of milling.

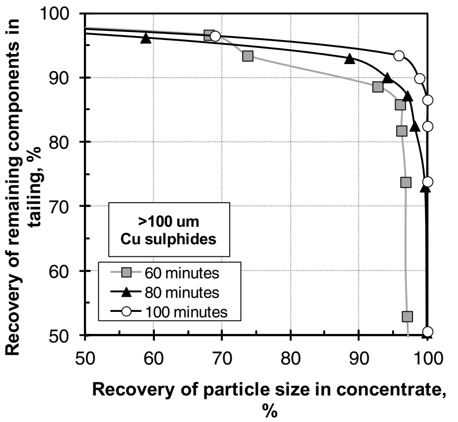

Fig. 12. A recovery of copper sulphides in concentrate vs. recovery of remaining components in tailing in grain fraction greater than 100 um obtained after 60, 80 and 100 minutes of milling.

In Figs. 13-16 the flotation results of free (liberated) sulphides particles is presented. As it can be seen in Fig. 13, the highest and similar upgrading selectivity of sulphides is observed for tests with milling time of 80 and 100 minutes. The lower selectivity was obtained after feed milling in 60 minutes. Similar results were obtained for the particle size in the range of 20–71 um. The comparable dependency is presented in Fig. 14 as grade-recovery curves. The quality of concentrate differs significantly for all tests.

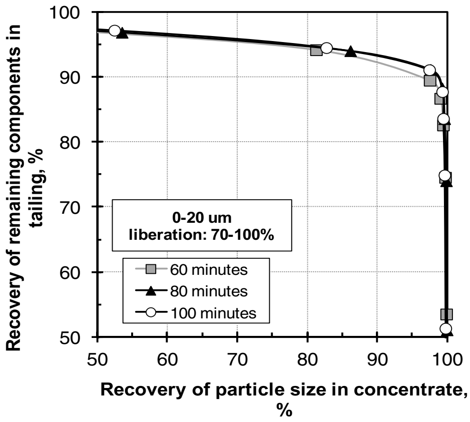

Fig. 13. A recovery of sulphides vs. recovery of remaining components in tailing in size range of 0–20 um liberated in 70–100% (free particles) obtained after 60, 80 and 100 minutes of milling.

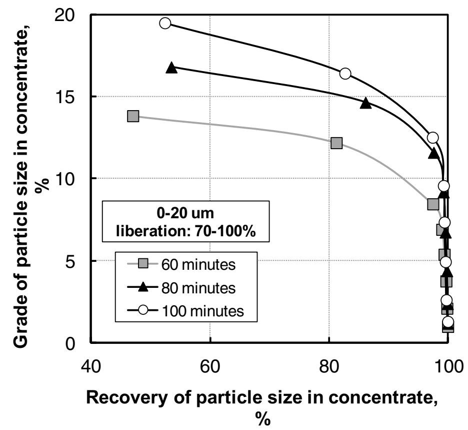

Fig. 14. Sulphides recovery vs. grade in size range of 0–20 um liberated in 70–100% (free particles) obtained after 60, 80 and 100 minutes of milling.

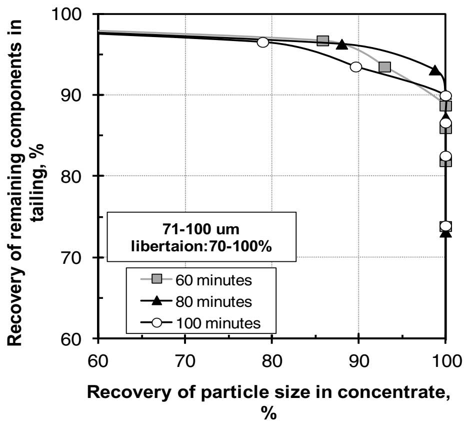

Fig. 15. A recovery of copper sulphides in concentrate vs. recovery of remaining components in tailing in size range of 71–100 um liberated in 70–100% (free particles) obtained after 60, 80 and 100 minutes of milling.

More significant differences are observed with increasing of particle size (Fig. 15). The upgrading selectivity of free particles flotation decrease in order: 80, 60 and 100 minutes of milling. The influence of the particle size on the upgrading selectivity is presented in Fig. 16. It can be seen that after milling time equals to 80 minutes, the flotation efficiency decreases while the size of the sulphides becomes smaller. The obtained results confirms data from the literature. Fines, especially in the size range of 0–20/40 um, float worse than coarse sulphides, even fully liberated. Similar dependences for the rest of flotation experiments were obtained. One of the reason of poor floatability of fully liberated fines can be too less dosage of collector. It is confirmed that fines because of a larger particle surface area demand higher doses of collector than coarse. This issue can be solved by separately upgrading fines from coarse.

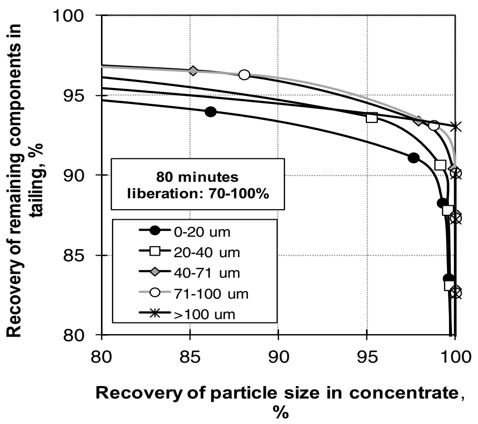

Fig. 16. A recovery of copper sulphides in concentrate vs. recovery of remaining components in tailing for sulphides liberated in 70–100% (free particles) obtained after 80 minutes of milling.

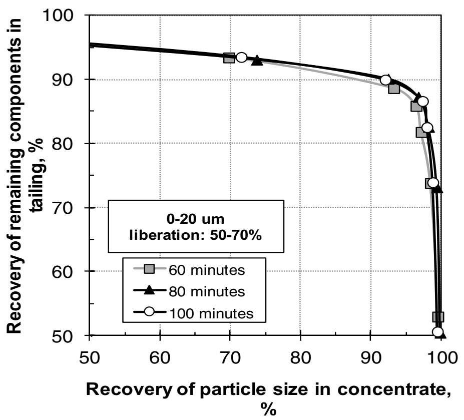

Fig. 17. A recovery of copper sulphides in concentrate vs. recovery of remaining components in tailing in particle size of 0–20 um liberated in 50–70% (impregnation) obtained after 60, 80 and 100 minutes of milling.

Next stage of the research was to study the flotation of sulphides in impregnations (Figs. 17-18). It was observed that there is no significant differences between two ranges of liberation: 20–50% and 50–70%. The results of the sulphides liberated in 50–70% flotation for particle size ranging from of 0 to 20 um and 20 to 40 um were presented. As it can be seen that for the particles size in the range of 0–20 um the best upgrading selectivity was observed after 80 and 100 minutes of milling. Insignificantly worse efficiency was obtained for the test with the feed grinded in 60 minutes.

Appreciably differences between the experiments are observed for particles in impregnations and greater than 20 um (Fig. 18). Similar results for the rest of results, not presented in the paper, were obtained. The poor upgrading selectivity was observed for the flotation test where the feed was grinded in 60 minutes.

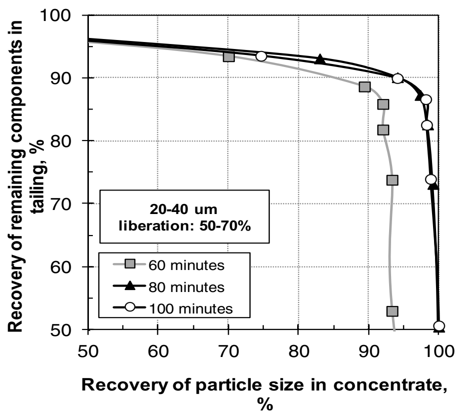

Fig. 18. A recovery of copper sulphides in concentrate vs. recovery of remaining components in tailing in size range of 20–40 um liberated in 50–70% (impregnation) obtained after 60, 80 and 100 minutes of milling.

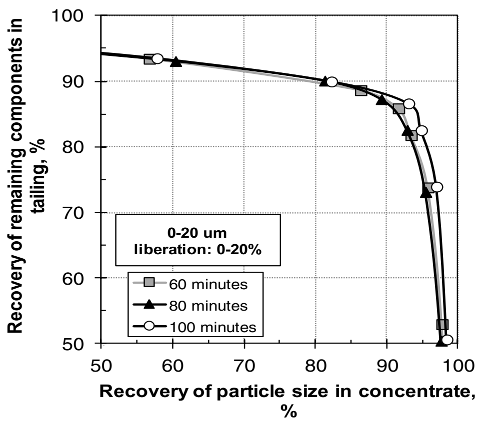

Fig. 19. A recovery of copper sulphides in concentrate vs. recovery of remaining components in tailing in particle size in the range of 0–20 um liberated in 0–20% (locked particles) obtained after 60, 80 and 100 minutes of milling.

In Figs. 19-21 the flotation results of locked sulphides particles (liberation: 0–20%) are presented. As it can be seen, the best upgrading selectivity of fines was obtained in the test with the feed grinded in 100 minutes (Fig. 19). Similar results of flotation were observed for particle size in the range of 20–40 um. For the coarser particles (+40 um) the differences between the plots courses become greater (Fig. 20). Despite the higher content of the locked particles (+40 um) in the flotation feed after 60 minutes of milling time, better upgrading selectivity of these particles was observed.

The influence of the particle size on the upgrading selectivity for the test with milling time equals to 80 minutes is presented in Fig. 21. It can be seen that the flotation efficiency increases while the size of the sulphides decreasing. These results are contrary to date obtained for fully liberated sulphides (Fig. 16).

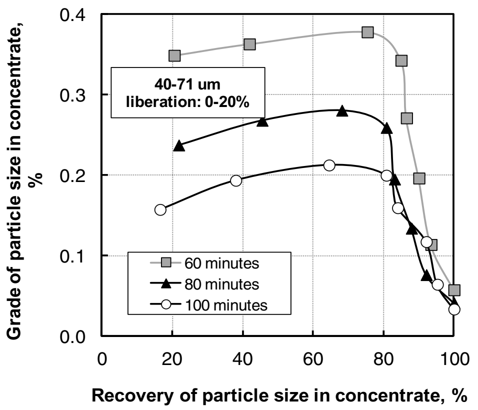

Fig. 20. Sulphides recovery vs. grade in particle size in range of 40–71 um liberated in 0–20% (locked particles) obtained after 60, 80 and 100 minutes of milling.

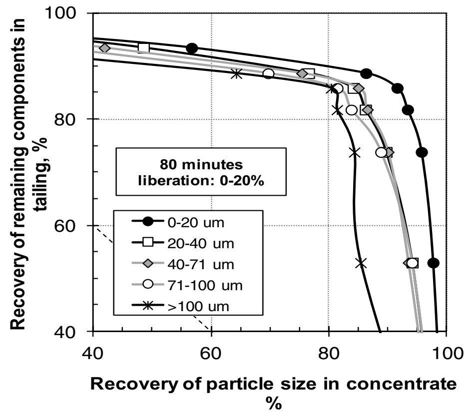

Fig. 21. A recovery of copper sulphides in concentrate vs. recovery of remaining components in tailing for sulphides liberated in 0–20% (locked particles) obtained after 80 minutes of milling.

2.3. Summary and conclusions

The influence of liberation and particle size of sulphide minerals on the laboratory flotation of sedimentary copper ore (Rudna mining area) is presented in the paper. The series of laboratory flotation experiments were performed. All flotation tests were conducted in the presence of collector and frother. Three different milling time of flotation feed were applied: 60, 80 and 100 minutes. Mineralogical analysis of all upgrading products using QEMSCAN system were conducted and analysed.

After 60, 80 and 100 minutes of milling 80% of the particles mass passing 52 um, 46 um and 38 um, respectively. In each flotation feed, the high contents of small particles (0–40 um) were observed. It was shown that the milling time of 80 minutes it is “enough” to obtained the maximum of liberation of sulphide minerals. After 100 minutes of milling the upgrading results were very similar. The milling time equals to 60 minutes is too short for the studied copper ore. Extremely similar observations were notice when it comes to liberation analysis. The comparable data of sulphides liberation for flotation feeds grinded by 80 and 100 minutes were obtained. For all flotation tests, similar results of sulphides liberation were observed for particle size in the range of 0–100 um. For the greater particles more noticeable differences between sulphides minerals in flotation feeds were obtained.

Liberation analysis confirmed upgrading selectivity of sulphides minerals in narrow fractions. The flotation efficiency was close similar for all particles in the size range of 0–100 um in each flotation test. More noticeable differences between all tests were observed for coarser particles. Moreover, even for coarse particles (+100 um) the best upgrading selectivity is obtained for the flotation feed after 100 minutes of milling.

Regardless of particle size and liberation degree, the best upgrading selectivity was obtained for experiments in which the milling time equals to 80 or 100 minutes was applied. Moreover, different dependence was observed for fully liberated and locked sulphides in analysed size fractions. With the bigger particle size, the higher upgrading selectivity of fully liberated sulphide minerals was observed. Quite different results was obtained for locked sulphide particles. The flotation efficiency increases with decrease of size of the sulphide minerals. These observations were noted for each experiment.

It is confirmed that the different flotation needs of fine and coarse particles are essential. A treatment an ore in a wide size distribution can result in poorer upgrading results [14]. One of the reason is i.e. different requirements of collector dosages for fine and coarse particles. Fines because of a larger particle surface area demand higher doses of collector than coarse. Probably, the dosage of collector which was applied in all examined experiments was insufficient for fines while enough for coarser particles. Moreover, flotation kinetics of fines is slower. Good flotation of small but locked sulphides could be the effect of hydrodynamics and low weight of these particles. It can result in mechanical entrainment. Additionally, the flotation of that kind of particles can result in poorer quality of final concentrate.

Concluding, with extending the milling time of copper sulphide ore, the liberation of valuable components becomes better, independently on particle size. In spite of poorer flotation of fully liberated and fine sulphides, deep grinding of sedimentary copper ore is essential to receive the greatest upgrading selectivity. Because of differences in flotation requirements for fine and coarser minerals, it seems to be justified to classification the feed into two or even more streams and processing in a narrow size distribution. The grinding of narrow size fraction separately and then float should result in better liberation of sulphides of each particle size, better overall upgrading efficiency and lower power consumption. This issue requires further investigations.

The research presented in the paper is the part of project titled "Development of highly effective technology of Polish copper ore beneficiation". This project is carried out by the consortium with the Wroclaw University of Science and Technology as leader, agreements No.: NCBR: *CuBR/I/7/NCBR/2015* and KGHM Polska Miedz SA: *KGHM-BZ-U-1023-2015*.

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