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Application of the grade-recovery curve in the batch flotation of Polish copper ore ☆

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ABSTRACT

Ores and raw materials are subjected to mineral processing operations to increase the content of their useful components. Upgrading of these materials can proceed with different selectivity and therefore, there is a need to know and monitor the separation results of the process. The separation results can be evaluated using upgrading tables and curves. The shape of lines of the upgrading curves are usually complex and depends on the graph type and process parameters. The grade-recovery curves are frequently used because are practical and indicate several characteristic features of separation results. Separations by means of batch flotation of several different shale, dolomite and sandstone Polish copper ores were performed along with determination of their mineral composition, copper content, liberation degree, and maximum theoretical copper content in the first concentrate. These data, plotted as the grade-recovery curves, allowed finding other characteristic points such as practical maximum copper content in the first concentrate, maximum curvature which, to a great extent coexists with the liberation degree, maximum recovery of ore valuable components, and the slope of the grade-recovery curve at high recoveries as a measure of liberation of middlings. Correlations between theoretical and practical maximum copper contents in the first concentrate, which depend on the ore type, were also presented. It was confirmed that the ore type significantly influences the shape of the grade-recovery curve and its characteristic points.

1. Introduction

Separation results can be presented either in tabular or graphical forms (Kelley and Spottiswood, 1982). The graphical versions, plotted as quality vs. either quantity or quality of separation products, are known as upgrading curves (Drzymala, 2007a), which are very practical and useful. The course of the upgrading curves is usually complex and depends on the type of the curve and process parameters. General properties of the grade-recovery upgrading plot (Halbich, 1934), which is one of many possible upgrading graphs (Drzymala, 2006, 2007b, 2008), are presented in Fig. 1.

Fig. 1 shows several essential and characteristic points which can be distinguished in the grade-recovery curve. They are: theoretical maximum content of the considered component in concentrate β_t, practical (extrapolated) maximum content of this component in the concentrate β_p, point of the maximum curvature ε_b (break point), and maximum recovery ε_α. Fig. 1 also presents important fixed elements of the grade-recovery curve that is no, ideal, and real separation lines as well as the line of remixing (Drzymala, 2006, 2007b, 2008).

The shape of grade-recovery curve is influenced by the feed grade represented by the content of the useful component in the feed. The advantage of the grade-recovery curve over other upgrading curves is that it relates two essential parameters of separation results, that is recovery and grade which are widely used in industrial (Barskij and Rubinstein, 1970; Napier-Munn, 1998), liberation (Miller et al., 2009, 2012), kinetic (Ball et al., 1970; Xu, 1998) and theoretical (Neethling and Cilliers, 2008) studies.

Different regions can be distinguished in the grade-recovery curve (Fig. 1) and in this paper the curve will be divided into three regions. The first, denoted as I is the region of rich concentrates, in which the concentrate grade is in the vicinity of the maximum theoretical content of a considered component in concentrate (β_t). When the considered component is a mineral, then β_t is equal to 100%. Instead of using concentration of a pure mineral or a group of minerals, the content of a chemical element is very often used in the grade-recovery curves. In such a case, β_t is the content of that

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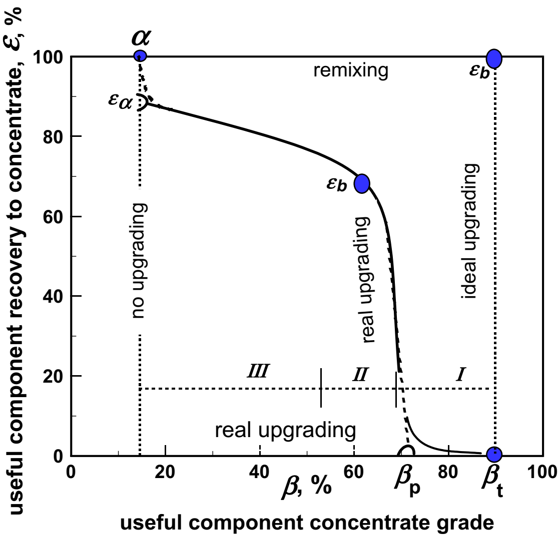

Fig. 1. Properties of the grade-recovery curve. β_p and β_t denote maximum practical and theoretical content of useful component in the first concentrate, respectively; ε_α means maximum recovery, and ε_b is the point of maximum curvature (break point).

element in the mineral. For instance for a chalcocite copper ore the maximum theoretical content is 79.9% because theoretical Cu content in Cu_2S is 79.9%. In many cases, the separation line starts at another point on the grade axis. It is called the practical maximum content of the useful component in the concentrate, or β_p, which is another characteristic point in the grade-recovery curve (Digre, 1960). The second (II, Fig. 1) region is in the vicinity of the maximum curvature of the separation results line denoted as point ε_b. This point is also called the break point. The ε_b point is very important in technological considerations because industrial separations are performed above this position. The third (III, Fig. 1) region of the grade-recovery curve is above ε_b up to the point of the maximum recovery (ε_α), which indicates the percentage of recoverable valuable components in the concentrate.

In this paper we analyze the influence of selected separation parameters on the shape of the grade-recovery curve using different sedimentary type Polish copper ores as the flotation feed.

2. Experimental

2.1. Materials

Geological samples were obtained from different places of the Legnica–Glogow Zechstein Copper Basin located in SW Poland. The Basin contains a copper and other metals underground deposit consisting of there layers: dolomitic (upper), carbonaceous shale (middle) and sandstone (lower). Micrographs presented in Fig. 2 show characteristic structures and compositions of the dolomitic, shale (clays) and sandstone layers. The layers are treated in investigations as different ores even though in practice they are mined together and process non-selectively. Furthermore, the same type of ore originated from different locations are labeled as Lubin, Rudna, and Polkowice ore, after the names of mines and concentrating plants processing the ores from a given geological region. Therefore, the symbols of the ores investigated in this work and given in Table 1 include region (L for Lubin, R for Rudna), ore geological location (G1, G2, etc.), and ore type (P for sandstone, D for dolomite, and L for shale). The ores were crushed in different size reduction devices to obtain particles smaller than 1 mm in size. Next, the samples were wet ground in a complex way to obtain the −0.1 mm feed with size distribution similar to that of the Lubin and Rudna industrial feeds.

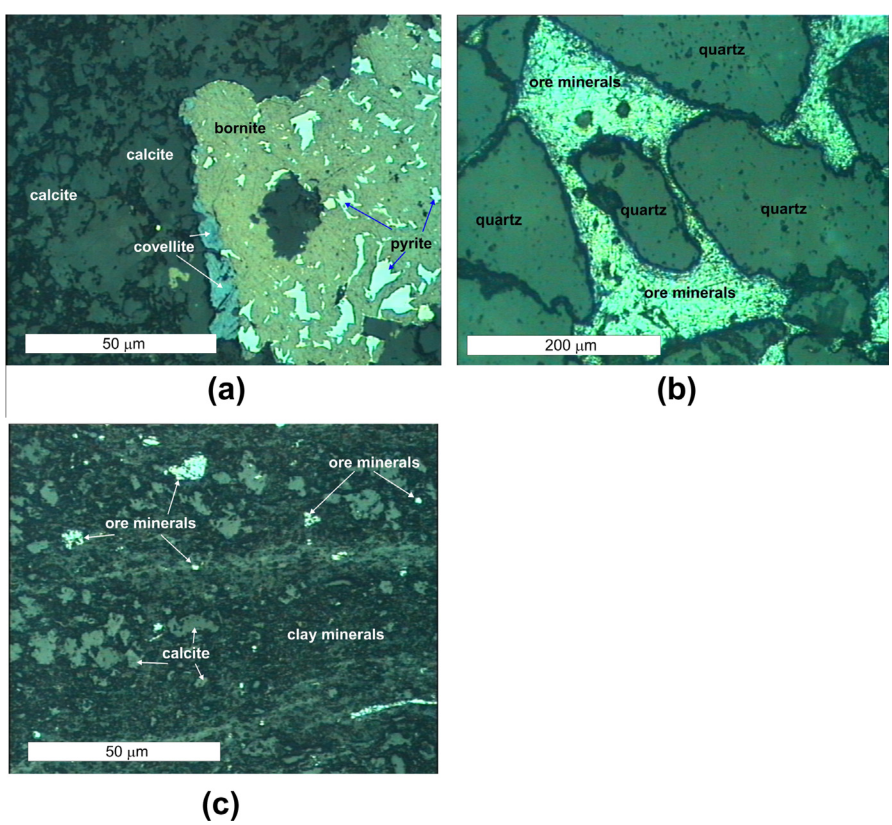

Fig. 2. Microphotographs (reflected light, without analyzer) of the three basic lithological layers containing copper and other metals (ore) minerals and forming copper deposit in Poland: (a) dolomitic (grained texture), (b) sandstone (mortar texture), and (c) shale (schistic texture, clays minerals with carbonaceous matter).

Table 1
Sulfides content *s*, feed grade α, liberation degree L_p, maximum theoretical content of Cu in concentrate β_t, maximum practical content of Cu in concentrate β_p, constants characterizing middlings liberation (k, ε_m), amount of recoverable Cu in concentrate ε_α extrapolated to concentrate grade equal to feed grade, and maximum curvature (break point, ε_b) for the investigated ores. Symbol * means that value of ε_b was not possible to evaluate. Meaning of sample symbols: L-Lubin, R-Rudna location of ore, G-location of geological sample, 1, 2, 3, etc. in the mine, P-sandstone, D-dolomite, L-shale ore.

Ore	Sulfides content, s (%)	α Cu (%)	L_p (%)	β_t Cu (%)	β_p Cu (%)	k	ε_m (%)	ε_α (%)	ε_b (%)
Sandstone ore									
L/G1/P	1.3	0.58	91.6	64.4	31	0.52	87.2	86.9	77
L/G2/P	2.4	0.59	90.5	38.1	21	0.25	92.7	92.5	87
L/G5/P	1.2	0.47	86.9	45.9	29	0.27	87.8	87.7	81
L/G8/P	3.3	0.83	87.2	39.0	21	0.24	95.6	95.4	90
L/G9/P	2.8	0.65	76.7	57.9	16	0.73	96.4	95.9	87
R/G12 + G15/P	3.2	2.02	86.1	77.5	67	0.37	97.8	97.2	88
R/G23 + G27/P	3.7	1.47	91.0	61.9	39	0.29	99.2	98.8	88
R/G1/7 + G3/4/P	4.6	3.31	94.0	76.0	80	0.14	96.2	95.8	87
Dolomitic ore									
L/G1/D	3.3	0.92	76.4	72.6	27	1.24	89.9	88.7	79
L/G5/D	3.5	2.54	82.7	78.9	62	0.41	90.3	89.3	78
L/G7/D	4.7	2.37	78.3	79.6	50	0.57	86.4	85.9	76
R/G12 + G15/D	2.3	0.96	90.8	71.1	17	1.36	92.3	91.0	73
R/G23 + G27/D	0.3	0.09	76.6	76.8	1	15.09	91.5	90.1	–*
R/G1/7 + G3/4/D	0.8	0.11	86.1	69.7	2	20.86	77.4	75.3	65
Shale ore									
L/G1/L	12.8	5.93	87.2	55.6	27	2.41	96.3	82.1	66
L/G2/LD	4.2	2.25	86.0	58.3	8	0.00	100.0	100.0	–*
L/G5/L	13.3	10.31	89.7	77.6	47	1.57	99.3	83.2	66
L/G9/L	4.6	2.53	77.5	63.3	7	1.98	100.0	90.9	–*
L/G7/L	9.0	6.72	80.7	79.5	35	0.00	100.0	100.0	90
L/G8/L	5.0	2.61	75.4	52.8	7	0.00	100.0	100.0	–*
R/G12 + G15/L	16.3	12.56	88.5	77.2	32	0.40	100.0	95.0	–*
R/G23 + G27/L	4.8	1.92	72.6	60.8	4	5.20	100.0	90.0	–*
R/G1/7 + G3/4/L	15.5	10.2	78.8	70.5	26	1.36	100.0	86.1	–*

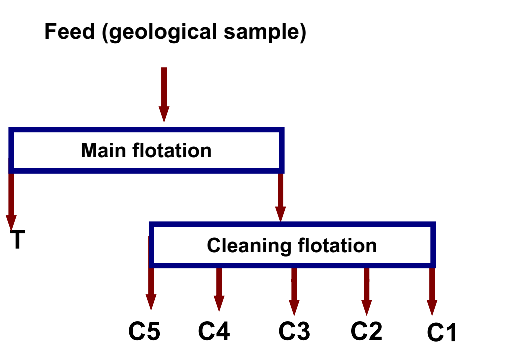

Fig. 3. Scheme of flotation experiments (T – tailing, C – concentrates).

2.2. Flotation procedure

Flotation experiments were carried out in a laboratory Mekhanobr type sub-aeration flotation machine equipped with a 1 dm^3 cell. The same flotation procedure was used for all samples (Fig. 3) while the reagents regime was different for the Lubin and Rudna ores. The applied reagents systems were designed to be relatively close to the ones used in Lubin and Rudna Concentrators. Shown in Fig. 3 flotation scheme was not designed to produce the so-called floatability curves but rather typical laboratory flotation results based on relatively simple tests which can be used for prediction of industrial flotation results by means of special scale enlargement factors (Trybalski et al., 2008). The Lubin ore samples were floated with ethyl + isobutyl xanthate mixed with Flotan (70:30 ratio) using 70 g per megagram (Mg) of the collector. The 20 g/Mg dose of Corflot and Nasfroth frothers, mixed at the ratio of 40 to 60, was applied. In the case of the Rudna ore, the geological samples were floated with ethyl + isobutyl xanthate + Flotan (50:50) using 78 g/Mg and 22 g/Mg of Nasfroth. Due to diminished floatability, the shale samples were floated with an increased by 40%, amount of collector, and increased by 100% amount of frother. The reagents were applied during the main flotation (flotation time 30 min) and cleaning flotation (12 min) (Fig. 3). The flotation products were dried, weighted, homogenized and subjected to chemical analysis for copper.

2.3. Mineralogical analysis

Samples of the feed and flotation products were split into five or six size fractions by wet screening using analytical screens with quadratic openings. The obtained size fractions were dried and weighted to determine their mass for further quantitative balancing of the sample components. Each size fraction of a sample was mixed with resin in a mounting cup to produce embedded samples, which were first surface ground and next polished using diamond pastes. The polished surface of the specimen was subjected to mineralogical analysis using reflected light Optiphot 2-Pol Nikon microscope. The microscopic image of the specimens was subjected to planimetric analysis. Thirty pictures of each size fraction of each flotation sample were analyzed using the Lucia M computer image analysis software. The minerals were diagnosed utilizing their optical properties. The quality, quantity, and liberation analysis were performed using the produced specimens. In the liberation analysis, a free particle was the one having less than 30% of its planimetric surface area filled with gangue minerals such as clay, carbonates and quartz. The liberation degree L_p was defined as a percentage of free copper minerals in relation to all particles containing copper minerals.

3. Results and discussion

The results of flotation of different copper ores plotted as the grade-recovery curves with indicated characteristic points such as theoretical (β_t) and practical (β_p) maximum contents of Cu in

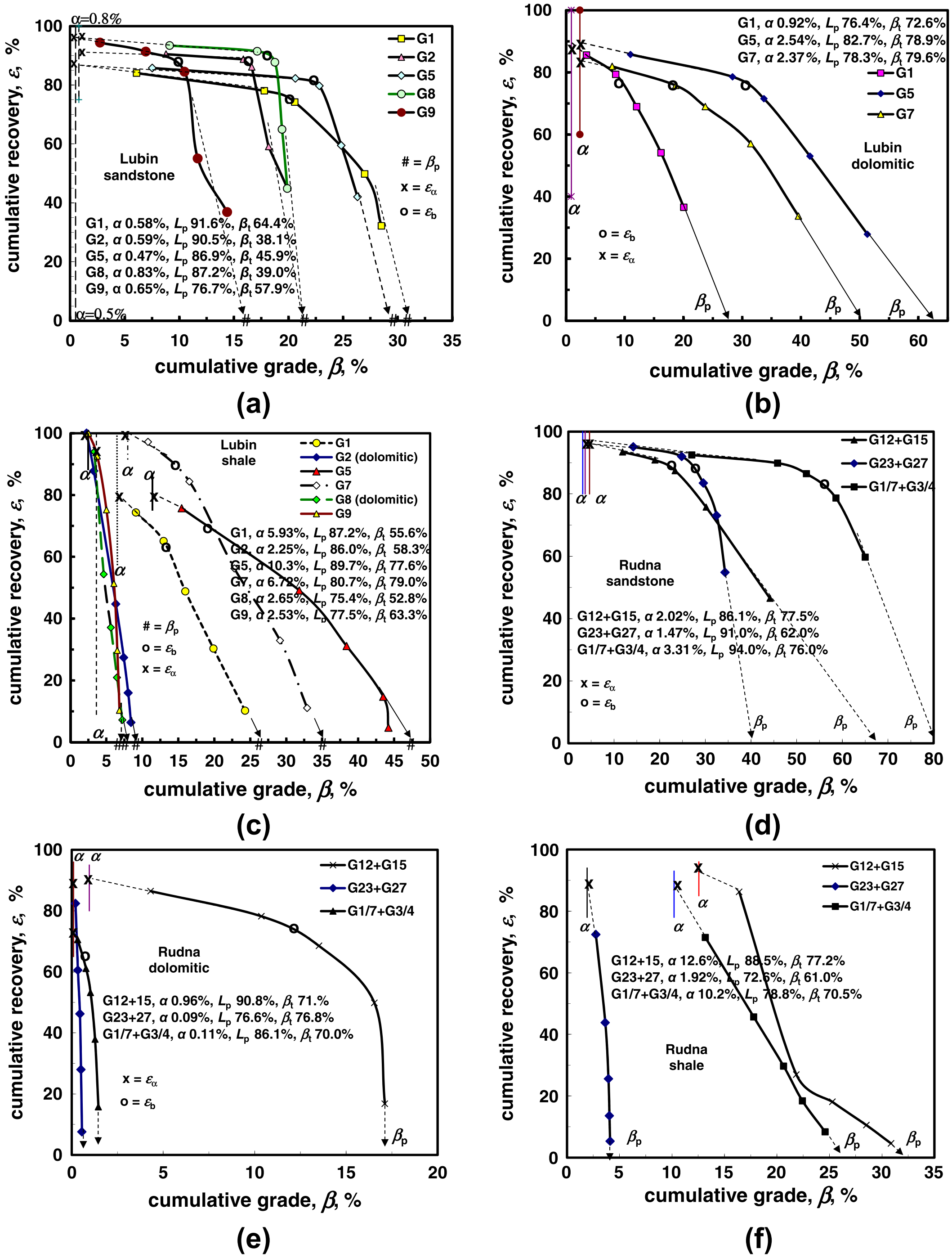

Fig. 4. Concentrate grade (% Cu) –recovery of Cu to concentrate (in%) upgrading plots for Lubin: (a) sandstone, (b) dolomitic and (c) shale ores, and for Rudna: (d) sandstone, (e) dolomitic and (f) shale ores. Symbols are explained in Table 1.

the concentrate (grades), maximum curvature (break point, ε_b), liberation degree (L_p), and maximum Cu recovery to concentrate (ε_α) are presented in Fig. 4a–f and Table 1. Since six products of flotation were produced during each experiment, the results are plotted as cumulative grades (Cu contents in concentrate) vs. cumulative recoveries of Cu to the concentrate by cumulating the products starting from the richest in Cu.

3.1. Region I. Rich concentrate

In batch flotation the most hydrophobic particles tend to report first to the concentrate. The most hydrophobic are the fully liberated particles of the useful component of the feed. If only fully liberated particles would float, the content of the considered component in the concentrate, that is grade, would be equal to the maximum theoretical content (β_t). However, partially liberated particles also slightly float, and there is always some entrainment of hydrophilic particles. As a result the content of the considered component in the first concentrate in the batch flotation is not equal to the maximum theoretical content, but is lower. It is called the maximum practical content of the considered component in the first concentrate (β_p) and can be determined by approximation to zero-recovery. It is usually determined by a simple extrapolation using the steepest part of the grade-recovery curve (Fig. 5). The

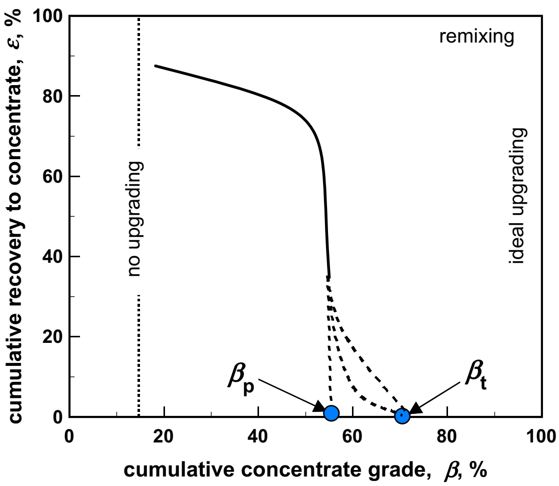

Fig. 5. Possible shapes of the upgrading lines between the maximum theoretical (β_t) and practical (β_p) contents of a useful component in the first concentrate plotted as the grade-recovery curve.

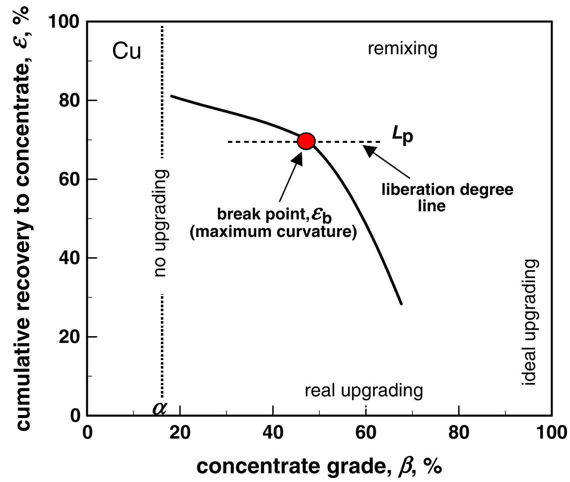

Fig. 7. Maximum curvature (break point) ε_b and liberation degree L_p. The break point is expected to occur in the vicinity of the liberation degree.

upgrading line between the maximum theoretical and practical content of component can assume different shapes as is shown in Fig. 5.

The values of the maximum theoretical (β_t) and practical (β_p) Cu contents for the investigated copper ores are given in Table 1, while the maximum practical content of Cu can also be seen in Fig. 4. To see how the maximum practical content is influenced by the maximum theoretical content a correlation between these two variables for the investigated samples is presented in Fig. 6. It can be seen that the correlation is influenced by the type of the ore. In addition, as expected, the maximum theoretical content is always greater than the practical ones. For the sandstone copper ores, the difference is small, while the dolomitic and shale copper ores provide very low values of the maximum practical content of Cu regardless of the theoretical. Low values of β_p for the shale ore result from enhanced flotation of shale particles consisting of carbonaceous matter, clay minerals and certain amount of copper sulfides. Only at very high values of β_t the values of β_p are also high. Fig. 6 indicates that at sufficiently high liberation (all samples were well liberated), the type of material seems to have the greatest influence on the difference between the maximum theoretical and practical content of Cu.

3.2. The central region (II) of the grade-recovery curve

The central region of the grade-recovery curve contains the maximum curvature of the upgrading line also called the break point (ε_b) (Figs. 1 and 7). In region II the degree of liberation of useful component in the feed L_p is a very important parameter because it limits how far the upgrading line goes steeply up. Later on, due to lack of fully liberated particles in the feed, the middlings start to dominate the floating material. This is the phase of remixing of the floated-up valuables with the newly floating middlings and gangue minerals. Therefore, a relationship between the liberation degree and the recovery at point of maximum curvature ε_b is expected to occur. Such a relation is presented in Fig. 8. It shows that for the investigated ores ε_b is nearly equal (sandstone and Lubin dolomitic ore) or smaller (Rudna dolomitic and Lubin shale ores) than L_p. A lack of smaller values of liberation degree L_p enables to check possible correlation between ε_b and L_p.

3.3. Region III. High recoveries

Dell (1969) using the grade of useful component vs. recovery curve noticed (Fig. 9) that industrial separation results tend to form

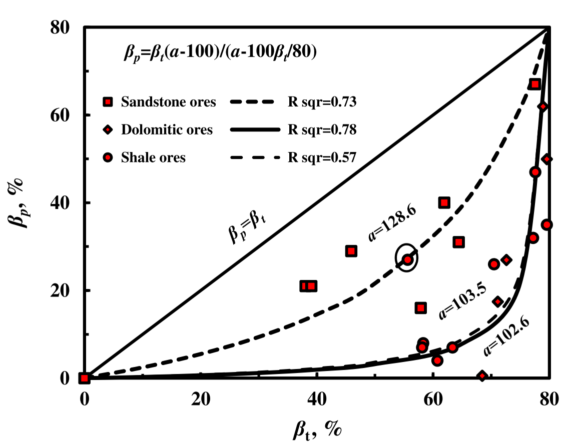

Fig. 6. Correlation between maximum theoretical (β_t) and practical contents (β_p) of Cu in concentrates for the investigated three ore types. The expression $\beta_p = \beta_t(-a-100)/(a-100\beta_t/80)$ was fitted to the data by a non-linear least squares regression using a Sigma Plot software package. In the equation a is a fitting parameter and 80 denotes the percent of Cu in chalcocite. R^2 denotes determination coefficient. O-point not taken into account.

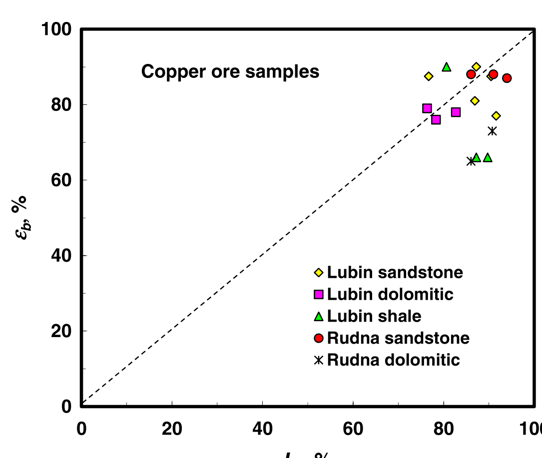

Fig. 8. A relationship between recovery at the maximum curvature point ε_b and liberation degree L_p for the investigated samples.

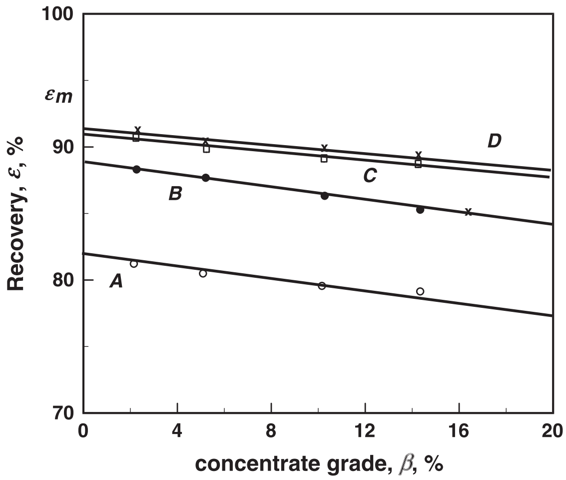

Fig. 9. Industrial flotation data for Luanshya ores reported by Dell (1969).

straight lines in the area of high recoveries. The lines are represented by a general equation:

$$\varepsilon = \varepsilon_m - k\beta \tag{1}$$

where k is a constant characterizing middlings liberation, while ε_m is a measure of the amount of the considered useful component which is upgradeable. This parameter is read off the grade-recovery curve at the concentrate grade equals to zero. According to Dell (1969) the slope k of the line can be used as a measure of liberation. In his approach k equal to zero means perfect liberation while k equal to infinity indicates no liberation. The grade-recovery curves in region III for our samples, as shown in Figs. 4 and 10, also follow this pattern.

Small slopes of the upgrading lines in region III result from a high liberation degree of the copper minerals in the feed making the location of the point of maximum curvature ε_b high on the recovery scale. As a result, the straight line from the vicinity of the point of maximum curvature to maximum recovery ε_α is not very steep. Of course, the distribution of the liberated middlings also influences the upgrading line in region III. It should be noticed that sometimes it is difficult to draw a straight line in region III, especially in the case when the maximum curvature point is difficult to determine as in the case of some shale copper ores, even though the liberation is reasonable. This is due to the fact the starting point of the upgrading curve is at low concentrate grade values resulting from a low value of the practical maximum contents of the useful component in the first concentrate β_p, caused by a relatively good flotation of organic carbon compounds present in the shale copper ore.

It should be also noticed that during flotation, the minimum content of the useful component which can be achieved in cumulative concentrate, is equal to the content in the feed (α). Therefore, a more precise relation between recovery ε and content β is:

$$\varepsilon = \varepsilon_\alpha - k_x(\beta - \alpha) \tag{2}$$

where k_x, as in Eq. (1), is the measure of middlings liberation while ε_α is the extrapolated maximum recovery The values of k (not recalculated into k_x) and ε_α are given in Table 1.

3.4. Separability and other aspects of the grade-recovery curve

The grade-recovery curves shown in Fig. 4 are based on laboratory tests. They can be used as a guide for expected industrial flotation results or for predictions based on special scale enlargement factors obtained by comparing laboratory and industrial separation results for the same material (Trybalski et al., 2008). It seems that more useful can be the so-called separability or upgradability curves which are based on image analysis techniques, including the Mineral Liberation Analysis (MLA) (Solomon et al., 2011; Kappes et al., 2009) or special separation methods such as the tree (Nicol et al., 1983), release analyses of Dell (1953, 1964) and their numerous modifications (Randolph, 1997). The upgradebility curves have different, more convex, shapes from these given in Fig. 4. They are usually located between the ideal and real upgrading lines (Figs. 1 and 11), and usually have a specific shape because they start at the recovery greater than zero and reach 100% recovery at concentrate grade β greater than the feed grade α (Fig. 11). According to Finch and Gomez (1989) the characteristic points of the upgradeability curves reflect the liberation of useful component in the feed. As can be seen in Fig. 11, the points are called coefficient and degree of liberation. The influence of liberation on the liberation-limited grade-recovery curves for specific feed material based on 3D mineral liberation analysis was presented by Miller et al. (2009).

Both upgrading and upgradability curves plotted in the grade-recovery curve can be approximated with different mathematical equation starting with simple multiparameter polynomials and

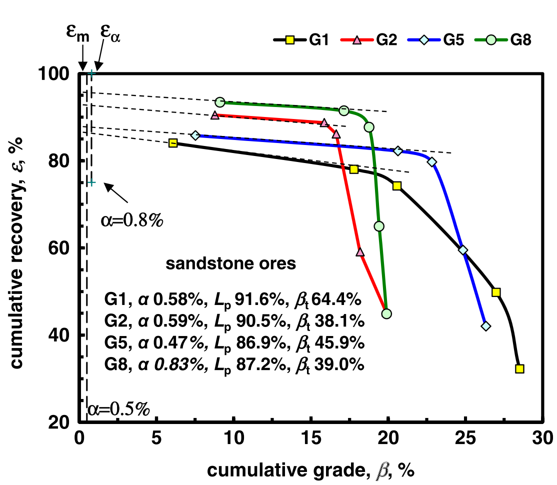

Fig. 10. Laboratory flotation data for Polish copper geological samples of sandstone ores showing region III of the grade-recovery curve with linear approximation of data and characteristic ε_m and ε_α points.

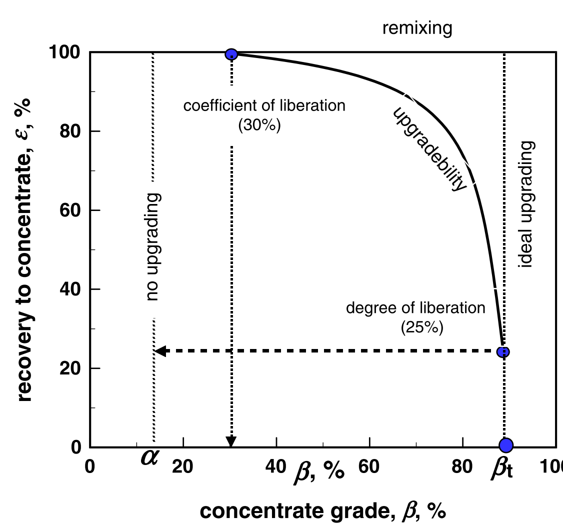

Fig. 11. Possible elements of the upgradability grade-recovery curve (after Finch and Gomez, 1989).

ending with more sophisticated formulas. The most useful are those which contain only one adjustable parameter which can be used as a measure of selectivity of separation. For this purpose Duchnowska and Drzymala (2011) proposed the following equation:

$$\beta = \frac{100\alpha(\varepsilon - A)}{100^2 - \alpha(100 - \varepsilon) - 100A} \tag{3}$$

where β stands for concentrate grade, ε recovery, α feed grade, and A is the separation selectivity factor. Vera et al. (1999, 2000) used another equation:

$$\varepsilon = \varepsilon_\alpha - a\sinh\left(b\left[\frac{\beta}{\alpha} - 1\right]\right) \tag{4}$$

where ε_α denotes maximum recovery, a and b are fitting parameters and sinh means hyperbolic mathematical function. In Eqs. (3) and (4) α, β and ε are expressed as a percentage.

4. Conclusions

The course of the grade-recovery curve is complex and determines several characteristic regions and points. The upgrading starts in region I with the maximum theoretical content of valuables in the first concentrate because the first floating particles are expected to be fully liberated. However, due to some co-flotation of gangue and partially liberated particles, the upgrading line starts from a point which is called the practical maximum content of the considered component in concentrate. In was established in this work that the value of the maximum content of Cu in the concentrate for the investigated samples depends mostly on the type of the ore. In region II, the upgrading line reaches the point of the maximum curvature also called the break point. This characteristic recovery (ε_b), when compared with liberation degree (L_p) for the investigated ores, is roughly equal to (sandstone ore) or smaller (shale ore) than L_p. Smaller values of the maximum curvature than liberation degree may also partially result from the adopted definition of the liberated particle based on the "up to 30% of gangue in the useful particle" formula. More tests with lower degree of liberation particles in the feed are needed to establish a more precise relation between maximum curvature and liberation degree.

A further upgrading of the ores becomes difficult due to incomplete liberation of the remaining minerals in the feed. Very often the grade-recovery curve in region III can be approximated with a straight line as it was proposed by Dell (1969). The parameters which influence slope k in region III are the liberation degree of middlings, distribution of not fully liberated particles, and the presence of non-floatable valuables. The upgrading line should finally reach the point of 100% recovery. However, this usually does not happen because there are some fully or partially liberated particles which do not float due to, for instance, excessive size. They cause an incomplete recovery of the useful components. This point is called the maximum recovery ε_α and is the extrapolated value of recovery to concentrate grade equal to feed grade. The values of k and ε_α were determined for the investigated ores but they were not dependent on the ore type. Observed for some ore samples very small values of the maximum recovery suggest a need for an additional treatment (finer grinding, longer flotation, etc.) of the middlings.

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