USE OF VIRTUAL FRACTIONS FOR MLA OF Y-BEARING REE ORES

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ABSTRACT

Ores enriched in critical rare earth elements (CREE) keep on being of particular interest. The CREE are Nd, Eu, Tb, Dy, including Y. In conjunction with beneficiation tests on Y-mineral bearing flotation tailings of complicated mineralogy, the Mineral Liberation Analyses (MLA) was used to work out processing characteristics fraction-wise in terms of modal mineralogy, mineral recovery, locking and liberation, and particle size.

Size fractions from mechanical sieving (“real fractions”) as well as fractions created virtually by using filters on the MLA-data of the unsieved head material (“virtual fractions”) were investigated comparatively thereby. The "virtual-sizing-approach" features a much simpler sample preparation procedure and considerably lower cost if a fraction-wise investigation of a head sample is needed. It can be conveniently applied to intimately locked ores needing a fine grinding to a particle size distribution (PSD) range below 25 microns for instance. However, its practical use is sometimes disputed in terms of the reliability of the results.

Results are shown for the main Y-bearing mineral xenotime and selected main gangue minerals of a low grade REE-ore.

The influence of sample representativeness, sample preparation and particle counting statistics turned out to be of crucial importance. Given that all preconditions for reliable results are met, locking characteristics, modal mineralogy, and even PSD trends can be read from virtual fractions.

The authors encourage processing engineers to use the virtual approach. They propose practical measures to improve the reliability of the MLA results, identify and tackle conflicting results, and mitigate evaluation problems that may arise.

KEYWORDS

Critical rare earth elements, automated mineralogy, mineral liberation analysis, xenotime, yttrium ore beneficiation, low-grade ore, Virtual and real screening, particle count statistics, gravity pre-concentration, gravity-made segregation.

INTRODUCTION

Any reliable fraction-wise evaluation of mineral processing results by automated mineralogy applying the mineral liberation analysis (MLA) for instance, usually is done on size fractions of the due products. Thereby, particularly when doing beneficiation tests on finely locked and low grade ores, the preparation of these fractions by mechanical screening in the size range of 5 to 20 microns is not a routine procedure.

Facing problems of fragile sieve wires and time consuming laboratory work to produce enough material for polished sections of each fraction, processing engineers have been searching for a simplified methodological approach that is simultaneously cutting down the cost for mineralogy caused by a multitude of size fractions. Thereby, any sizing would be done exclusively on a "virtual" basis applying due MLA software size filters to unsieved head samples.

But, this approach is in dispute. Petruk (2000) generally comments that a virtual sizing on unscreened samples would "not be as accurate as those obtained by analyzing sieved fractions, but would be adequate for routine analysis related to processing". Greet (2013), working on a gold ore, recommends not to use a virtual sizing that may deliver biased liberation data leading to an oversized milling step. A recent study done by Lastra and Petruk (2014) shows, that unsieved samples can be used for comparative studies around processing nodes.

In this paper, the authors intend to figure out the differences between the mineralogical results of the "real screening" and the "virtual screening" approach by comparative investigations applying the MLA to a low grade Y-minerals bearing ore and its fractions.

From the results presented in this paper, own conclusions are supposed to be drawn as to the reliability and applicability of the virtual screening on unsieved samples as an easier way of fraction-wise investigation.

Selected problems of this cost saving preparation way that may arise, will be highlighted and practical recommendations for recognizing and tackling conflicting results as well as mitigating evaluation problems will be developed.

METHODOLOGY

For the tests, a low grade REE-ore that is showing a considerable Y-proportion with 0.2 to 0.3 wt% Y_2O_3 was used. The mineralogy of the samples is characterized by xenotime (Xtm) and Y-fluorite as valuables totaling to a mineral grade of about 0.4 to 0.7 % with a gangue matrix that is dominated by various silicates like quartz and zircon.

An idea about the first step of the investigation procedure can be seen from the following flow chart (Figure 1). Thereby, two head samples were prepared by sample splitting and sieve classification. The first one was an original flotation tails sample (called "-500 feed") that was used for beneficiation tests. The second sample was a coarse screen fraction +125 microns of the first head sample treated by dry regrinding in a screen discharge ball mill and called "+125 regrind".

From both samples, screen fractions ("real fractions") as well as unscreened material was used for the preparation of polished sections for the automated MLA subsequently.

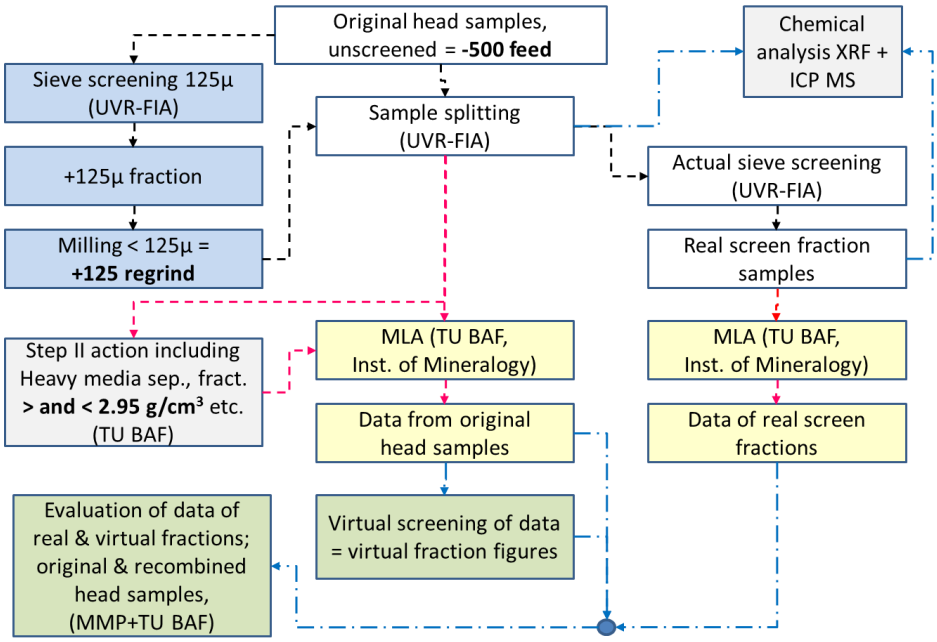

Figure 1 – Simplified flowsheet of sample preparation and methodology of step I and II work

On the data produced by MLA, a comparative evaluation of "real" fractions (mechanically screened) versus "virtual" fractions (on unscreened feed) focused to the main Y-mineral xenotime was carried out afterwards.

When evaluating the first step results, the following challenges were faced partly:

- Uncertain particle count statistics by too few xenotime grains in virtual size fractions from unsieved head samples on one hand and,
- Obvious deviations between results from real and virtual size fractions on the other hand.

Hence, a second investigation step was set up and carried through to figure out the reasons of seemingly biased and uncertain results respectively, comprising the following measures:

- Heavy media gravity separation of the "-500 feed" applying the MLA to both the light and heavy mineral fraction separately to improve the counting statistics of xenotime,
- A second section ("second cut") made on the grain mound from the original "first cut" of the sample "+125 regrind" to check if any bias could have been caused by gravity-made segregation of minerals according to its specific weight and particle size when preparing the MLA images,
- A repeat of the preparation and measurement of both feed samples to check if any bias was caused by sample splitting (particularly on sample "+125 regrind").

The heavy media separation was done at the Institute of Mechanical Processing Engineering of TU BAF using sodium-polytungstate at a density of 2.95 g/cm^3. All mechanical screening was carried through at UVR-FIA GmbH Freiberg on a Retsch AS 200 Rotap machine with DIN sieves of 32; 63; 90; 125; and 160 microns sieve aperture.

All produced polished blocks of screen fractions and the unscreened input were subject to an MLA image analysis conducted at the Geometallurgy Laboratory of the Institute of Mineralogy of the TU Bergakademie Freiberg/Saxony (TU BAF). As software, the MLA-Suite 2.907 of FEI with a standard-data set of EDX-Spectra was used. For affiliation of EDX-Spectra to minerals, the MinIdent, Webmineral, IMA-Mineral list and own EDX analyses were applied.

For any chemical assaying on feed and real fraction samples, XRF and ICP MS were applied to analyze the content of rare earths elements (REE) and Yttrium among others.

RESULTS AND DISCUSSION

The first step interim results will be displayed together with the results of step two investigations comprising a heavy media gravity separation of the "minus 500 microns" head sample, a deeper going second cut of the original first cut section of the "+125 regrind", and a repeat of the complete sample preparation and MLA measurement of new subsamples of both feed materials (see chapter Methodology).

Thereby, conflicting results and deviations as far as they occurred will be put to the focus in favor of non-confliction ones. Investigations that were conducted to overcome and mitigate those problems respectively will be highlighted.

Insufficient Xenotime Particle Count Statistics of Sample "-500 feed"

From the results of step one of the first head sample "-500 feed", a fairly good agreement in terms of the mineralogy and PSD numbers of real and virtual screening could be stated (see Figure 2). A slight difference partly could be found when comparing the "virtual" PSD curves to the one on the real size fractions from sieved samples (real screening). Best fit with real fractions in terms of PSD was stated for the virtual recombination of the gravity separation products.

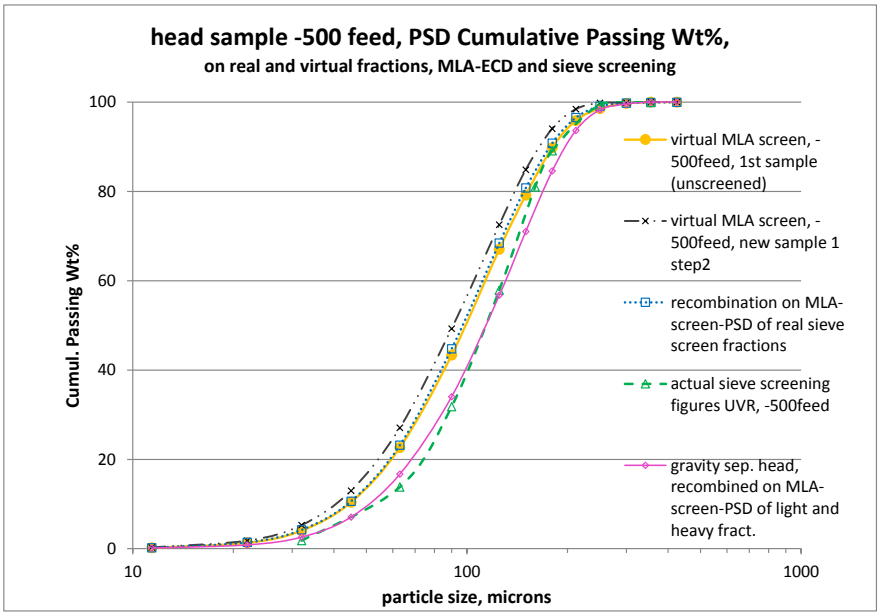

Figure 2 – Comparison of PSD from actual sieving and virtual screening (-500 feed)

However, in case of the mineral locking characteristics, statistical problems occurred for the virtual fractions that were obviously caused by low particle count numbers for minerals like xenotime featuring a low feed grade of about 0.7 weight-% or less.

In the next figure (Figure 3), an overview of the particle counts of xenotime for all investigated samples and fractions is shown in comparison to a minimum particle count limit number *N* of 25. This number *N* was calculated following an equation used by Lamberg (2007):

$$N = (100/CV)^2. \quad (1)$$

Representing the minimum count number of a particle bin of an MLA image, *N* is chosen here to be large enough to ensure a variation coefficient CV of 20 % (Lamberg, 2007).

As can be seen clearly on the left side of the graph, all the head samples (sieved as well as un-sieved ones) generally start with a sufficiently big pool of xenotime particles. Following the trend of the curves towards the right side, all real fractions from sieved head material keep on being above the base line of *N*=25. That is completely true to all the fine fractions minus 63 microns too. However, beginning with the fraction 63-90 microns, the simply virtually screened samples start plunging below *N* = 25.

By measures like gravity pre-separation (+2.95 gravity curve) before the MLA as well as the combination of the counts of repeated cuts, and reducing the number of virtual coarse fractions by means of the MLA-software, this trend could be mitigated keeping *N* at least partly above 25 and improving the reliability of results on virtual fractions.

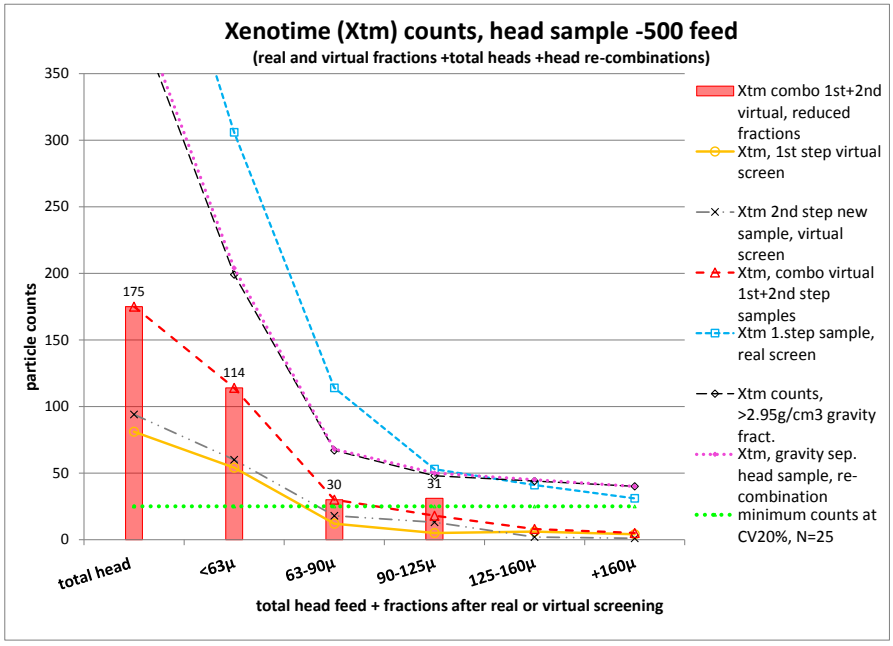

Figure 3 – Particle count numbers of head samples and fractions from real and virtual screening (-500 feed)

Some of the negative effects deriving from statistically insufficient count numbers are exemplified in the following figures.

The left series of 6 columns of Figure 4 displays the main binary xenotime lockings of head and real fractions (-500 feed, screened) with statistically sufficient count numbers for all fractions.

In the left middle, a seemingly erratic trend of lockings for the fractions of the first virtual fractions series (next 6 columns) indicates a falsifying influence of too few xenotime particle counts (see *Figure 3* too).

On the right middle, another 4 columns show a combination of a virtual repeat measurement with the first virtual screening.

By that repeat measurement and a reduction of coarse fractions ensuring more than 25 counts for each fraction, a reliable size dependent trend could be regained. Nevertheless, and even after some amendments, most of the virtual screening series on unscreened samples showed a somewhat higher proportion of lockings than the real one. That reminds of similar results reported by Greet (2013).

However, the virtually screened and recombined gravity separation fractions (<2.95 plus >2.95 g/cm^3 = ><2.95) on the most right side of Figure 4 seemingly go off that trend. Here, nearly the same locking proportions could be stated with even a slight tendency of less lockings of the coarsest fraction.

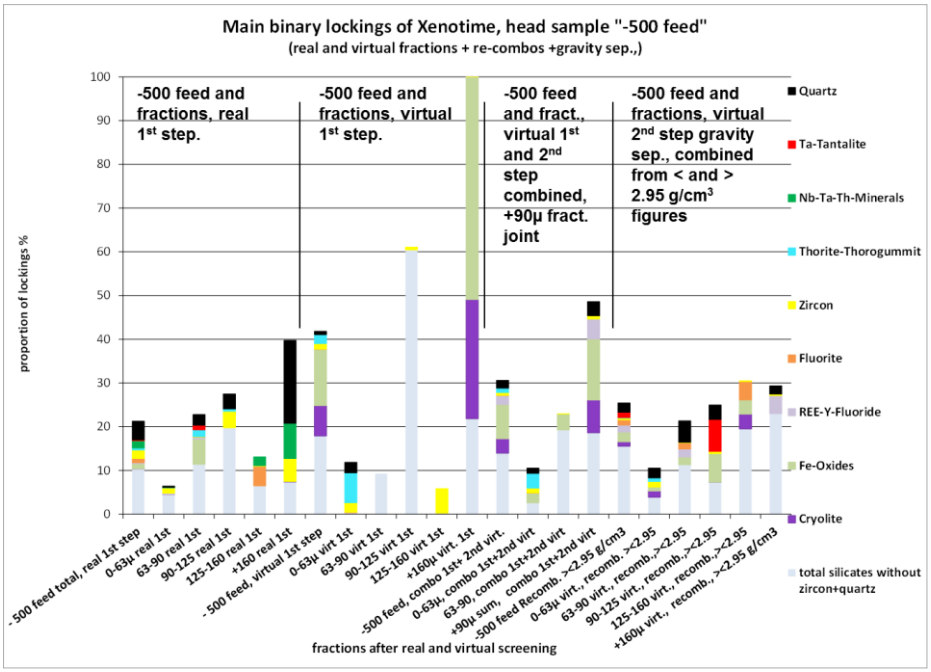

Figure 4 – Binary xenotime main lockings of screened and unscreened samples (-500 feed)

As a detrimental side effect of too low particle counts, even for the combined virtually screened samples “1st” and “new1”, no coherent grade versus recovery curve could be calculated for the coarse fractions above 125 microns. However, by combining the three coarse fractions in one counting bin +90 microns, a reasonable trend curve was restored just by gathering more xenotime particles.

The next table (Table 1) is supposed to show a fraction-wise comparison of the theoretical xenotime recovery at a cumulative mineral recovery value of 95 % (as it can be read from the grade-versus-recovery-curves). It turned out that only small differences between the real and the virtual approach occurred for the size fractions below 90 microns. In terms of the coarser fractions +90 microns, a trend of a generally lower recovery on virtually screened samples can be stated which is obviously in accordance with the higher proportion of lockings displayed by Figure 4 for instance.

The trend of seemingly somewhat more lockings above 90 microns is underlined by Figure 5 too.

This Figure as well shows how helpful a pre-concentration step by a heavy media separation can be to improve the counting statistic of rare minerals and stabilize the reliability of the mineralogical figures of a virtual screening approach.

Throughout all virtual fractions created from the >2.95 g/cm^3 head sample, there is quite a good agreement with the real screening fractions in terms of the grade-recovery curve (Figure 5).

But, this "heavy fraction" approach required some additional laboratory work and two MLA-shots extra. Furthermore, some "loss" of valuable xenotime grains in the light fraction was to be concerned. It turned out that 9 out of 407 xenotime particles were deported to the light fraction. That is a relatively low loss of about 2 particle-% only. Translated to an elemental deportment of Y that was calculated on assay figures from the MLA data base, 0.8 % of the Y traveled to the light fraction.

In terms of xenotime locking and grade-recovery characteristics, next to no difference between the head of gravity separation and the mere >2.95 fraction was found (see Table 1).

By recombining the gravity separation head from the MLA-numbers of heavy and light fraction samples, the best agreement with the real fraction PSD (from actual sieving) was accomplished amazingly as can be seen in Figure 2.

Table 1 – Cumulative xenotime recovery at a mineral grade of 95 % (-500 feed)

Sample -500 feed, cumul. Rc at 95% Xenotime grade,	**sieved real screening**	**unscreened**				
		1st step virtual screen	**new sample 2nd step, virt. screen**	**combo 1st + 2nd step virtual**	**density fract. >2.95 g/cm3, virtual**	**gravity head recombined, >< 2.95, virt.**
total head	**92**	**86**	**87**	**87**	**90**	**90**
0-63 µ	**100**	**99**	**99**	**98**	**99**	**99**
63-90µ	**96**	**98**	**95**	**95**	**98**	**98**
90-125 µ	**91**	**99**	around **70**	around **80**	**93**	**92**
+80µ combo		*72*	*63*	*68*		
+90µ combo	*86*	*74*	*58*	*68*		
125-160 µ	**88**	around **93**	**?**	around **92**	**88**	**88**
+125 combo	*82*	*62*	*?*	*52*		
+ 160 µ	around **77**	around **50**	**?**	**< 40**	**78**	**79**

Note 1 for Table1: Question mark "?" means no figures available;
Note 2: Italic numbers show results of joined fractions

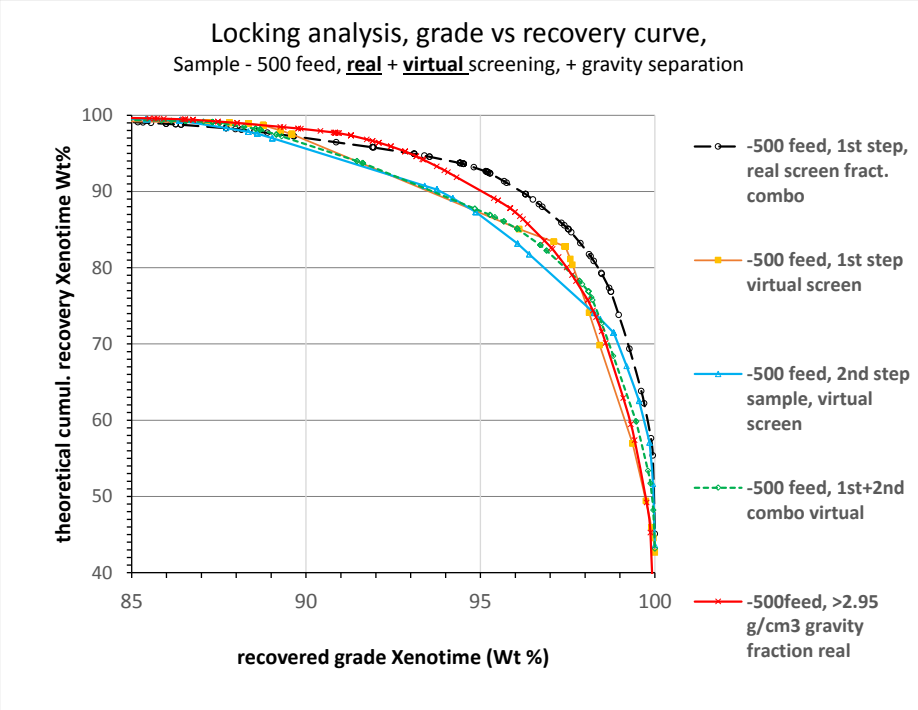

Figure 5 – Grade – recovery curves of xenotime on screened and unscreened head samples (-500 feed)

Representativeness and Segregation with Sample "+125 regrind"

Regarding the second sample series "+125 regrind" and contrary to the sample "-500 feed", the particle count statistics of xenotime were acceptably good. In terms of xenotime locking, only slightly higher locking proportions could be stated from the first section of unscreened head ("first cut"). This difference of about 6 % for the head samples between real and virtual approach could be reduced down to about 4 % by calculating a combined data base of the first cut with a second section ("second cut") as shown in Figure 6.

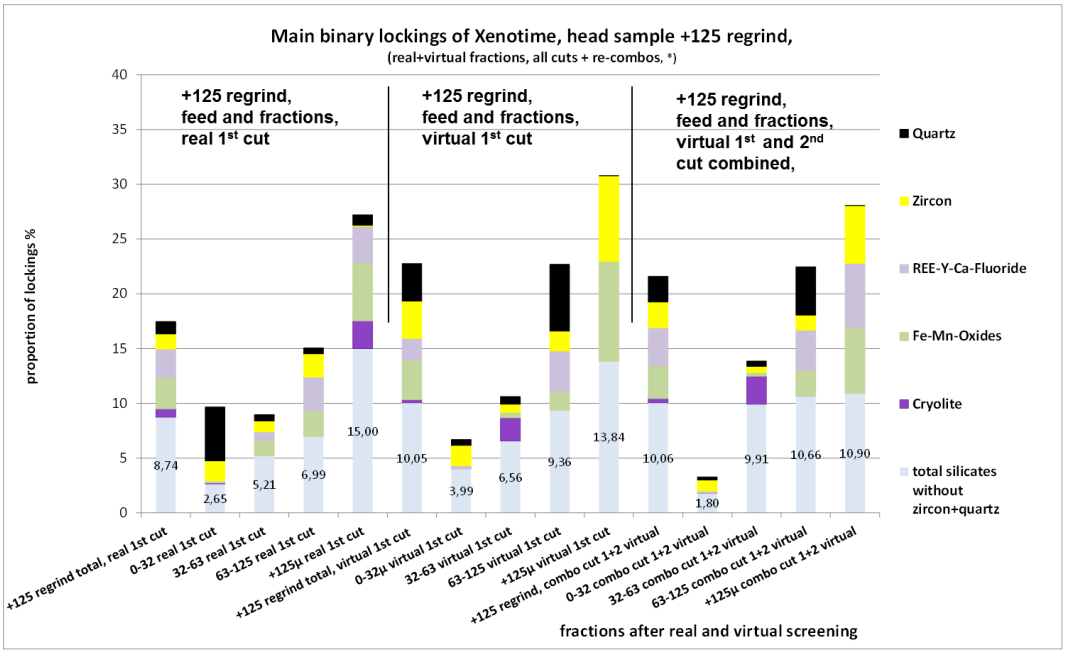

Figure 6 – Comparison of binary xenotime main lockings of real and virtual fractions (+125 regrind)

But, comparing real and virtual fractions, a certain bias in terms of mineralogy and PSD is thought to be indicated by a couple of results as follows.

In Figure 7, a considerable deviation can be seen between 4 out of 5 curves of the "+125 regrind" samples exemplified by the PSD. Thereby, the differing PSD trends of the unscreened samples and the sieve-screened ones remind of the results of Petruk (2000) and Lastra (2014). The cause couldn't be completely worked out, though a biased sample splitting or preparation problem was suspected to be occurred.

After having done a second section ("second cut") on the available first unscreened image ("first cut"), a segregation according to the size and density is concluded to have been the main reason probably. Clearly, the particle size difference between cut 1 and 2 can be seen in Figure 7 with cut 2 showing remarkably more fines. This gap could be explained by a size-driven segregation.

A complete new sample preparation and repeat of the first cut did not bring about any considerable PSD difference. That is taken as some evidence of a proper sample splitting job.

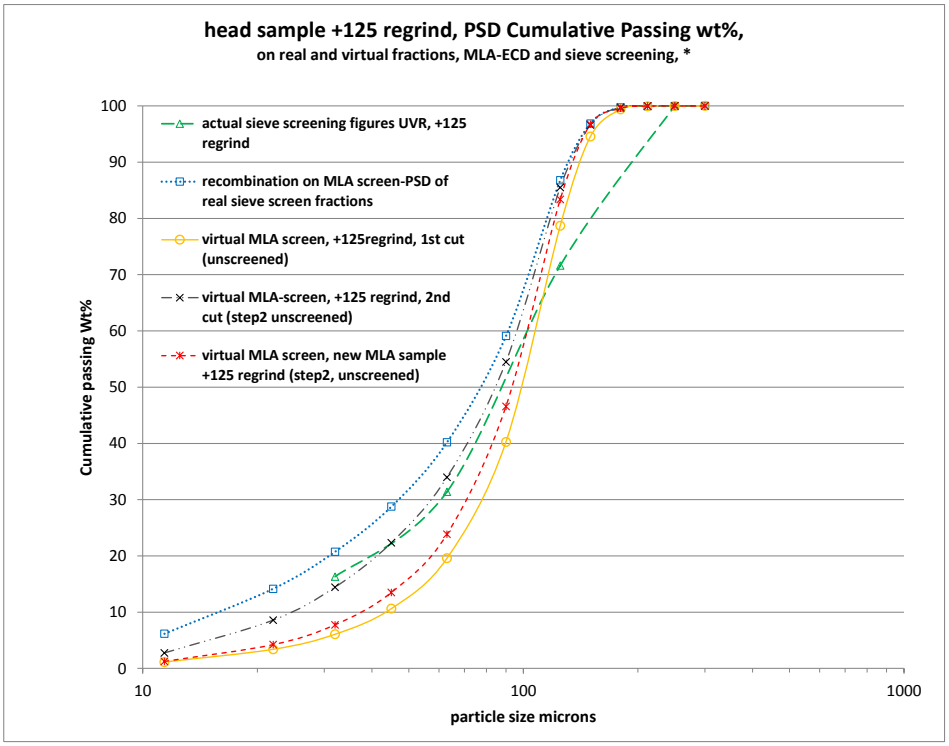

Figure 7 – Comparison of PSD from actual sieving and virtual screening, (+125 regrind)

In order to deliver another evidence of a segregation-made bias of the mineralogy of the 1st and 2nd cut, the zircon content is displayed in Figure 8. The first cut shows a generally higher zircon level of up to nearly 20 % more zircon. This can be explained by a seemingly happened segregation of the heavier zircon and other minerals at the cost of light silicates like quartz when embedding the sample in resin. Accordingly, the content of xenotime was higher in the first cut sample likewise.

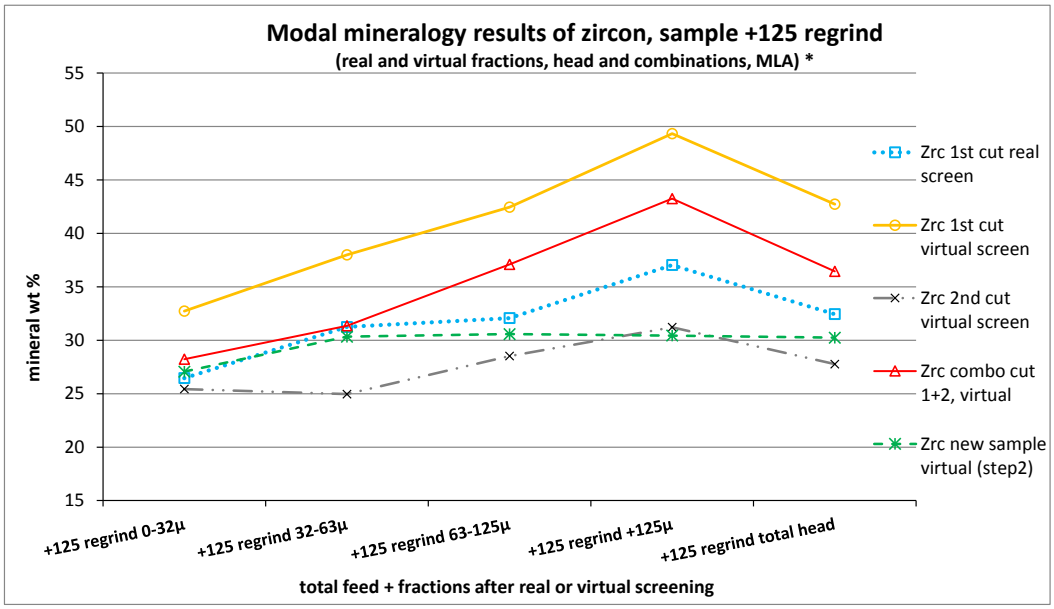

Figure 8 – Comparison of the zircon content of virtual and real fractions (+125 regrind)

Simply by calculating an average curve on combined 1st and 2nd cut numbers, the mineralogical gap between real and virtual approach could be remarkably (but not completely) reduced for the finer fractions above all.

CONCLUSIONS AND RECOMMENDATIONS

From the results, it can be concluded that the virtual-sizing-approach on unscreened samples can be used if the particle counting number of each and every single size fraction is high enough for the mineral of interest to ensure a variation coefficient of maximum 20 % as suggested by Lamberg (2007).

Furthermore, any gravity- or particle size-made segregation of mineral grains in the resin bed must absolutely be avoided.

These preconditions are particularly crucial in case of low-grade ores with mineral grades far below 1% as well as grain size fractions +90 microns.

Nevertheless, the authors encourage processing engineers to use the easier and usually less expensive virtual approach on unscreened samples, particularly if a finely intergrown ore has to be treated. In agreement with Petruk (2014), they can confirm that the virtual screening approach is suited to carry out comparative mineralogical studies and yield similar conclusions to the real screening approach on sieve fractions.

In case that conflicting mineralogical results or evaluation problems may loom, the following practical measures are recommended to verify and improve the reliability of this simplified way of investigation:

- Repeat the MLA using a representative second sample image. This measure roughly may double the number of particles of rare minerals in a count bin by simply combining both MLA results in case of uncertain particle counting statistics.
- Try a pre-concentration step by a suited procedure like gravity separation if that is doable. This measure can considerably enlarge the countable particle pool in case of valuable (and heavy) rare minerals contained in the head sample at a very little concentration below 1 weight-%.
- Reduce the number of virtual size fractions as far as doable to maximize the countable particle pool of rare minerals per fraction. This is true particularly to the coarse fractions. Adjust the size increments for the same reason.
- Apply a second section to the original MLA image in case of a looming biased representativeness that may be caused by gravity and/or size induced settling and segregation effects during the image preparation phase. Try to use the results of both sections as a virtual combination.
- Alternatively, in case of a settling induced bias, one may apply a cut in the direction of the gravity settling (Heinig, 2015).

However, for low grade ores that are characterized by a fairly coarse PSD (e.g. considerable proportions +90 microns), the traditional way on “real fractions” from sieved feed material should be preferably used.

Now as before, for any calculation of processing balances, no mineral grades from MLA but a suited chemical assaying should be used as a basis.

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