Procedure for quantitative evaluation of mineral liberation.

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In mineral processing plant there is a challenge in turnaround time for issuing results timely and consequently the decision making of the process is delayed. The hierarchy of steps involved in procedural analysis could be one of the causes. Mineral liberation is considered as one of the efficiency drivers in informing the decision for the downstream process such as flotation process. Thus, delayed decision could be costly. As a result, the attention of this research was drawn to the development of a procedure to quantify and minimize the time analysis for mineral liberation. The improvement of turnaround time was observed.

Keywords: Mineral liberation, particle size

1. Introduction

Many mineral processing plants encounter a challenge in turnaround time for issuing results timely and consequently the decision making of the process is delayed. One of the causes is due to procedural analysis. For a 12 hr shift where routine material analysis is done for every 2 hrs, collection of one to two milled samples supposed to be collected and analysed for mineral liberation quantification. However, this is not usually done because most mineral liberation analysis techniques take 3 to 8 hrs to complete the analysis of particles. Very few liberation techniques could offer the analysis in less than 2 hrs. Grant et al., (2018) took 1 to 1.5 hours to acquire mineral liberation analysis for iron ore in the -200 mesh size fraction. Therefore, any decisions that must be informed by the analysis results are delayed and thus could be costly.

Mineral liberation of particles is the driving factor of potential high value mineral recoveries (Little, 2016). It is widely known that without exposure of value minerals from within the particles to be separated by either flotation or leaching separation processes, is costly and drastically minimise the value recoveries.

The measurement of mineral liberation is usually attained from mineral liberation analyser (MLA) which makes use of the software having measurement modes such as XBSE and GXMAP. All two modes can be used for the liberation measurements but it all depends on the ore type under study. For ore where the mineral phase of interest is in bulk such as hematite ores, usually XBSE is used whilst for tiny mineral phase like gold or platinum group metals and thin sectioned samples, GXMAP is used. However, mostly the clients prefer XBSE analysis for gold or platinum group metals due to its thorough measurements but because GXMAP analysis takes more time to generate the mapping of phases and also expensive if one is outsourcing the service. They would rather opt for XBSE. This is owed to the relatively-close point grid method GXMAP uses to map the 2D image, hence lengthy run time required.

For example just to map one sample it could take 5 hours or even a day as opposed to XBSE which typically takes less than about 3 hrs (Sylvester, 2012). It all depends on the type of MLA software one is using, the objective of the analysis and resolution required. Others are faster than the others. In the instance where the XBSE is applied to map tiny mineral phase, often counting time is increased instead until enough particles are representative. In this regard, the mapping time could be from 3 hours to 7 hours (Sylvester, 2012). It also depends on the type of MLA software one is using, the objective of the analysis and the resolution required. In the case of mapping bulk minerals the XBSE can take 4 to 7 hours as well, depending on the objective, resolution, size of frames to be covered. Frame sizes is a function of particle sizes (Sylvester, 2012). The other factor that adds to mapping run time is when there are many unknown minerals to database library of X-ray spectra. The exercise of having to identify the unknown minerals and eventually upload them in the library data can sometimes take time since it is manually done. Supplementary to that, if MLA breaks down an operator must do spectra scavenging from other projects and make their own data. This also take time. Upon completion of X-ray mapping of both modes, the acquisition of mineral liberation data along with modal mineralogy, mineral associations, particle and grain size distribution, and particle shape parameters can be generated (Becker et al., 2009; Cropp et al., 2013) and it takes few seconds to issue all the measurements.

The other technique that could be used to measure liberation is X-ray computed tomography. However, the commercial application of it is currently scarce and expensive (Camalan et al., 2017; Zhang and Subasinghe, 2013). Very few mineral industries have implemented geo-metallurgy prediction beneficiation strategy to mitigate different challenges including liberation measurements but many of industries are still behind. Addition to this, many industries find it difficult to predict mineral liberation and other elements of the process starting from mining including geochemistry and geo-statistics and financial modelling in order to maximize sustainability. Two causes of the difficulties are due to geological resource constraints which arises from exploitation of deposits with more complicated ore properties (Rosenkranz and Lamberg, 2015) and sampling constraints. In many cases, more of sampling in mining and mineral processing plant is very costly and time consuming. Thus, they rather focus more on production. Therefore, spatial distribution model will cause biggest error. As a result, geo-metallurgy is rarely applied to predict mineral liberation in advance.

The challenges mentioned above are some of the reasons causing delayed informed decision for metallurgists. The challenges become even more if the plant laboratory does not have its own equipment instead outsource the liberation analysis service. Hence, most of the industry they would prefer to relate mineral liberation with particle size. In essence, when milling the ore true liberation is not often monitored because of the time constraint to measure liberation data (Mariano, 2016). As a result of this type of liberation assessment, many parameters such as mineral texture and degree of value mineral liberation and exposure are neglected. In addition, having to estimate mineral liberation as a function of only particle size, causes the metallurgists to not notice when the ore begin to change in the system. This, also factor into delaying the constructive decision to be made on time. This type of practice causes the mineral industry losses that could be avoided. Only if the correct and inexpensive tool can be used for liberation estimations.

These are indications of the challenges faced in obtaining accurate characterisation of mineral liberation of particles upon breakage within a reasonable time during mineral processing operations. Consequently, upon recognising a need in this area, this study was undertaken. The aim was to develop a simple procedure, for mineral processing plants, which can better estimate the quantification of mineral liberation within acceptable time. The procedure developed is proposed to assist timely decision making for metallurgists during plant operation. The procedure developed is also aimed at not only using index values but eliminating the use of X-ray detector for XBSE and GXMAP analysis which is usually utilized after the acquisition of the backscattered images. The mineral present in a sample should be known before hand before using the method.

2. Experimental method

The iron ore was the subject of this study. It was selected based on its binary phases which are convenient for the objective of this research. Firstly, backscattered images obtained from SEM of MLA was analysed by MLA and the outputs, namely modal mineralogy, mineral associations, particle and grain size distribution, particle shape parameters and mineral liberation data were recorded. The same captured backscattered images were used in a method developed in this study.

The four iron ore feeds of different particle sizes were presented to the MLA as a standard polished section (27 x 46 mm). Prior to presentation of the samples to MLA, thorough sampling preparation was conducted. Upon the completion of thorough sampling using blending equipment which was followed by cone and quartering, about 10 kg ore was crushed. Then, the whole 10 kg crushed sample was further sampled using spinning riffle to generate almost 10 bags of 1 kg's of which 4 * 1 kg's bags were set aside for milling at different grinding times. Further sequential sampling was performed for each 1 kg of four bags milled material by spinning riffle to obtain representative sample for polished sections. The iron feeds were prepared by crushing them to 5mm top size, and thereafter milled separately from 5 minutes to 20 minutes, with an increment of 5 minutes. All four polished sectioned samples were processed through MLA from which the backscattered images were analysed by the MLA software. They were all measured in XBSE mode. Once the analysis was done, the same captured backscattered images were to be used on the proposed procedure. The use of the same backscattered images for MLA and proposed method was to eliminate bias in calculations.

3. Development of a quick and inexpensive procedure for the liberation measurement

The image processing tool of MATLAB software 2016 version was used to capture backscattered images and process them as instructed, for example one of the images used in this study is in **Figure 1**.

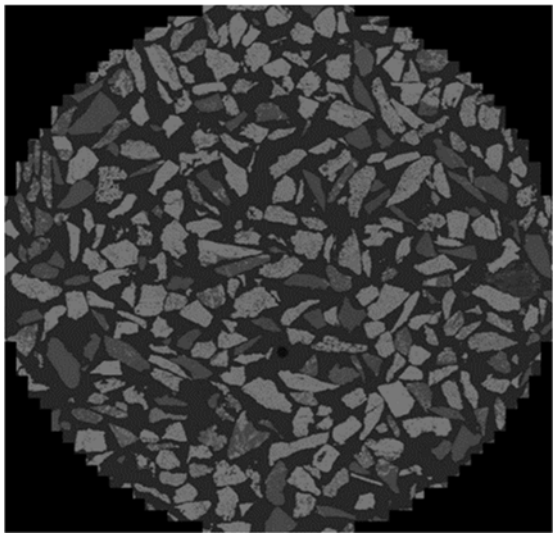


Figure 1. Backscattered image from SEM of MLA.

Firstly, the borrowed backscattered images were stored as an indexed images in the image processing tool as illustrated in **Figure 2**. The indexed and RGB values were automatically displayed for each phase as an outputs of the indexed images. From the figure, it can be seen that each phase has its own unique index and RGB's values. The brighter phase has index value of 98 and RGB values of [0.3843: 0.3843: 0.3843], and grey phase has 53 and [0.2078: 0.2078: 2078]. The two backgrounds with different index values were also detected. One background is located within a polished section frame and the other one is outside the frame. The dark grey background within the frame of the sample is found to be 28 and [0.1098: 0.1098: 0.1098] while the black background outside the round sample frame has 0

index and [0 0 0] RGB values. The different phases observed are hematite mineral, which is the brightest due to its atomic number's heaviness as opposed to quartz phase which is of grey phase and the dark grey area is a carbon resin. In the SEM, the elements of higher atomic number always appear brighter contrary to their counterparts (Sylvester, 2012). Due to these different index values of different phases it was relatively easy to demarcate the boundaries between different mineral grains based on their index values in the BSE images. Thus, the analysis began at identifying the mineral phases present using index values.

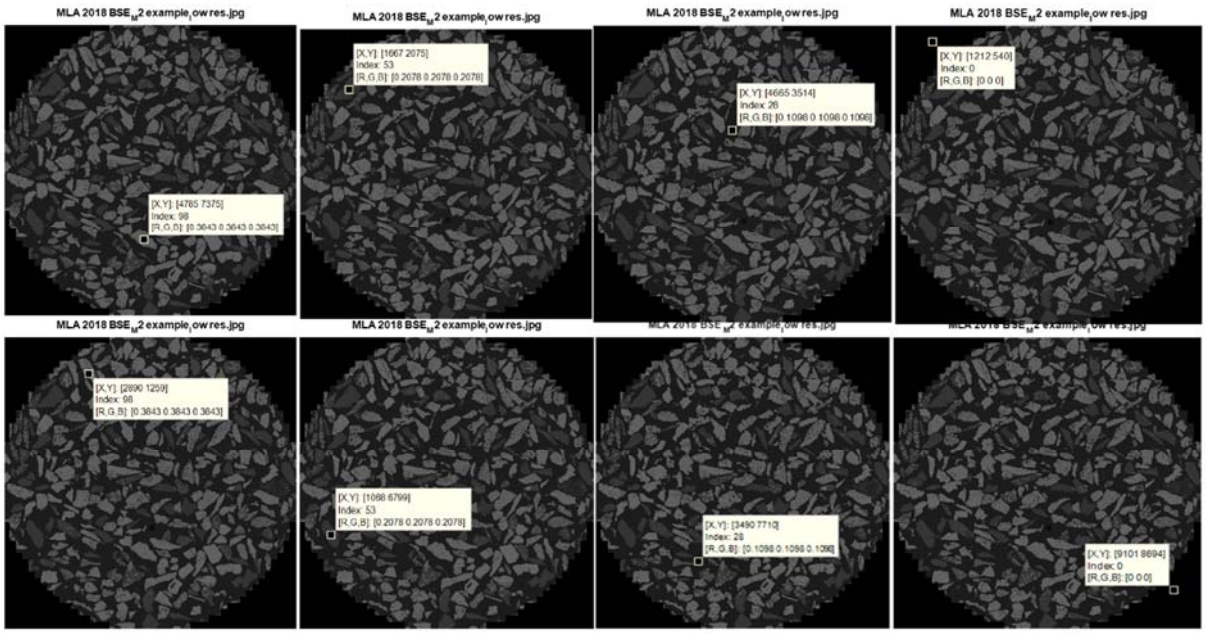


Figure 2. An indexed image.

Following the identification of each minerals index values, the enabling of image modification was possible prior to liberation quantification measurements.

3.1 Image modification

Firstly, the 28 indexes of the background within the polished section frame was converted to index 0 so as to have uniform background all across the image, see **Figure 3**.

Figure 3. Background conversion of the background within to make it the same as the background from outside the polished section frame.

Once the background was converted, the image was then changed to binary to have only two numbers which are 0 and 1. The colour black was represented by 0 and 1 represented white as demonstrated by **Figure 4**. As a result, the total number of particles contained across the image were counted and registered for statistical purpose.

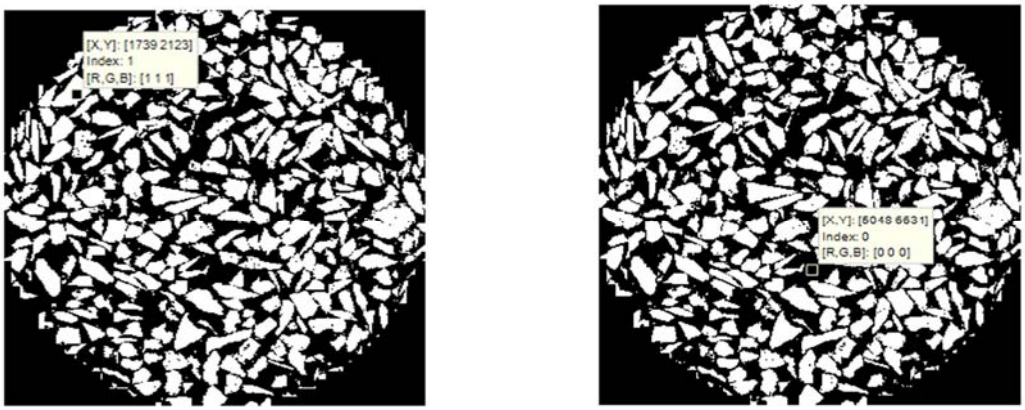


Figure 4. Binary image for easier calculation of the total number of particles across the image

The 34314 of particles were obtained in an array of the total image. Followed was the calculation of mineral liberation classes for Fe_2O_3 and SiO_2 in percentages.

3.2 Liberation measurements

Prior to quantification of mineral liberation of each particle counted, each particle had to be segmented by placing a frame around. The target aimed at the whole single particle to be within a frame imposed in it. Any particle(s) that was found in the same frame as the whole single particle was filtered out.

3.2.1 Filtering of particles within a frame and segmentation of phases present

Each particle had to be cropped out and bounding boxed as in **Figure 5(a).** It was then changed to binary image as it is in **Figure 5(b),** to enable filtering of any undesirable particles that might be present within the frame as it is in **Figure 5(c).** The filtering was done by removing any pixels of particles that are not part of the whole particle. This was performed so that ultimately only phases in a particle within, are calculated accordingly.

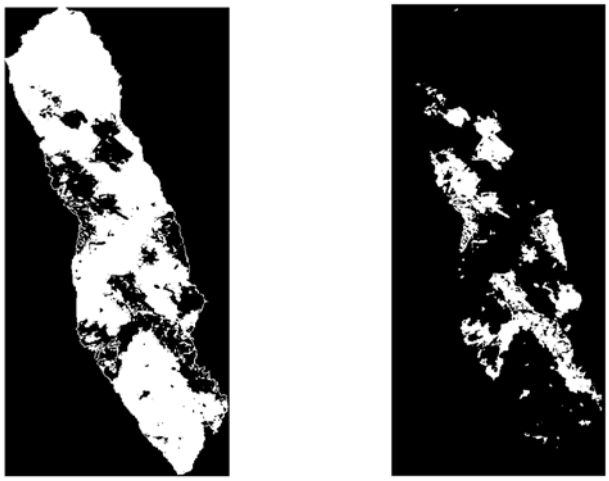
(a) **(b)** **(c)**

Figure 5. (a) Cropped image and (b) cropped image converted to binary image and (c) the masked binary image filtered of unwanted pixels

Then the segmentation of phases was conducted by using thresholding. The thresholding process of a particle was achieved by camouflaging the original image with the binary image and the background of the camouflaged image was set to be zero. This was done so that different pixels of silicate and hematite phases could be visible in the white background. Hence possible quantification of each phase and their liberation measurements.

3.3 Liberation determination

As it was observed from the original indexed image that SiO_2 phase pixel values are between any pixel values greater than 0 but less than 0.3 whilst for Fe_2O_3 phase values are found to be greater than 0.3. The threshold of pixel values of two phases were used as a basis for quantification and calculating mineral liberation from filtered camouflaged image in **Figure 5 (c)**. For example, to quantify Fe_2O_3 phase of a particle in the Figure below, the threshold of any values greater than 0.3 was set. As a result, only Fe_2O_3 phase values remained while the rest disappeared as it is in **Figure 7(b)**. Therefore, quantification of Fe_2O_3 phase values and its liberation calculation per area of the whole particle could be done efficiently. The same principle was applied for SiO_2 phase values but the threshold was set to be between any values greater than 0 but less than 0.3, see **Figure 7(a)**.



(a) **(b)**

Figure 7. Segmentation of phases by applying thresholding (a) $0 < SiO_2 > 0.3$ and (b) $Fe_2O_3 > 0.3$ were applied.

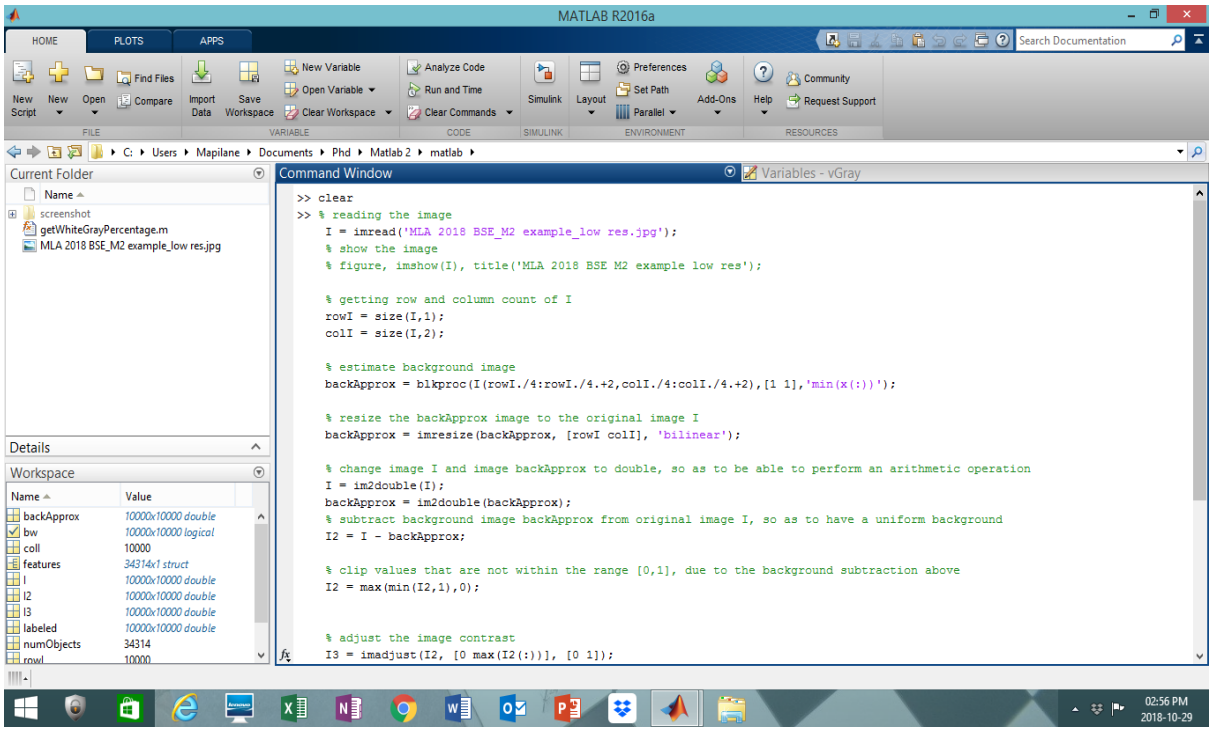
The contribution of values of Fe_2O_3 phase in a particle was summed and subtracted from the total pixel values of the whole particle. Same calculation phenomenon was applied to SiO_2 percentage relative to the whole particle. As a result, ten liberation classes were generated which are from 0% > x < =10%, 10% > x <= 20%, 20% > x <= 30% and classes increased with an increment of 10% until the class of 90% > x <= 100%. For example, any particles that have 5% Fe_2O_3 would report to a class of 0% > x < 10%, refer to **Table 1**.

Table 1: Liberation classes of Fe_2O_3 and SiO_2

	Locked				Middlings					Liberated	
Composition	0>x<1%	1>x<10%	10>x<20%	20>x<30%	30>x<40%	40>x<50%	50>x<60%	60>x<70%	70>x<80%	80>x<90%	90>x<100%
Fe_2O_3 pixels	0.0204	0.5696	0.7349	0.6294	0.6732	0.394	1.6385	1.5951	5.117	7.7233	5.8956
Quartz pixels	2.6937	3.1376	1.1799	0.8471	1.0262	0.3774	1.078	1.119	1.3789	1.6068	0.7903

The results in **Table 1** was further categorised in three classes of locked mineral, middlings and liberated mineral. The cut-off points for locked mineral class was between 0% > x <= 30%, whilst the middlings class was 30% > x <=80% and for liberated class was between 80% > x <=100%. It only took run time of less than five minutes per backscattered image to obtain all calculations mentioned above. This approach is easy to follow because it only requires a backscattered image and the attached function file under current folder as it is in **Table 2** below, to conduct mineral liberation analysis. The first part of code used is also demonstrated under command window.

Table 2: The screenshot showing MATLAB command and files used



4. Discussion

This method does not need big storage for large data files as much as MLA and QEMSCAN. It is designed for few images and it measures the whole area of the sample as opposed to several frames. It only took run time of less than five minutes per backscattered image to obtain liberation measurements using the code developed as opposed to conventional particle X-ray mapping time by MLA which takes more than 2 hrs to 6 hrs to issue liberation results. This approach is easy to follow because it only requires a backscattered image and the attached function file under current folder as it is in **Table 2** below, to run the test. The liberation analysis by this proposed approach can be cost- effective as opposed to having the mineral processing plant to frequently outsource samples to be analysed. The following is the economic analysis of the proposed mineral liberation analysis procedure versus traditional mineral liberation analysis.

5. Economic analysis of the proposed procedure

The implementation of this proposed approach can be cost- effective as opposed to having the mineral processing industry to frequently outsource the liberation analysis. **Table 3** shows the economic analysis comparison of the proposed method and MLA route. If the mineral processing plant was to purchase MLA unit and all the consumables required for sample preparation before the liberation analysis, it will costs 547 688 USD. However, the proposed procedure requires the backscattered image from scanning electron microscope (SEM/EDX) which can be acquired from benchtop SEM amounting to 98 861 USD. Once the image is acquired it can be analysed offline using the code developed from MATLAB software – image processing tool which only requires a computer and a MATLAB licence. Since in most cases sample preparation of MLA analysis is conducted on polished section sample, the preparation takes almost more than 8 hours which includes the vacuuming of a sample in order to remove bubbles from the resin mixture. Then, followed by polishing of the sectioned sample and eventually carbon coating which generally takes less than 10 minutes to carbon coat one batch of samples. Once the sample is ready for mineral liberation analysis, it could take more than 3 to 5 hours

phase X- Ray mapping or even more time depending on the type of analysis mode. As for proposed approach instead of using polished section for sample preparation, thin sectioning is recommended which only requires glass slide and a glue. Its preparation could take close to an hour including grinding, polishing and carbon coating of the sample. To capture backscattered image from benchtop SEM would take less than 10 minutes. While code developed will run for less than 10 minutes to issue mineral liberation results. Overall time needed to generate the mineral liberation results using the proposed approach could be less than 1h 30min as opposed to MLA route which needs 3 hours to 7 hours. In addition, if the mineral processing plant was to implement the proposed approach it will only need 152 560 USD as opposed to 547 688 USD if it purchases the MLA and follow the traditional mineral liberation analysis route. The proposed approach could easily save up to 395 128 USD operational costs as indicated in **Table 3**.

Table 3: Economic analysis of the proposed procedure versus the conventional MLA route

	Prices (US Dollars)		
	Proposed integrated approach	MLA route	
Consumables			
Glass slide and a glue	211 USD		
Resin, epoxy and moulding cups		423 USD	
Grinding polishing machine	14 123 USD	14 123 USD	
Piano plates, polishing cloths, Si lubricant	2 471 USD	2 471 USD	
Vacuum Pump + Compressor		1 059 USD	
Carbon Coater equipment	35 307 USD	35 307 USD	
Benchtop Scanning Electron Microscope (SEM)	98 861 USD		
MATLAB licence (designated computer)	459 USD		
High speed and 4 - 8GB Computer	1 128 USD		
Mineral Liberation Analyser (MLA) Unit		49 4305 USD	
Total	152 560 USD	547 688 USD	395 128 USD

The MLA maintenance, installation and infrastructure costs are not included. If they were to be included the operational costs were to increase. However, the use of the benchtop SEM requires minimal maintenance, can be installed very easily and do not need infrastructure.

In the event where the plant was to outsource the services, usually for one sample mineral liberation analysis would cost close to 353 USD or even more depending from company to company. It implies in the 12 hours shift where usually samples are taken every two hours, the analysis of at least 12 samples would cost 4 660 USD if two samples are analysed in every two hours at the milling discharge or flotation feed. For 24 hour shift it will be 9 320 USD. In a month the analysis can easily amount to 260 960 USD. This is still more as compared to 152 560 USD and equipment used is plant – owned as opposed to outsourcing option.

6. Conclusions

The proposed procedure can be implemented in, but not limited to, the old existing mineral processing plants which might not have been designed for the ores that they are treating. This is because they were built long time ago and technology has changed so is the upgrading of processes. As a result, they take the loss because it will cause them too much money to change.

In addition for image capturing, procedure can be integrated with a benchtop SEM such as IEM 10, IEM 10+, IEM 11 and IEM 11+ depending on the affordability and specification requirement by mineral processing plant. They are five times less than the cost of mineral liberation analyser and are also of the size of the old computer box. Specially trained operators are not required for the operation as opposed to traditional MLA and the liberation code developed is easy to use as it only requires high quality image as an input and the function file created will automatically run the test and generate the mineral

liberation classes. It would take eight minutes to issue the liberation results. This is due to three minutes execution runtime of a code per BSE image and five minutes from high quality image capturing by benchtop SEM at 100 000 or 150 000 magnification. Therefore, in a 12 hrs shift, when samples collected from milling are to be analysed in every two hours, this proposed integration strategy will make it possible. The implementation of the proposed approach would save close to 395 128 USD or more as indicated in **Table 3**. It is important to note that the proposed procedure was only tested on hematite ore used in this research.

For existing optimised plants, this method can complement the results from mineral liberation techniques. This means while waiting for the mineral liberation analysis from MLA this method can be employed to continuously assess liberation analysis.

In conclusion, the study used indexed values of the phases from the backscattered image, including codes to measure mineral liberation. Contrary, to MLA traditional approach of using XBSE or GXMAP mode (Sylvester, 2012) which usually take more run time to map the sample's particles then eventually issue the outputs. While it is true that using MLA, one could determine many outputs from the analysis but the improved analysis time emerged from the proposed method would be advantageous in the event where fairly quick examination of mineral liberation is desirable. Supplementary, the proposed procedure developed is implemented on a user-friendly and inexpensive software.

7. Future development

In the future development, it is recommended that multiphase ore be used since thus far the application of this method is limited to binary ore particularly of iron ore.

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