

Research paper

## The effect of graphite filler in sample preparation for automated mineralogy – a preliminary study

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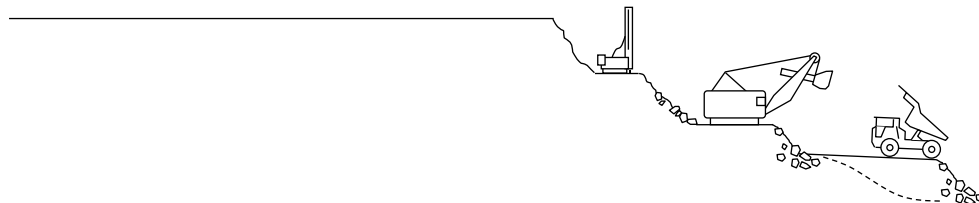
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### ABSTRACT

Mineral particulate sample material is often prepared as polished sections for SEM based automated quantitative mineralogical analysis. A common practice is to mix ultra-pure graphite with sample material to avoid segregation and touching particles, and aid random orientation, as suggested by Jackson et al. (1984). However, the actual effect of adding graphite on these parameters is not well documented. This paper presents a preliminary study on the effect of adding graphite on segregation and touching particles. A mineral particle concentrate, with equal volumes of quartz and pyrite, was made in two size fractions (53-74 µm and 104-147 µm). Each fraction was mixed with graphite in sample:graphite weight ratios 1:1 and 1:2. In addition, 1:3 was made for the finer fraction. Two types of graphite were used: fine grained (<20 µm) ultra-pure graphite, and coarse grained ultra-pure graphite in the same size range as the sample material. Reference samples without graphite were made for both fractions. The results suggest that using graphite as filler does not necessarily prevent segregation. However, it introduces a positive effect on preventing touching particles; fine graphite is the most efficient. The epoxy resin:sample ratio needs to be better controlled to confirm the results.



### I. INTRODUCTION

Scanning electron microscope (SEM) based automated quantitative mineralogical analysis (automated mineralogy) is a commonly used analytical technique in mining and mineral processing (e.g. Malvik, 2014; Sandmann, 2015). It is used to quantify ore properties such as mineral liberation, modal mineralogy, element deportment, mineral associations, textural information and various particle and grain properties (size, shape, etc.). Acquiring representative results require stable measurement conditions, correct analytical settings, representative sampling and unbiased sample preparation.

In mineral processing, mineral liberation is an important concept. Mineral liberation is achieved by crushing and grinding of the raw material, in order to

obtain relatively clean particles of the mineral of interest and gangue minerals, i.e. liberated particles. The goal is to obtain acceptable liberation at the coarsest particle size possible, before concentration of the mineral of interest (Wills & Finch, 2016). Hence, a correct measure of liberation is vital.

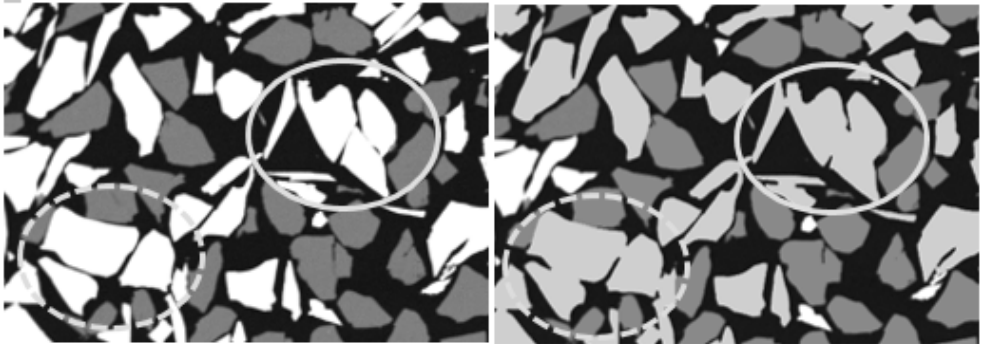
In automated mineralogy, mineral identification and grain/particle discrimination are usually achieved by energy dispersive spectroscopy (EDS) mapping and spectra comparison, or alternatively by combining grey level backscatter electron (BSE) images and chemical information from EDS with a mineralogical library (e.g. Gottlieb et al., 2000; Fandrich et al., 2007; Lotter et al., 2011; Wightman et al., 2016). Several types of sample material can be analyzed, such as mineral particulates or whole rock samples prepared from e.g. diamond drill cores or hand specimen (e.g. Pirrie & Rollinson, 2011; Wightman et al., 2016). The results from automated mineralogical analysis can be used for e.g. resource estimation, mine-, plant- and waste planning, problem solving and optimizing mineral processing plants etc. (e.g. Bradshaw, 2014; Malvik, 2014).

In recent years, increased availability of automated mineralogical systems has resulted in numerous publications in the field of process mineralogy, often presenting modal mineralogy, mineral liberation data and particle size distribution etc. from polished sections of mineral particulates (Sandmann, 2015). These studies are dependent on a series of samples, where a small sample volume is representing a larger batch of granular material. To ensure this representativeness the need for correct sampling (Gy, 1979) and unbiased sample preparation is fundamental (e.g. Jackson et al., 1984; Gottlieb et al., 2000; Baum, 2013; Sandmann, 2015; Wightman et al., 2016). Different sample preparation procedures for automated mineralogical analysis are well described in Sandmann (2015).

Regarding the preparation of polished sections of mineral particulates, the goal is to reduce the number of touching particles and maximize particle packing density, while maintaining a random distribution of the particles in the sample surface. Jackson et al. (1984) described possible sources of error, concerning the properties of the sample material and the sample preparation process, which might affect the results of an automated mineralogy analysis. These include differential settling of particles (segregation), touching particles, and systematic orientation of particles.

The term «touching particles» is in this context used to describe two or more particles erroneously interpreted as one particle by the automated mineralogy software (Figure 1), i.e. adjacent particles that appear to share a grain boundary segment. It is crucial to avoid high numbers of touching particles as they might influence the liberation analysis, and cause erroneous results. To reduce the number of particles incorrectly identified as touching, most quantitative

mineralogical analysis software uses some image processing techniques to de-agglomerate particles (Fandrich et al., 2007) e.g. by erosion and dilation. However, such image processing techniques involve removing and adding pixels to particles, and may change the size and/or morphology of the resulting particles compared to the original particles. Hence, excessive use of such image processing may introduce new errors, thereby affecting the results.



**Figure 1.** Examples of touching particles. BSE image (left) and binary image of reference sample QP 104-147  $\mu\text{m}$ . Dark particles=quartz, Light particles=iron sulfide. The grains in the solid circle are adjacent iron sulfides grains, interpreted as one particle. Grains in dotted circle are adjacent quartz and iron sulfides interpreted as one particle.



**Figure 2.** BSE image of vertical cut polished section. Silicate (gray) and iron sulfide (white) concentrate (104-147  $\mu\text{m}$ ) mixed with fine graphite, sample:graphite 1:1 weight ratio. Segregation is evident as the heavy particles are concentrated towards the bottom.

The term segregation is used to describe differential settling of particles due to density and morphological differences during the process of molding the sample in epoxy resin or other mounting media. The degree of segregation will depend on the density contrast of the different minerals in the sample, and the viscosity of the mounting medium (e.g. epoxy resin). Heavy particles will sink to the bottom of the mold faster than the lighter particles, as shown in Figure 2, a vertical section through a polished section. If this particular sample was prepared as a regular

polished section, the heavy minerals would dominate the sample surface and thus, the amount of heavy minerals will be overestimated.

Several issues introduced during sample preparation of polished sections may impact the results. According to Heinig et al. (2015) the epoxy resin:sample ratio will influence the results, as increasing epoxy resin proportions will aid segregation. Thin epoxy, un-sized samples and density differences will contribute to a greater segregation effect (Kwitko-Ribeiro, 2012). Other uncertainties introduced during sample preparation of polished sections of granular materials include the polishing depth, which can vary slightly from sample to sample, and cause biased results if material is segregated (e.g. Jackson et al., 1984). The sample surface must be planar and polished smooth to ensure correct quantification during the analysis (Goldstein et al., 2003). In addition to the factors concerning the sample preparation, analytical settings and particle statistics will influence the results of the automated mineralogical analysis (Hrstka, 2008). The issue of stereological error should also be mentioned, as the area of the minerals on a 2D surface is considered as equal to the volume of a mineral in a 3D sample in automated mineralogy (e.g. Wightman et al., 2016).

This preliminary study focused on the sample preparation of polished sections of mineral particulates. The aim was to show the difference in segregation and number of touching particles of different sample:graphite weight ratios using a systematic approach. The sample preparation procedure and amount of graphite used in this study were based on the recommendations by Jackson et al. (1984) and experience from different academic and commercial labs. The epoxy resin:sample ratio was not considered. Determining the segregation and the number of touching particles was attempted using a known sample material, with known composition. Two size fractions were prepared. Two types of graphite were used; fine grained (<20  $\mu\text{m}$ ) ultra-pure graphite, and coarse grained ultra-pure graphite sieved to the same size range as the sample material. A reference sample without graphite was made for both size fractions.

## **2. BACKGROUND**

Jackson et al. (1984) proposed a procedure for preparing unbiased polished sections of mineral particulates. Using sized samples has been suggested to minimize the segregation caused by particle size differences. The proposed method involves mixing the sample material with graphite in the same size range as the sample to prevent segregation and touching particles (graphite:ore weight ratio 2:1 for particles >106  $\mu\text{m}$  and 3.5:1 for particles <106  $\mu\text{m}$ ). It is emphasized that the amount of epoxy should be kept to a minimum.

A few studies addressing the importance of the sample preparation process have been published during recent years. Hrstka (2008) found that the reproducibility of the results of automated mineralogy analysis were significantly dependent on the sample preparation. Pooler and Dold (2017) showed that making vertical sections was the least biased sample preparation method regarding segregation. The effect of different sample material to resin proportions was studied, and showed that different amounts of epoxy have a large effect on the segregation (Heinig et al., 2015). Lastra and Petruk (2014) investigated the difference between sieved and unsieved samples, using a different method of sample preparation, whereby, molding a monolayer of particles avoids segregation biases. Dynamic curing of samples to avoid segregation was suggested by Kwitko-Ribeiro (2012). The use of fast curing epoxy to eliminate segregation was proposed by Bouzahzah et al. (2015).

Published research shows a large variation in practice of using graphite as an inert filler in this type of samples. It seems like the majority of the sample preparation labs use a variation of the method proposed by Jackson et al. (1984), many using very fine grained (< 20  $\mu\text{m}$ ) graphite as filler (e.g. Brochure on MLA, Geomet. lab. Freiberg, n.d.). According to Pirrie and Rollinson (2011), the standard practice in the mining industry is to use graphite with a slightly smaller particle size than the sample material.

Several studies prepare vertical cut sections instead of (or in addition to) regular polished sections (e.g. Smythe et al., 2013). I.e. the sample is first molded as a regular polished section before it is cut perpendicular to the bottom surface, flipped 90°, and molded again, before grinding and polishing, as recommended by e.g. Butcher (2010). This type of polished section is often referred to as transverse sections, cross sections or vertical sections. The drawback of making vertical sections is the added time to the sample preparation process. However, this procedure is expected to eliminate the bias caused by segregation, thus resulting in more representative results from an automated mineralogical analysis.

A substantial amount of the available literature unfortunately avoids presenting details of sample preparation. Typically, the description of the sample preparation is limited to: “sample material was mixed with graphite and embedded in epoxy resin”. Further explanations of the properties of the graphite and sample:graphite ratio etc. is sporadic. Lastra and Paktunc (2016) performed an inter-laboratory test on a reference material to check the variability of the results from automated mineralogical analysis of 17 different laboratories, which also illustrates the variation of sample preparation; twelve labs chose to make regular polished sections, four created vertical sections and one made a monolayer. Five of the labs reported using graphite as filler.

The chosen sample preparation procedure is likely to influence the results of an automated mineralogical analysis. To allow reproducible results, details of the sample preparation should be made available in publications of such research.

This preliminary study focuses on the effect of adding different amounts and different types of graphite as filler in polished sections of particulate materials, and emphasizes how variations in sample preparation procedure can impact the analytical results.

### 3. MATERIALS AND METHODS

#### 3.1 *Materials*

A mineral particulate sample material, consisting of 50 vol.% quartz and 50 vol.% pyrite, was prepared using a pure quartz particulate concentrate and a pyrite particulate concentrate. The two mineral concentrates were chosen due to their large difference in density. The correct weight of each mineral concentrate to achieve 1:1 vol. ratio, was calculated based on the approximate theoretical density,  $2.65 \text{ g/cm}^3$  for quartz and  $5 \text{ g/cm}^3$  for pyrite (Deer et al., 1992). Two size fractions were prepared: 53-75  $\mu\text{m}$  and 104-147  $\mu\text{m}$ . The quartz concentrate is an ultra-pure concentrate containing 99.99 % quartz. The pyrite concentrate contains 86.7 % pyrite, 9.5 % chalcopyrite, 2.1 % other heavy minerals ( $\rho > 4 \text{ g/cm}^3$ ) and 1.7 % light minerals ( $\rho < 4 \text{ g/cm}^3$ ) (Table 1). The chalcopyrite (and other mineral) content was not accounted for when weighing out the sample material. The density of chalcopyrite is lower than pyrite ( $4.4 \text{ g/cm}^3$ ), resulting in a sample material with a true volume ratio silicate:iron sulfide, deviating from 1:1. For the purpose of this preliminary study, as the light minerals in the pyrite concentrate are liberated and thus behave similar to the quartz during the molding process, the light minerals from the pyrite concentrate is grouped with the quartz for the results handling. The material is grouped into two groups, iron sulfides and silicates, according to Table 2. Automated mineralogy results of a vertical section of the sample material show that the ratio by volume silicate:iron sulfide is not exactly 1:1, but close to 0.9:1. The true volume distribution in the sample material is 52.68 vol.% iron sulfides and 47.32 vol.% silicates (Table 2).

**Table 1.** Modal mineralogy of the two mineral concentrates forming the sample material.

Vol.%	Quartz concentrate	Pyrite concentrate
Quartz	>99.99	
Other silicates	<0.01	
Pyrite		86.7
Chalcopyrite		9.5
Other heavy phases ( $\rho > 4 \text{ g/cm}^3$ )		1.8
Light minerals ( $\rho < 4 \text{ g/cm}^3$ )		1.7
Unknown		0.3
<b>Total</b>	<b>100</b>	<b>100</b>

**Table 2.** Average modal mineralogy (vol.%) of the sample material from automated mineralogy analysis of vertical sections of six samples.

Vol.%	Silicates	Iron sulfides
Quartz	46.42	
Other silicates	0.90	
Pyrite		46.46
Chalcopyrite		5.10
Sphalerite		0.38
Pyrrhotite		0.52
Other heavy phases		0.04
Unknown		0.18
<b>Total</b>	<b>47.32 %</b>	<b>52.68</b>

Two types of graphite were used, fine grained and coarse grained. The fine grained ultra-pure graphite was acquired from Millipore Corp. (now Merck) under the brand: Graphite – fine powder extra pure. The properties of the ultra-pure graphite are given as 99.5 % <50  $\mu\text{m}$ , density 2.2  $\text{g/cm}^3$  at 20 °C (Merck, 2017). The coarse grained graphite was supplied by Dept. of Materials Technology, NTNU. The graphite was supplied as an unused (clean), pure graphite crucible. The crucible was crushed and sieved. Two size fractions were prepared to match the size fraction of the prepared sample material: 53-75  $\mu\text{m}$  and 104-147  $\mu\text{m}$ .

### 3.2 Methods

#### 3.2.1 Sample preparation

A rotary riffle splitter was used to make aliquots of the sample material of approx. 0.5 g. Each aliquot was weighed, and graphite was added in order to fulfill the sample:graphite ratios presented in Table 3, one set of samples was mixed with fine graphite, and one set with coarse graphite, totally 10 polished sections. The

1:3 sample to graphite wt. ratio was not made for the coarsest (104-147  $\mu\text{m}$ ) fraction, as Jackson et al. (1984) and common lab practices use more graphite in the small size fractions samples. One reference sample for each size fraction was made (QP1 and QP2), containing only quartz/pyrite concentrate and no graphite. Sample names and descriptions are presented in Table 4. Two duplicate samples were made to check the validity of the experiment. The samples were prepared as polished sections at the thin section laboratory at the Dept. of Geoscience and Petroleum, NTNU. The samples were molded in 25 mm diameter casts using epoxy resin Epotec 301 A and B (4:1), using as little epoxy resin as possible to avoid an excess epoxy resin layer above the particles. The sample was placed in a vacuum chamber to remove air bubbles, before cured in room temperature for 24 hours. A thin layer of carbon coating was applied. Note that due to large variations in sample volume (caused by the differing graphite content), the amount of epoxy differed for each sample. The amount of epoxy was added by freehand, just enough to make the sample adequately fluid. This resulted in slightly different (and uncontrolled) settling condition for the particles in the different samples. The issue is commented further in the discussion.

**Table 3.** Samples were prepared with the following sample:graphite ratio (weight). Respective approximate volume ratios are included for informational purposes.

Size fraction	Sample:graphite (weight)	Sample:graphite (volume)
53-75 $\mu\text{m}$	1:1, 1:2, 1:3	1:1.7, 1:3.5, 1:5
104-147 $\mu\text{m}$	1:1, 1:2	1:1.7, 1:3.5

**Table 4.** Sample overview. Sample names and description of content. The ratio in parentheses refers to the weight ratios in Table 3.

Sample name	Description
QP1	Reference sample. Silicate/iron sulfide concentrate, size 53-75 $\mu\text{m}$ . No graphite
QP2	Reference sample. Silicate/iron sulfide concentrate, size 104-147 $\mu\text{m}$ . No graphite
FG 53-75 $\mu\text{m}$ (1:1, 1:2 or 1:3)	Fine graphite mixed with silicate/iron sulfide concentrate size 53-75 $\mu\text{m}$ . Denoted by sample to graphite weight ratio.
CG 53-75 $\mu\text{m}$ (1:1, 1:2 or 1:3)	Coarse graphite size 53-75 $\mu\text{m}$ mixed with quartz pyrite concentrate size 53-75 $\mu\text{m}$ . Denoted by sample to graphite weight ratio.
FG 104-147 $\mu\text{m}$ (1:1 or 1:2)	Fine graphite mixed with silicate/iron sulfide concentrate size 104-147 $\mu\text{m}$ . Denoted by sample to graphite weight ratio.
CG 104-147 $\mu\text{m}$ (1:1 or 1:2)	Coarse graphite size 104-147 $\mu\text{m}$ mixed with silicate/iron sulfide concentrate size 104-147 $\mu\text{m}$ . Denoted by sample graphite to weight ratio.



### 3.2.2 Automated mineralogy analysis

The samples were analyzed in a Zeiss Ultra 55 LE SEM, with two Bruker Xflash 4010 EDS detectors. SEM settings: analysis time 1 s, accelerating voltage: 15 keV, beam current: 44  $\mu\text{A}$ .

The Bruker Esprit 1.9 software was used to set up automatic scans. The selected areas were imaged using the BSE signal. The Feature mode in the program was used to set up the analysis. The grains were discriminated based on BSE gray level contrast. The chemical composition was acquired using one EDS analysis per grain. Subsequent, the mineralogy was determined based on the chemical composition. A classification file with predefined approximate mineral stoichiometry was created in the Esprit software. In order to highlight the issue of touching particles and its effect on the liberation analysis, only a minimum of filters was applied. The same analytical settings and filters were used for all analysis.

The result files and images from the Esprit software constitute the basis for the following automated mineralogical analysis. Automated mineralogical analysis was carried out using the quantitative mineralogical software developed at NTNU, the Particle Texture Analysis (PTA) software (Moen et al., 2006). The minerals were categorized mainly as quartz or iron sulfides. Minerals other than quartz and iron sulfides, were put into these two categories based on theoretical densities;  $>4 \text{ g/cm}^3$  in iron sulfides,  $<4 \text{ g/cm}^3$  as silicates. Unclassified grains were found to be mainly heavy minerals. They were manually categorized in to the two categories based on gray level. The mineral liberation analysis measures how much of the area of a particle is covered with the mineral in question. The modal mineralogy is calculated based on the particle area covered by each mineral.

To show how the added graphite reduces the particle packing density, i.e. the area covered with particles relative to the background epoxy and graphite, ImageJ GNU software (Schneider et al., 2012) was used.

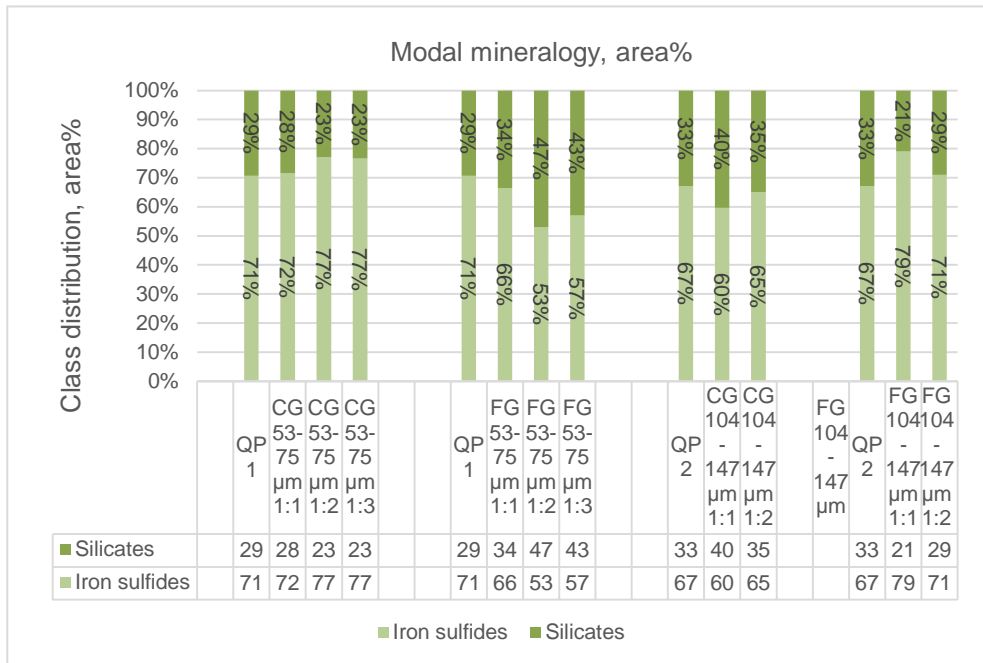
## 4. RESULTS

### 4.1 Modal mineralogy

The mineralogical composition of the sample material is known to be approx. 53 vol.% iron sulfide and 47 vol.% silicates. Due to the relative higher density of iron sulfide, it was expected that when preparing regular polished sections the material

would segregate and result in a higher concentration of iron sulfide in the sample surface.

Figure 3 shows the modal mineralogy results from the PTA analysis. In reference sample QP1, 71 % of the particle area is iron sulfides and 29 % is silicates. Reference sample QP2 show that 67 % of the area is iron sulfides and 33 % is silicates.

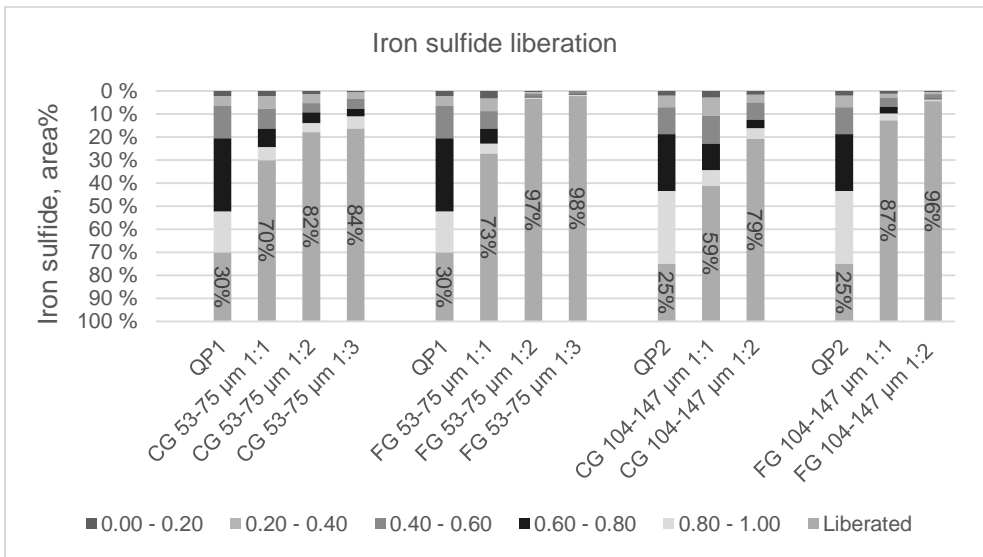


**Figure 3.** Modal mineralogy from PTA analysis. The areal distribution of silicates and iron sulfides.

The samples in the CG 53-75 μm series show >70 area% of iron sulfides for all sample:graphite ratios. In the FG 53-75 μm series, the sample with the 1:1 sample:graphite ratio show the most iron sulfides (66 area%) and the sample with 1:2 show the least (53 area%). The samples in the CG 104-147 μm series, both show >60 area% iron sulfides. The samples in the FG 104-147 μm series show >70 area% iron sulfides.

## 4.2 Mineral liberation

The studied sample material is known to contain only fully liberated particles of both iron sulfide and silicates. Hence, the optimum sample preparation (provided correct analytical settings) would result in an analytical result of 100 % fully liberated particles, both for the sulfide and silicate group. However, the liberation analysis will not (without applying filters) be able to distinguish actual unliberated particles from adjacent particles appearing to be one unliberated particle. Hence, due to touching particles, the measured degree of liberation is lower than the true liberation (Figure 4 and Figure 5).

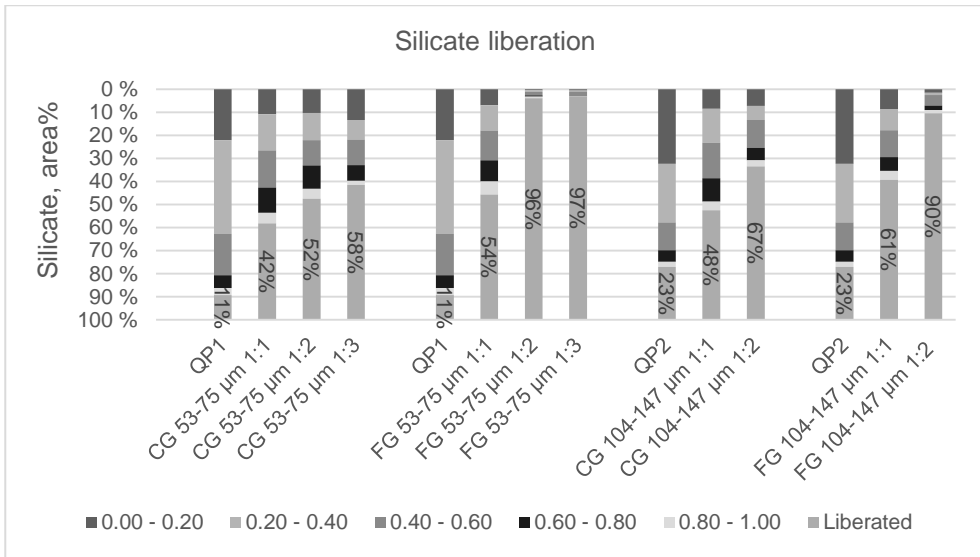


**Figure 4.** Iron sulfide liberation. Liberation classes in 20 % intervals. The liberation increases with added graphite. The effect is more prominent in the sample series mixed with fine graphite.

The iron sulfide liberation for all samples is shown in Figure 4. For reference samples QP1 and QP2 the liberation is 30 % and 25 %, respectively. Both fractions, 53-75 µm and 104-147 µm, show that the mineral liberation increase with increasing amount of graphite added. Fine graphite gives a higher liberation than the graphite.

Fine-grained graphite reaches liberations of > 95 area% fully liberated for sample:graphite ratios 1:2 and 1:3.

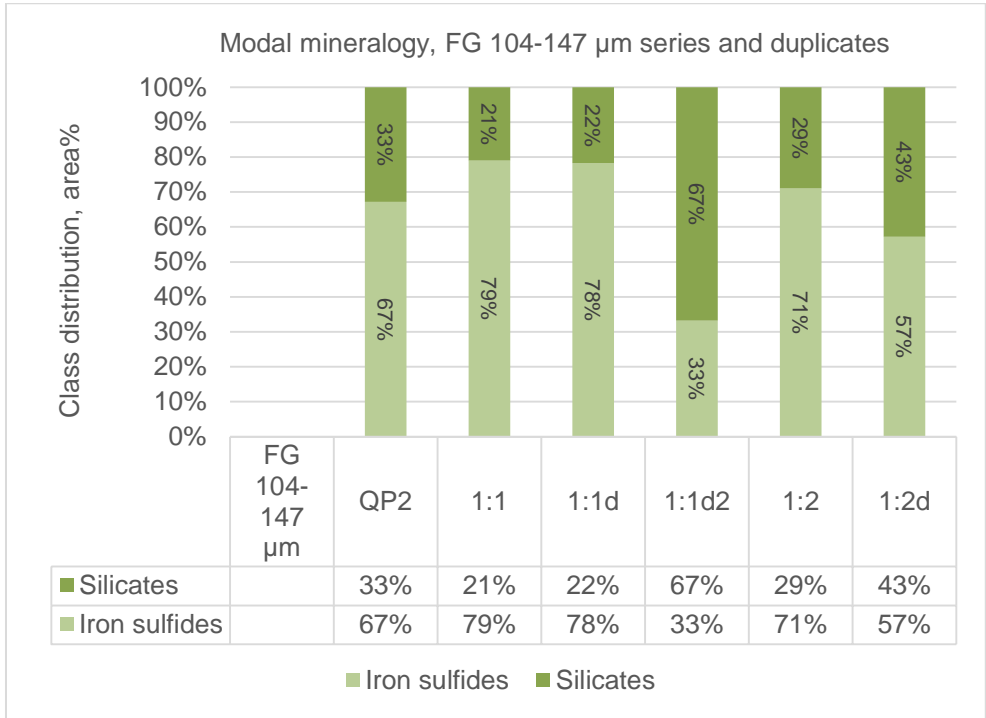
The silicate liberation is displayed in Figure 5. The two liberation diagrams show the same trend regarding liberation and graphite content. The silicate liberation is generally lower than iron sulfide liberation.



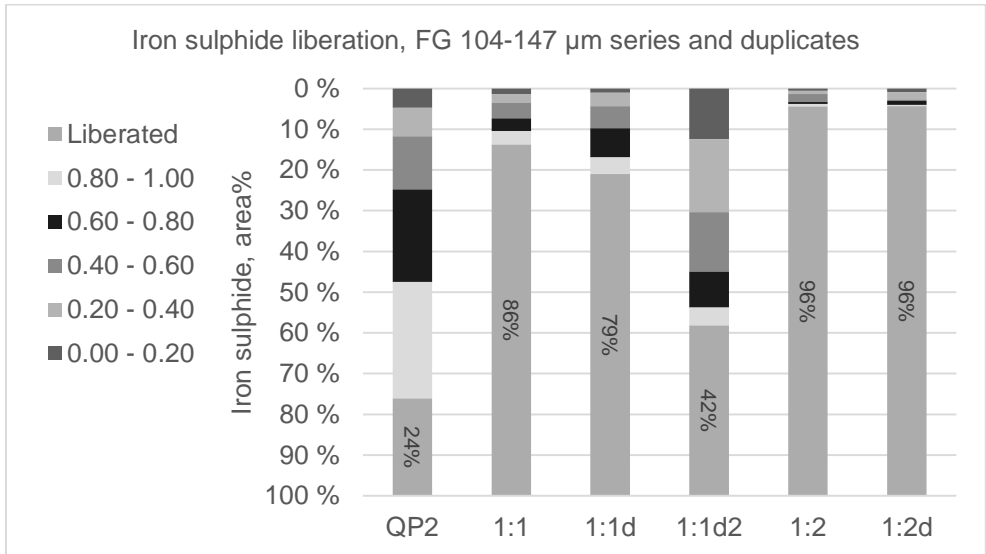
**Figure 5.** Silicate liberation. Liberation classes in 20 % intervals. The liberation increases with added graphite. The effect is more prominent in the sample series mixed with fine graphite.

### 4.3 Duplicate samples

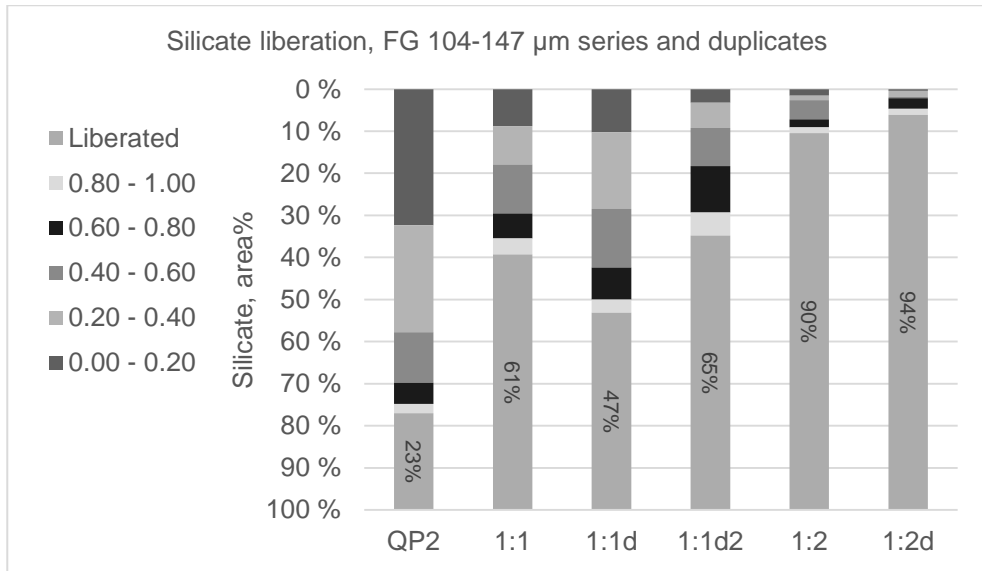
Two duplicate samples were made of FG 104-147 µm 1:1 and 1:2 to check reproducibility. One erroneous sample of FG 104-147 µm 1:1 is included. The erroneous sample, 1:1d2 show a relative high content of silicates (67 vol.%, Figure 6). The 1:1 and 1:1d show a relatively similar modal mineralogy, whereas 14 % distinguish the 1:2 and 1:2d (Figure 6). The number of liberated particles differs for the 1:1 and the 1:1d (Figure 7 and 8). The silicate liberation is notably lower than the iron sulfide liberation for these samples. For the 1:2 and 1:2d sample, the liberation (both silicate and iron sulfide) is more similar (Figure 7 and 8).



**Figure 6.** Modal mineralogy of samples from the FG 104-147  $\mu\text{m}$  fraction and duplicate samples.



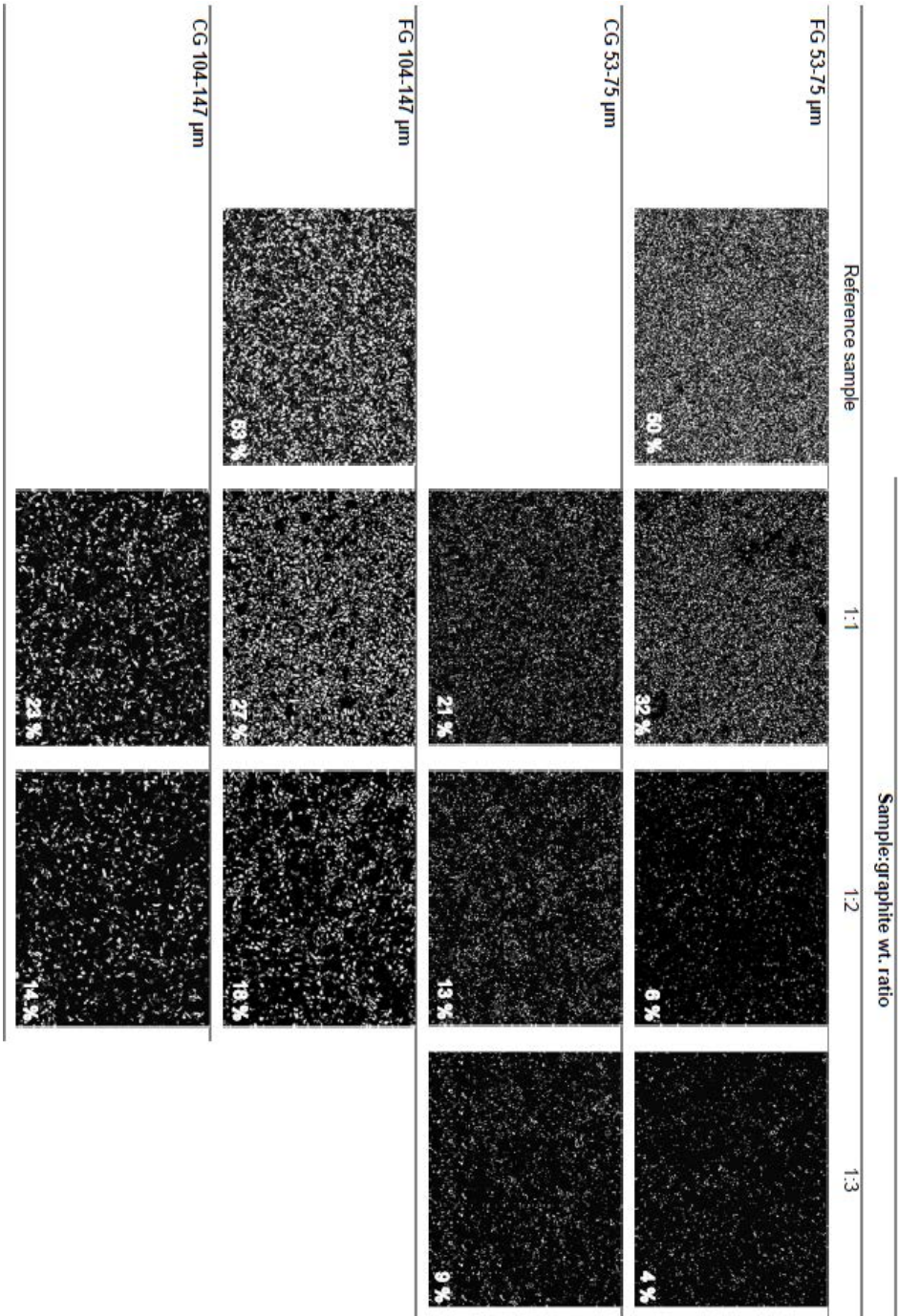
**Figure 7.** Iron sulphide liberation of samples from the FG104-147  $\mu\text{m}$  fraction and duplicate samples of 1:1 and 1:2.



**Figure 8.** Silicate liberation of samples from the FG104-147  $\mu\text{m}$  fraction and duplicate samples of 1:1 and 1:2.

#### ***4.4 Particle packing density***

As expected, the particle packing density is decreased when diluting the sample with more graphite. The reduction in analyzed particles by adding graphite is illustrated in Figure 9.



**Figure 9.** Stitched BSE images of all samples analyzed. Number of particles in the same field of view decreases with increasing graphite content.

## 5. DISCUSSION

### 5.1 Segregation

The object of this preliminary study was to quantify the effect of mixing different amounts of graphite with the sample material, on segregation and touching particles. The modal mineralogy (Figure 3) was used in order to estimate the effect of segregation. The sample material had a known content of approximately 47 vol.% silicates and 53 vol.% iron sulfides (Table 2). Ideally, this would be corroborated by the analytical results from the automated mineralogical analysis. However, as heavy particles will sink faster than light particles, the sample surface (the bottom of the mold) would most likely show a higher content of heavy minerals than the true content of the sample material. Hence, in this experiment, the iron sulfide content was expected to be overestimated. The higher the result for iron sulfide content, the more segregated the sample material is believed to be.

The results in Figure 3 demonstrate that the iron sulfide content is overestimated in all samples, except the FG 53-75  $\mu\text{m}$  1:2. This shows that mixing sample material with graphite does not necessarily prevent segregation. In the CG 53-75  $\mu\text{m}$  series, the iron sulfide content is higher in the samples mixed with graphite, than in the reference sample with no graphite (QP1), indicating that mixing coarse graphite with the 53-75  $\mu\text{m}$  fraction actually has aided segregation. Samples in the FG 53-75  $\mu\text{m}$  series show less segregation than the reference sample with no graphite (QP1). The sample from this series with the sample graphite ratio 1:2 actually shows the true modal mineralogy of the sample material. Considering the low particles packing density obtained using this high amount of graphite (Figure 9), 1:2 is not necessarily the best ratio. For the 104-147  $\mu\text{m}$  series, the results are opposite from the finer fraction; the samples mixed with fine graphite show a greater segregation than the reference sample, and the samples mixed with coarse graphite show a lower degree of segregation than the reference samples. None of the samples manage to reproduce the true modal mineralogy.

The presented results indicate that the best effect on preventing segregation was achieved using fine graphite for the 53-75  $\mu\text{m}$  sample material in a 1:2 wt. ratio. Whether this is the correct, coincidental, or an effect of lower polishing depth relative to the other samples, is not possible to determine based on this single experiment.



### ***5.2 Touching particles***

The true liberation of the sample material used in this study is known to be practically 100 %, as it consists of only monomineralic particles. However, the results from the mineral liberation analysis shows large variations in mineral liberation (Figure 4 and 5). The liberation is generally reported as lower than 100 %, demonstrating the issue of touching particles i.e. adjacent particles of silicates and iron sulfides are interpreted as one particle by the automated mineralogy software. When increasing the amount of graphite relative to sample material, the effect is reduced particle packing density (Figure 9). Consequently, the number of touching particles is reduced, and the measured liberation increased. Note that, for the purpose of this study, image processing was kept to a minimum and no de-agglomeration was conducted. Hence, measured liberation results are considerably lower than what would be expected for a routine analysis.

The fine graphite causes higher measured liberation than the coarse graphite (Figure 4 and 5), and is thus found to be the better choice to avoid touching particles. The results closest to the true liberation (100 % liberation), are for the FG 53-75  $\mu\text{m}$  1:2 and 1:3 (e.g. iron sulfide liberation: 96.5 % and 97.7 %, respectively, Figure 4). These are also the samples with the lowest particle packing density (6 % and 4 % respectively, Figure 9). The optimal sample:graphite weight ratio is probably below 1:2, where the particle packing density is higher, and the liberation results are still fairly high. Generally, increasing the particle packing density also increases the risk of more touching particles. A compromise between the two parameters is necessary to avoid analyzing an excessive number of samples in order to obtain adequate particle statistics. As previously mentioned, the issue of touching particles might be minimized further by others using more filters, and de-agglomeration functions of the automated mineralogy software.

The liberation analysis can be affected by the segregation, as the ratio of heavy and light minerals in the sample surface of a segregated sample is altered and the modal mineralogy is reported erroneously; overestimating heavy minerals and underestimating light minerals. Comparing the liberation of iron sulfides and silicates (Figure 4 and 5), the iron sulfide liberation is always reported higher than the corresponding silicate liberation. As a result of the segregation, all the samples except FG 53-75  $\mu\text{m}$  1:2, show a higher content of iron sulfide particles (Figure 3) in the sample surface than the true iron sulfide content of the sample material. Hence, the silicate particles will have a higher risk of touching iron sulfide surfaces, than iron sulfide particles will have touching silicate surfaces. The measured liberation will only be affected when the touching particles consist of adjacent mineral grains of different minerals. Touching particles consisting of grains of the same mineral will not affect the liberation. In the case of FG 53-75

$\mu\text{m}$  1:2 and 1:3, the samples with the most correct modal mineralogy (Figure 3), the liberation of silicates and iron sulfides is equal (Figure 4 and Figure 5).

The samples in this preliminary study demonstrate that the measured liberation for a specific mineral is influenced by segregation. For sample material containing minerals/particles of different densities and/or size/shape representative liberation result is dependent on a representative sample preparation, i.e. no segregation occurring (when preparing regular polished sections). Note that correct modal mineralogy results do not necessarily confirm correct liberation results.

### ***5.3 Quality of results***

This study is based on a limited number of samples, and thus, must be regarded as a qualitative. However, the results illustrate the importance of awareness during sample preparation, as the results are strongly dependent on preparation conditions.

The representativeness of the results is limited by the fact that amount of epoxy was not controlled. Epoxy was added by experienced lab personnel with instructions of adding only enough, as stated by Jackson et al. (1984). This approach resulted in uncontrolled variations in the epoxy column. The graphite ascended during molding, creating a layer of only graphite and epoxy at the top of the samples, hence making it unfit to make exact comparisons between samples, as the layer is of variable thickness. It is possible that the amount of epoxy has a greater effect on the segregation than the amount of graphite. It is not possible to prove how much of the segregation is a result of graphite content or an effect of epoxy content. The liberation results do, however, follow a logical trend.

The sample polishing depth is difficult to control with high precision. Hence, different polishing depths might result in different results due to segregation. An evident example of this is the sample FG 104 147  $\mu\text{m}$  1:1d2 where the modal mineralogy result shows 67 % silicates and 33 % iron sulfides (Figure 6). As iron sulfide have a higher specific density than silicates, it is expected that iron sulfide particles, and not silicates, will accumulate at the bottom when preparing regular polished sections. In this case, the sample have been ground to a level beyond most of the sulfide particles, leaving mostly silicate particles, and thus discarded. Making vertical sections instead of regular polished sections will eliminate the sources of error related to segregation.

The two duplicates FG 104 147  $\mu\text{m}$  1:1 and 1:1d show similar modal mineralogy, but different liberation, whereas the two duplicates FG 104 147  $\mu\text{m}$  1:2 and 1:2d, show different modal mineralogy, but similar liberation characteristics (Figure 6, 7 and 8). In the latter case, the similarity in liberation (despite difference in modal

mineralogy) is possibly due to the high amount of graphite (1:2 sample:graphite wt. ratio), i.e. adding this high amounts of graphite to a sample will result in low numbers of touching particles. Consequently, the effect of changed modal mineralogy (due to segregation) on liberation results will be minimized. The differences in liberation and modal mineralogy are thought to be a result of a lack of control on the epoxy resin:sample ratio. Consequently, the segregation process has occurred under different conditions in different samples. Also the curing time for the different samples might have varied slightly, as they were prepared in batches.

#### ***5.4 Overall discussion***

The amount of graphite suggested by Jackson et al. (1984) is, based on this preliminary study and general experience from sample preparation, believed to be excessive. The number of particles is drastically reduced for sample:graphite wt. ratios 1:2 and 1:3 (Figure 9). For samples FG 53-75  $\mu\text{m}$  1:2 and 1:3, the iron sulfide liberation (Figure 4) is very high (>96 % fully liberated grains). The small increase in liberation from the 1:2 to 1:3 ratio (1 2 % for iron sulfides) is considered insignificant due to the relative large error margins of this study. However, this indicates that the added graphite will contribute more to reduce the particle density than aid liberation, and the optimal balance is probably found below the sample:graphite wt. ratio 1:2. The reported liberation is generally lower for the coarser fraction samples, but the same trend is shown regarding difference in fine and coarse graphite.

Regarding the different graphite types, it is not possible to definitely state that one is better suited to prevent segregation and touching particles, due to the lack of control of the epoxy resin:graphite:sample ratio. The experience made during the sample preparation indicates that the fine graphite can contribute to a higher viscosity in the epoxy resin, thus preventing segregation. The modal mineralogy results for the 53-75  $\mu\text{m}$  fraction support this, showing results closer to the true modal mineralogy (i.e. less segregation has occurred) with the higher amounts of fine graphite (Figure 3). This is not the case for the 104-147 $\mu\text{m}$  fraction, however there are indications this might be due to use of excessive epoxy resin. Overall, the liberation results show that the fine graphite has a better effect on preventing touching particles than the coarse graphite (Figure 4 and 5). The fine graphite seems to be the better choice based on the viscosity effect and ability to separate the particles. It is also preferred due to convenience and availability.

Particle behavior during the molding process is affected by many factors. Some factors may be standardized: resin viscosity, curing time, polishing depth, graphite type and epoxy resin:graphite ratio, and some are material specific properties:

specific gravity, particle shape, particle size, and particle size distribution. This makes it difficult to establish an optimal universal epoxy resin:graphite:sample ratio so that samples of differing mineralogical content can be compared without making vertical sections. As the volume of a mineral particulate varies with the density of the minerals, the optimal epoxy resin:graphite:sample ratio would be given in volumes, not weight. An alternative solution can be to find the epoxy:graphite ratio where the least segregation occurs, and keep this constant (applies to the use of fine grained graphite), while using a fixed weight of sample material. However, this may cause relative bias in liberation analysis within a sample series with strong variation in mineral densities, resulting in different particle packing densities, and hence variations in the risk of touching particles.

Preparation of polished sections, without making vertical sections, is sensitive to polishing depth. Samples are not always polished to the exact same depth. Although the same procedure was followed for all samples in this study, some required more polishing than others. Some of the initial samples showed very little iron sulfides, that due to the higher density of iron sulfides, descended faster than the silicates, and accumulating in the bottom of the mold, and thus being polished off (sample 1:1d2, Figure 6). These samples were discarded.

The sample preparation procedure is very sensitive. Adding graphite can minimize the number of touching particles. The only way to avoid the problem of segregation and polishing depth seems to be to make vertical sections of the material. Adding graphite can limit the segregation effect in certain cases, but the effect is variable and should be further investigated. The epoxy resin:graphite:sample ratio should be controlled.

## **6. CONCLUSIONS**

The results and experience from this preliminary study indicates that fine graphite is the better choice to use as filler in polished sections of particulate materials. Compared to the coarse graphite, the fine graphite has the best ability to separate grains, and to some degree prevents segregation. To avoid biased results due to segregation and difficulties of controlling polishing height, preparing vertical sections is advised for most studies of mineral liberation and modal mineralogy.

The results show a trend that strong segregation might occur regardless of use of a graphite filler. Using graphite as a filler does not necessarily prevent segregation, at least when not considering the epoxy resin:graphite:sample ratio. To acquire reliable results from regular polished sections, if even possible, a strictly controlled

epoxy resin:graphite:sample ratio is required. The uncertainty introduced by polishing will always be present for regular polishing sections.

Adding more graphite can improve the results from the liberation analysis, by increasing space between particles and thus preventing touching particles, both for regular polished sections and vertical sections. The balance between particle packing density (i.e. number of particles in section) and liberation should be considered for the individual purpose.

The amount of graphite relative to sample material suggested in Jackson et al. (1984) is believed to be excessive. Considerably smaller amounts of graphite should be used.

This preliminary study confirms the importance of awareness in sample preparation when working with automated mineralogical analysis of polished sections of mineral particulates. The sample preparation procedure is crucial for the result of quantitative mineralogical investigations. Results based on different preparation procedures cannot necessarily be directly compared.

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