Effect of Particle Shape on the Stereological Bias of the Degree of Liberation of Biphase Particle Systems

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It is well known that in sectional measurements of polished ore samples, the degree of liberation is overestimated by stereological bias. The stereological bias is affected by particle shape, but the effect of particle shape on the stereological bias has not been studied systematically. In this study, particles of various shapes were modeled using a geodesic grid, and the internal structures of the particles were randomly designed. The stereological bias of liberation was assessed quantitatively by comparing the computed sectional information and the original three-dimensional information. The following results were obtained: 1) the effect of aspect ratio on the stereological bias is less than 12% when comparing cases with \( \alpha \) ranging from 1.0 to 2.0; 2) the effect of particle surface roughness on the stereological bias is smaller than 7.6% when comparing cases with surface roughness (the quotient of the surface area of the volume equivalent ellipsoid and the surface area of a particle) ranging from 0.833 to 0.996. It was also confirmed that the previously proposed stereological correction method is applicable to irregularly shaped particles because the estimation error of the degree of liberation dramatically dropped from 56.4%–64.4% without any correction to 1.16%–3.41% using the proposed method. [doi:10.2320/mtatertrans.M-M2016837]

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1. Introduction

One of the primary purposes of comminution in mineral processing is to achieve valuable mineral liberation at the coarsest particle size possible. This makes it possible to save energy by reducing fine powder production and reducing the cost and effort in the separation process. It is also important to accurately assess the degree of liberation of the comminuted products to feedback correct information and to develop a mineral processing strategy.

In general, to assess the degree of liberation, comminuted materials are hardened with resin and cut, and the polished section is analyzed using an optical microscope or a dedicated analyzer, such as the mineral liberation analyzer\(^1\). This two-dimensional (2D) sectional analysis overestimates the degree of liberation, which is called the "stereological bias." In principle, the stereological bias is unavoidable because in the cutting process, locked particles may become free but free particles cannot become locked\(^2\).

The stereological bias is influenced by the internal particle structure. The stereological bias is large in simply locking biphase particles and negligibly small in particles with complex internal structures such as intergrowth\(^3\)–\(^5\). Several studies have been conducted on the stereological bias using spherical particles\(^6\)–\(^7\) and simple regular shaped particles\(^8\)–\(^9\). Because the majority of particles handled in the mining and recycling industries do not have spherical or regular shapes, it is beneficial to study the effect of particle shape on the stereological bias. Remarkable approaches in modeling random particle shape include using Poisson polyhedra\(^10\) and PARGEN program wherein the particles are generated through a process that resembles nucleation and growth\(^11\). In this study, we propose a new method to model random irregular particle shape with a given value of the aspect ratio and the surface roughness.

A stereological correction method using an integral transformation with the kernel function, which is applicable to irregularly shaped particles, has been proposed\(^12\)–\(^14\). In general, these studies successfully predict the effect of the stereological bias on the liberation distribution of well-examined samples; however, a further systematic investigation is required to confirm their versatility.

Computational simulation is an effective tool in the systematic analysis of the stereological bias because it can accurately and rapidly simulate various patterns of the internal particle structure. To date, methods to model particles with agglomerates of cubic elements with various grain types\(^12\) and with spherical particles hollowed out from a Boolean based biphase structure\(^15\) have been proposed. We have developed a unique numerical approach wherein the stereological bias of degree of liberation was quantitatively assessed by comparing the three-dimensional (3D) particle internal structures with 2D sectional structures\(^16\), a correlation between the 2D texture characteristics and the 3D liberation state was determined\(^17\), a stereological correction method was established in which particle sectional textures are assessed, and the 3D liberation state was estimated only from the 2D measurable parameters using a preliminary computed correlation between 2D texture characteristics and 3D liberation state\(^17\). Since our numerical simulation has been conducted on spherical binary particle systems, its applicability to more realistic-shaped particle systems needs to be investigated.

In this study, we conducted a series of simulations of the stereological bias of the liberation assessment of particles with various shapes. The goal of this study is to evaluate the effect of particle’s aspect ratio and surface roughness on the stereological bias of degree of liberation and to assess the applicability of the proposed stereological correction method to irregularly-shaped biphase particle systems.
2. Methodology

2.1 Particle modeling

2.1.1 Irregularly shaped particles

Particle shape is quantified using several indices. In this study, we used two indices: aspect ratio ($\alpha$), which represents the general shape of a particle and the surface roughness ($\rho$). $\alpha$ and $\rho$ are defined as follows:

\[
\alpha = \frac{a}{c},
\]

\[
\rho = \frac{A_c}{A},
\]

where $a$ and $c$ denote the lengths of the long axis and the short axis, respectively, and $A$ and $A_c$ denote the surface areas of a particle and of an ellipsoid that has the same volume and aspect ratio as the particle, respectively.

The irregularly shaped particles were modeled using a “geodesic grid,” which is a method to subdivide a sphere using polyhedrons. First, a regular icosahedron inscribed in a sphere was computed (Fig. 1(a)), which is called the first generation. Second, midpoints of all sides were projected on the sphere and new faces were created (Fig. 1(b)), which is called the second generation. This process was continued until the necessary resolution was obtained. The third generation model was used to model particles in this study. Note that a third generation particle has 162 nodes and 320 faces. This procedure was applied to ellipsoids with several values of aspect ratios. Because the aspect ratios of particles produced by brittle fracture take wide range of values but the average is generally less than 2.018, we investigated particle models with $\alpha/\text{uni00A0}_0 < 2.0$ in this study.

Then, the position of each node was dispersed toward the direction from the center of mass of the particle to the node with the following value:

\[
\text{Dispersion} = \frac{a\gamma}{2},
\]

where $\epsilon$ is a uniform random number distributed from $-1$ to $1$ and $\gamma$ is the dispersion parameter. Figure 2 shows spheres based on third generation particles with $\gamma = 0.0$ (no dispersion), 0.5, and 1.0. In this study, the surface roughness of the particles was controlled using various $\gamma$ values.

2.1.2 Internal structure of biphase particles

Particles composed of two phases, phases $A$ and $B$, were modeled as follows. Figure 3 shows the modeling method schematically.

1) A total of 7,463 virtual spheres whose diameter ($d_A$) ranged from 1.0 to 2.0 were generated at random positions in a cuboid with a width of 30, a depth of 30, and a height of 20, and the virtual spheres were freely fallen using DEM (Fig. 3(a)). The simulation continued until the entire kinematic energy of the virtual spheres became negligibly small.

2) The third-generation geodesic grid modeled particles were replaced with randomly orientated virtual spheres (Fig. 3(a)). The particle size was set to not exceed the virtual sphere size. Figure 4 shows the modeled particle assembly as an example.

3) The core phase elements modeled by the first-generation geodesic grid particles were generated using a similar procedure to that in the cuboid. Note that this process was conducted independently of the particles and the interaction forces between the particles and the core phase elements were neglected; therefore, the core phases can overlap the particles. Figure 3(b) shows the phase $A$ elements and their virtual spheres together with the posi-
The particles and the core phases were superposed, and the domain in the particle overlapping the core phase elements was called phase A, while the remaining domain in the particle was called phase B, allowing biphasic particles with phases A and B to be modeled (Fig. 3(c)). These processes can be interpreted as hollowing particles from biphasic materials with phases A and B assuming non-preferential breakage. For convenience, the initial state of phase A (Fig. 3(b)) will be called the "phase A element" and the domains of phases A and B (Fig. 3(c)) in the particles will be called "phase A domain" and "phase B domain", respectively.

This process is similar to that of Gay6) in the sense that the particles are hollowed from biphasic materials; however, it is unique in the modeling of randomly packed particle structures using DEM and irregularly shaped particles. DEM is effective in providing randomly sectioned particles by a single sample sectioning.

Note that various types of 3D biphasic structure model has been proposed: the Boolean model6,15), the Poisson mosaics20–22), and the Voronoi tessellation20–22). All of them provide more complex structure than the abovementioned method. Considering the goal of this study of investigating the effect of the particle shape on the stereological bias and on the stereological correction method described below, the abovementioned 3D structure plays a sufficient role.

The sectional information of the biphasic particle assembly was calculated using the Monte Carlo method with solid angle calculations, as discussed later. Because the sectional information calculated from different sections varies, this variation should be taken into account along with the stereological bias when comparing sectional information with 3D original information. To avoid the effect of dispersion of each section and investigate the influence of only the stereological bias, multiple sectional information sets should be calculated and statistically processed. With a greater number of calculated sections, the influence of dispersion of each section will lessen; however, this requires more calculation time. In this study, we calculated 20 sections per sample.

### 2.2 Liberation assessment

#### 2.2.1 Volume and area estimation via the Monte Carlo method with solid angle calculations

The Monte Carlo method was applied to estimate the volume (or area) of the particles and phase A domains. A number of plots were set at random positions in a volume (or area) including the particles and the phase domain (or their section) and volume (or area), and the volume (or area) of the particles was estimated from the ratio of the numbers of plots inside or outside of the particles and the volume (or area) of the domain. The solid angle calculation described later was used to judge whether the plot was inside or outside the particles. It is possible to estimate the volume (or area) of irregularly shaped particles using the Monte Carlo method; however, its accuracy depends on the number of plots. The effect of the number of plots on the estimation accuracy of the Monte Carlo method was investigated using the error rate ($E_v$) as follows:

$$ E_v = \frac{100|V_{\text{geo}} - V_{\text{mont}}|}{V_{\text{geo}}} $$

where $V_{\text{geo}}$ and $V_{\text{mont}}$ are the volumes (or areas) calculated geometrically and estimated via the Monte Carlo method, respectively.

Figure 5(a) and 5(b) plot $E_v$ versus the number of Monte Carlo plots when a sphere and a circle are inscribed in a cube and a square, respectively. The number of plots was increased from 50 in steps of 100, and the average values and standard derivations of every 100 cases are shown as plots and error bars, respectively, in Fig. 5. The error rate decreases with the increasing number of plots in both cases. We applied 80,000 plots in the 3D calculation and 20,000 plots in the 2D calculation as a standard number of plots, in terms of adequately under-run error rate, 0.5% $E_v$. In the 3D calculation, 80,000 plots were used in the cube circumscribed to a virtual sphere of each particle. In the 2D calculation, the sectional area of the particle varies according to the position of the section. Using the radius of the sectional circle of the virtual sphere ($r_1$) and the radius of the virtual sphere ($r_2$), $20,000 \times (r_1/r_2)^2$ plots were used in a square circumscribed to the sectional circle.
To judge whether the Monte Carlo plots were inside or outside the particles, the solid angle (Ω) of the particle surface was calculated as follows:

$$\Omega = \int_S \frac{t \cdot n}{r^2} dS = \begin{cases} 0 & \text{ (when the plot is outside S)} \\ \frac{4\pi}{(4\pi)^2} & \text{ (when the plot is inside S)} \end{cases}$$

(5)

where $S$ is the particle surface as a closed surface, $t$ is the magnitude, $t$ is the unit position vector, and $n$ is a unit normal vector of the position vector of a micro region on the particle surface.

### 2.2.2 Assessment of the degree of liberation and the stereological bias

In 2D, total sectional areas of particles ($A_P$), phases A domain ($A_A$), phase B domain ($A_B$), apparently liberated phase A domain ($A_{Alib}$), and apparently liberated phase B domain ($A_{Blib}$) in an arbitrary sample section were calculated through the process in subsection 2.2.1.

In 3D, similarly to 2D, total volumes of particles ($V_P$), phase A domain ($V_A$), phase B domain ($V_B$), liberated phase A domain ($V_{Alib}$), and liberated phase B domain ($V_{Blib}$) in the cuboid were calculated.

In statistical processing of the 2D and 3D information, the area fraction and volume fraction of the phase $F_A$ and $F_v$ for phases A and B ($L_{2D}^A$, $L_{2D}^B$) and the liberation in 3D for phases A and B ($L_{3D}^A$, $L_{3D}^B$) were calculated as follows.

$$F_A = \frac{1}{n} \sum A_A$$

(6)

$$F_v = \frac{V_A}{V_P}$$

(7)

$$L_{2D}^A = \frac{1}{n} \sum A_{Alib}$$

(8)

$$L_{2D}^B = \frac{1}{n} \sum A_{Blib}$$

(9)

$$L_{3D}^A = \frac{V_{Alib}}{V_A}$$

(10)

$$L_{3D}^B = \frac{V_{Blib}}{V_B}$$

(11)

Here, $n$ is the number of sections. All the parameters have values ranging from zero to one, and further, $L_{2D}^A \geq L_{3D}^A$ and $L_{2D}^B \geq L_{3D}^B$. For convenience, the generic term of $L_{2D}^A$ and $L_{2D}^B$ will be called $L_{2D}$ and similarly the generic term of $L_{3D}^A$ and $L_{3D}^B$ will be called $L_{3D}$.

In addition, to assess the stereological bias quantitatively, the difference between the degrees of apparent liberation in 2D and those of liberation in 3D for phases A and B ($L_{2D-3D}^A$, $L_{2D-3D}^B$) and the ratio of the overestimated liberation in 2D for phases A and B ($\sigma_A$, $\sigma_B$) were calculated as follows.

$$\sigma_A = \frac{L_{2D-3D}^A}{L_{2D}^A}$$

(14)

$$\sigma_B = \frac{L_{2D-3D}^B}{L_{2D}^B}$$

(15)

$L_{2D-3D}^A$, $L_{2D-3D}^B$, $\sigma_A$, and $\sigma_B$ all have values ranging from zero to one. For convenience, the generic term of $\sigma_A$ and $\sigma_B$ will be called $\sigma$.

### 2.3 Stereological correction method

A stereological correction method using the fractal dimension ($\delta$) of the virtual surface area of the image intensity has been developed. A brief introduction to the method is given below.

First, $\delta$ is obtained as follows:

A) On the cross-section of a sample, squares of size $d_{max}$ (maximum particle diameter) are superimposed on each particle section (Fig. 6 (a) (after17)).

B) Each square is divided into $N^2$ small squares with size $r = d_{max}/N$ (Fig. 6 (b)).

C) On the small squares enveloped in the particle cross-section (the shaded area in Fig. 6 (b)), the image intensity surface area is estimated via a summation of the areas of triangles ABD and BCD (Fig. 6 (c)). The summation of all the image intensity areas is defined as $A(r)$.

D) Fifty values of $A(r)$ with $N$ ranging from 1 to 50 are calculated. $\delta$ is obtained from eq. (16) with the fitting line deduced from the least-squares method of the $A(r)$ plots on a double logarithmic chart:

$$\log A(r) = (2 - \delta) \log r + C,$$

(16)

where $C$ is a constant.

Second, the ratio of the overestimated liberation in 2D ($\sigma$) is estimated using Fig. 7 (after17) $\sigma_B$ is shown here, which is an isogram of $\sigma$ obtained from the encompassing calculation of 2,764 types of biphase spherical particles17. $\sigma$ value estimated from Fig. 7 is defined as $\sigma'$ for convenience.

Finally, the degree of liberation in 3D is estimated using the following equation with $\sigma'$ and $L_{2D}^D$.

$$L_{3D}^D = (1 - \sigma')L_{2D}^D,$$

(17)

where $L_{3D}^D$ is the estimated degree of liberation in 3D.

![Fig. 6 Estimation of image surface area $A(r)$. (a) $d_{max}$ sized square mesh are superimposed on particle cross-sections. (b) The square is divided into $r$ mesh, where the mesh elements enveloped into particle cross-sections are shaded. (c) Surface area of the image intensity of mesh elements in particle cross-sections at a resolution of $r$ is calculated.](image-url)
3. Numerical Simulations

A total of 12 types of particles were designed, where the particles were modeled using the third generation of a geodesic grid with three values of aspect ratio ($\alpha$), 1.0, 1.5, and 2.0, and four values of dispersion ($\gamma$), 0.0, 0.5, 1.0, and 1.5. Figure 8 shows the surface roughness ($\rho$) and $\alpha$ of the 12 types of particles together with their parameters. The ratio of lengths of the long axis ($a$) and short axis ($c$) is determined by $\alpha$, and length of the middle axis ($b$) is designated as the geometric mean of $a$ and $c$, $\sqrt{ac}$. In the case of $\alpha /=2.0$, $a$ is equal to the diameter of the vertical sphere ($d_A$), and $b$ and $c$ are calculated from the abovementioned ratio. In cases with $\alpha /=1.0$ and 1.5, $a$, $b$, and $c$ are determined from the particle volume of the corresponding $\alpha /=2.0$ case satisfying the abovementioned length ratio.

The phase $A$ element is modeled using a first-generation sphere-based geodesic grid with $\gamma /=0.0$. As shown in Table 1, two types of sizes and distributions of phase $A$ elements are computed.

4. Results and Discussion

4.1 Simulation results

Figure 9 shows part of a section of a simulation sample with type 11 particles ($\alpha /=2.0, \rho /=0.914$) and small phase $A$ elements as an example. Middling particles and apparently liberated particles with phases $A$ and $B$ were successfully computed in a random manner.

4.2 Effect of the aspect ratio

Figure 10 compares $L_A^{2D-3D}$ and $L_B^{2D-3D}$ for various values of $\gamma$ and $\alpha$. Regardless of the $\gamma$ values, the $L_A^{2D-3D}$ of large phase $A$ elements becomes larger than that of small phase $A$ elements, in agreement with the previous study$^5$. The $L_A^{2D-3D}$ of large phase $A$ elements is dispersed with $\gamma$ values. This is because the number of phase $A$ elements is small in this case (Table 1) and the liberation state variations from each particle shape has a considerable effect on the cumulative $L_A^{2D-3D}$ values. The $L_A^{2D-3D}$ of particles with $\alpha /=2.0$ becomes 0.014 (12%) larger than that of particles with $\alpha /=1.0$ in this case. Meanwhile, the $L_A^{2D-3D}$ of the small phase $A$ element case slightly decreases with increasing $\alpha$. The $L_A^{2D-3D}$ of particles with $\alpha /=2.0$ becomes 0.0056 (7.5%) smaller than that of particles with $\alpha /=1.0$ in this case.

<table>
<thead>
<tr>
<th>Type</th>
<th>Phase $A$ element size ($d_A$)</th>
<th>Volume fraction*</th>
<th>Number of phase $A$ elements</th>
</tr>
</thead>
<tbody>
<tr>
<td>Small phase $A$ element</td>
<td>1.20</td>
<td>0.152</td>
<td>1,290</td>
</tr>
<tr>
<td>Large phase $A$ element</td>
<td>1.90</td>
<td>0.151</td>
<td>325</td>
</tr>
</tbody>
</table>

* Volume fraction here represents that of the initial condition (Fig. 3(b)).
On the other hand, the $L_{A}^{2D-3D}$ of large phase $A$ elements becomes smaller than that of small phase $A$ elements regardless of $\gamma$ values, also in agreement with the previous study (5). The $L_{B}^{2D-3D}$s of both the large and small phase $A$ element cases slightly increase with increasing $\alpha$. The $L_{B}^{2D-3D}$ of particles with $\alpha = 2.0$ becomes $0.0030 (1.3\%)$ larger than that of particles with $\alpha = 1.0$ when the phase $A$ element is small. Similarly, it becomes $0.0099 (7.4\%)$ larger when the phase $A$ element is large.

To summarize the above results, the effect of the aspect ratio on the stereological bias is less than $12\%$ when comparing cases with $\alpha$ ranging from 1.0 to 2.0.

### 4.3 Effect of surface roughness

Figure 11 compares $L_{A}^{2D-3D}$ and $L_{B}^{2D-3D}$ for various values of $\alpha$ and surface roughness ($\rho$). In Fig. 11, $L_{A}^{2D-3D}$ of large phase $A$ elements is dispersed with $\gamma$ values for the same reason discussed for Fig. 10. First, let us compare cases with $\rho = 0.996$ (for $\gamma = 0.0$) and $\rho = 0.833–0.882$ (for $\gamma = 1.5$). When the phase $A$ element is large, $L_{A}^{2D-3D}$ with $\alpha = 1.5$ increases but those with $\alpha = 1.0$ and 2.0 decrease with decreasing $\rho$, and $L_{A}^{2D-3D}$ decreases $0.0099 (7.6\%)$ on average. When the phase $A$ element is small, $L_{A}^{2D-3D}$ with $\alpha = 2.0$ increases slightly but those with $\alpha = 1.0$ and 1.5 decrease slightly with decreasing $\rho$, and $L_{A}^{2D-3D}$ decreases $0.0023 (3.2\%)$ on average.

On the other hand, in Fig. 11, $L_{B}^{2D-3D}$s of both large and small phase $A$ element cases increase slightly with decreasing $\rho$. The $L_{B}^{2D-3D}$ with $\rho = 0.833–0.882$ becomes $0.0042 (3.1\%)$ larger than that with $\rho = 0.996$ when the phase $A$ element is large. Similarly, it becomes $0.011 (4.8\%)$ larger when the phase $A$ element is small.

In summary, the effect of the particle surface roughness on the stereological bias of the liberation is smaller than $7.6\%$ when comparing cases with $\rho$ ranging from 0.833 to 0.996.

### 4.4 Stereological correction

Stereological correction method in subsection 2.3 was applied to the large and small phase $A$ cases (Table 1) with 12 types of particles (Fig. 8). Phase $B$ liberation was assessed here because the isogram of $\sigma_{B}$ (Fig. 7) was presented in Ref. 17. The computational simulation results show that for small phase $A$ elements, $F_{x} = 0.1514–0.1518$ and $\delta = 2.217–2.222$. Similarly, for large phase $A$ elements, $F_{y} = 0.1516–0.1522$ and $\delta = 2.217–2.222$. Here, the small phase $A$ element case is included in the map in Fig. 7, but large small phase $A$ element case is not included. In a future study, we will enlarge the map area with additional simulation cases, but here just the small phase $A$ element case was validated. The estimation error of the degree of liberation ($E_{lib}$) was compared between cases with and without stereological correction.

\[
E_{lib} = 100 \left( \frac{|L_{lib}^{B} - L_{lib}^{BP}|}{L_{lib}^{B}} \right),
\]

where $L_{lib}^{BP}$ equals $L_{lib}^{B}$ in the case without correction and $L_{lib}^{BP}$ in the case with correction.

Figure 12 compares the $E_{lib}$s of 12 types of particles (Fig. 8). The $E_{lib}$s without the stereological correction have values of $56.4–64.4\%$, while after the proposed correction,
they dramatically decrease to 1.16–3.41%. Therefore, the proposed stereological correction method is applicable to irregularly shaped particles, although further validation with various types of particles is required.

5. Conclusions

(1) The effect of the aspect ratio on the stereological bias of degree of liberation is less than 12% when comparing cases with $\alpha$ ranging from 1.0 to 2.0.

(2) The effect of the particle surface roughness on the stereological bias of liberation is less than 7.6% when comparing cases with surface roughness ranging from 0.833 to 0.996.

(3) Applying the proposed stereological correction method to irregularly shaped biphase particle systems, the estimation error of the degree of liberation dramatically dropped from 56.4%–64.4% without any correction to 1.16%–3.41% with the proposed method.

REFERENCES

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