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Original Research Paper

Experimental analysis of mineral liberation and stereological bias based on X-ray computed tomography and artificial binary particles

Takao Ueda *, Tatsuya Oki, Shigeki Koyanaka

Environmental Management Research Institute, Department of Energy and Environment, National Institute of Advanced Industrial Science and Technology (AIST), 16-1 Onogawa, Tsukuba, Ibaraki 305-8569, Japan

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ABSTRACT

Accurate evaluation of the mineral-liberation state is important for mineral processing. The liberation state is commonly measured by two-dimensional (2D) observations of the particle sections. However, this method inevitably suffers from stereological bias. Research on stereological bias and its correction method is an ongoing field of research. Stereological bias experiments require that the condition of the particles' internal structure can be systematically designed and the three-dimensional (3D) and 2D liberation states can be obtained. An experimental method combining artificial binary particle production and X-ray CT is proposed in this research, in response to the abovementioned requests. Liberation and stereological bias analyses were conducted on 16 samples with various internal structures using the proposed method. In addition, a recently proposed stereological correction method based on texture analysis of particle sections was attempted and its basic correction effect was validated.

1. Introduction

Accurate evaluation of the mineral liberation state is important for efficient mineral processing. The liberation state is commonly assessed by two parameters: *degree of liberation*, which is the fraction of the liberated particles of a mineral of interest over the total amount of the mineral, and *liberation distribution*, which is the cumulative distribution of particles with respect to their content of the mineral of interest. In general, polished sections of ore particles mounted in resin are observed to measure the liberation state. Recently, it has become possible to automatically identify and visualize the mineral phase allocation in the particle sections using scanning electron microscope (SEM) / energy dispersive X-ray analysis based automated analyzers (e.g., mineral liberation analyzer (MLA) [1], quantitative evaluation of minerals by SEM [2], and TESCAN integrated mineral analyzer [3]). This recently popularized technology enables us to measure a great number of particle sections within acceptable time, thus allowing us to obtain statistically credible data.

However, two-dimensional (2D) measurements of mineral liberation inevitably suffer from an error known as *stereological bias*. This bias is caused by the fact that sections of composite particle can be apparently liberated in 2D but those of truly (three-dimensionally (3D)) liberated particle is always liberated in 2D. This bias is unavoidable in principle as long as the liberation state is measured in 2D. Recent studies have clearly shown that the magnitude of this bias is associated with the particles' internal structure and is very large under some conditions [4,5]. The bias cannot be avoided by taking a great number of particle sectional measurements because its occurrence mechanism is independent of statistical error [6].

Since the stereological bias of the degree of liberation was pointed out by Gaudin in 1939 [7], it has been heavily studied. One approach is to obtain the 3D liberation state directly by X-ray computed tomography (X-ray CT) [8–12], which is of significance as basic research. Another approach is to estimate the 3D liberation state from 2D measurements and is known as *stereological correction*. Miller & Lin [13] and King & Schneider [14] converted the 2D liberation distribution into their 3D counterparts using a kernel function. Barbery [15] proposed a method using the particle structure models of the Poisson mosaic and the Boolean and particle shape models based on the line assessment of particle sections. Gay has developed various methods [16]. In his representative method, nine stereological equations that correlate 2D and 3D are applied, and 3D liberation was determined to minimize the errors in the equations. Ueda and his colleagues [4,17] have developed a method in which *texture* (i.e., 2D pattern) of mineral phases

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* Corresponding author.
E-mail address: t-ueda@aist.go.jp (T. Ueda).

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is analyzed; the best-fit 3D-particle model is selected from the database of 3D-particle model via encompassing computation; and the 3D liberation is estimated by the best fit 3D particle models.

Experimental validation of the stereological correction method has encountered difficulty. In many cases, the stereological bias of actual ore samples is measured as follows: (1) their 2D liberation is measured using automated analyzers such as MLA; (2) their 3D liberation is obtained via heavy medium separation; and (3) 2D and 3D liberations are compared to calculate the stereological bias. However, there are two problems with this approach. First, the experimental errors in 2D and 3D are quite different; hence, it is difficult to compare them. Second, systematic study on the influence of the particles' internal structure is difficult using a real ore; hence, global discovery of the stereological bias and versatility assessment of the stereological correction method are not achieved. Based on the abovementioned considerations, an experimental study using artificial particles (whose internal structures can be systematically studied), and X-ray CT, which permits 2D and 3D liberation observations with the same accuracy, is conducted in the present study.

Artificial particle samples have been used in liberation studies. Lin et al. produced *binary* particles composed of two materials (namely, epoxy resin and silica sand) for their liberation investigation [18]. Owada et al. used artificial binary particles of ordinary Portland cement and glass beads to establish a liberation model by taking material boundary breakage into account [19]. The artificial particles used by them are produced by a common procedure as follows. First, a *dispersed material*, such as silica sand or glass beads, is poured into a *matrix material* such as epoxy resin or cement in the liquid state and allowed to become rigid. Second, the binary material is crushed, comminuted, and screened to produce binary particle samples. Using this procedure, it is possible to control the volume fraction, size, and shape of dispersed materials, as well as the size of the particles, leading to a systematic study of the influence of the internal structure of particles upon the stereological bias. Therefore, this procedure is adopted in the present study.

X-ray CT has been used for mineral liberation analysis as well. Miller, Ling, and their colleagues have conducted pioneering research on this topic: coal washability analysis was conducted using X-ray CT in 1992 [20]; the 3D liberation states of dolomite and sphalerite particles were measured using cone beam X-ray CT in 1996 [8]; mineral exposure of copper ore was observed to investigate ultimate recovery in heap leaching operation in 2003 [11]; 3D watershed segmentation and finite mixture distribution modeling was proposed for the post-analysis of X-ray CT in 2007 [10]; and the grade and recovery curve of phosphate ore were measured using X-ray micro CT in 2009 [9]. Besides this group, Schena et al. proposed an X-ray CT system with micrometer-size resolution specifically for mineral liberation analysis [21], and Agorhom et al. observed the concentration of gold in a vertical water stream using X-ray CT [22]. A study on stereological correction using X-ray CT was conducted by Gay and Morrison in 2006 [12]. Gay and Morrison took images of ore containing silver, lead, and zinc minerals with siliceous gangue by X-ray CT, binarized the images with relatively heavy minerals such as galena, pyrite, sphalerite, and pyrrhotite, and the other; calculated 3D liberation from X-ray CT data and 2D liberation via MLA. Then, Gay's stereological correction was attempted upon the 2D data for comparison with 3D data. For a systematic study of stereological bias, it is reasonable to combine the X-ray CT analysis with the artificial materials.

With this background, the following investigation was conducted in this study. First, 16 types of artificial binary particles composed of a combination of epoxy resin, cement, silica sand, and glass beads were produced. Second, 2D and 3D liberation states of the binary particles were computed using X-ray CT and post-calculation analyses such as morphological operation. Third, the textures of particle sections were analyzed. Finally, the stereological correction method based on the texture analysis [4,17] was conducted to estimate 3D liberation, and the estimated values were compared with the truly measured 3D liberation.

2. Methodology

2.1. Binary materials preparation

Fig. 1 illustrates a binary particle composed of dispersed material and matrix material. When generalized expressions for the dispersed and matrix materials are needed, they will be called phase A and phase B, respectively, and the solid granules composing phases A and/or B will be called particles. The key point of this experiment is to use the different specific gravities (SGs) of dispersed and matrix materials to distinguish them by X-ray CT at the following step. Therefore, epoxy resin (SG = 1.1) and ordinary Portland cement (SG = 3.2) are used for the matrix material, whereas glass beads (SG = 2.5) and silica sand (SG = 2.2) are used for the dispersed phase.

2.1.1. Epoxy-resin based materials

The glass beads or silica sand used as the dispersed material are poured into the liquid state epoxy resin. Because the dispersed material has a larger SG than the epoxy resin, it settles before the resin hardens without special care. The well-dispersed condition of the dispersed material is achieved by an appropriate combination of agitation (which prevents settlement of the dispersed material) and heating (which accelerates the hardening of the epoxy resin).

The mixture of high-viscosity epoxy resin and dispersed material is poured into a truncated square pyramid shaped silicon form that has a 13 × 13 mm^2 bottom face, has a 65 × 65 mm^2 top face, and 35 mm height, and is completely hardened.

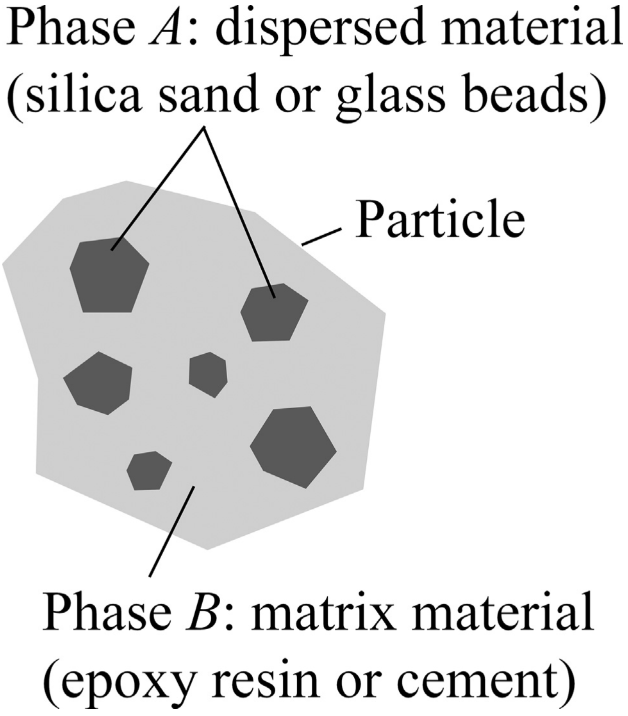

Fig. 1. Conceptual diagram of a binary particle.

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