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Modeling and measurement of abraded particles

Alexander Reinhold [a], Stefan Schorsch [b], Marco Mazzotti [b,*], Heiko Briesen [a,*]

[a] Chair for Process Systems Engineering, Technische Universität München, 85350 Freising, Germany
[b] Institute of Process Engineering, ETH Zurich, Sonneggstrasse 3, 8092 Zurich, Switzerland

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ABSTRACT

A novel particle representation is presented that allows the description of particle roundness as an additional measure, aiming at the characterization of entire particle ensembles during an attrition process. The particle representation is based on a combination of a convex polyhedron and a solid sphere which is known as Minkowski addition in the field of convex geometry. An image analysis procedure and a subsequent roundness estimation procedure are presented, utilizing a Hough transformation and a local optimal fit of the proposed shape model. The technique has been verified based on simulated particles and tested in a proof of concept experiment featuring potassium alum which was exposed to high stirring rates resulting in considerable attrition and abrasion. The results demonstrate that the proposed model and image analysis procedure are valuable tools to monitor and study attrition behavior.

1. Introduction

Crystallization is an important unit operation in the production of pharmaceuticals, bulk, and fine chemicals. In laboratory research, the focus is often put on phenomena such as nucleation, growth, and agglomeration whereas in a real production environment attrition is an important mechanism that has to be accounted for as well. If attrition occurs, small particles are generated at the expense of larger ones which thus undergo a change of shape due to abrasion. Those small particles can play a central role during crystallization as they typically are the primary source of secondary nuclei [25]. Both effects, i.e. the shape change of initial particles and the generation of a second population of small particles, contribute to a drastic impact on the final particle size distribution and hence influence further downstream processes [44] as well as product characteristics themselves [8].

It follows that attrition must be accounted for when developing models for the scale up of crystallizers [18,43]. [6] and Shoji and Takiyama [39] presented multidimensional population balance equations that included attrition phenomena characterized by an attrition kernel [10–12,26,32]. Theoretical models for predicting the attrition rate have been proposed by Briesen [5] and Ghadiri and Zhang [13]. In order to improve these existing models, in principle, any new laboratory experiment can focus on monitoring the small particles produced through attrition (daughter particles) [15] or the change of shape and size of mother particles during the occurrence of attrition because they determine the parameters that define the process, e.g. through the influence of the impact energy in particle collisions [9]. Additionally, mother particles often carry the majority of the product mass so that they are important contributors to the final product properties. If one is interested in particle shape, imaging techniques are an interesting and promising option as they allow tracking the change in size and shape of particles in suspension that is not accessible by other measurement techniques. Recently, a stereoscopic imaging setup has been proposed [20,36,37] and its application has been demonstrated, for example, in a multidimensional crystal growth rate parameter estimation [29]. In these works, both generic particle models (cuboids, needles, spheres) and convex polytopes, reflecting actual facets, have been used. For attrition, a new particle characteristic arises which is not captured by any of these models. Particles evolve into a rounder shape which is one of the key features of the attrition process. In order to account for this behavior in a process model, a new particle representation has been developed that is based on the Minkowski addition of spheres to convex polytopes and is presented in Section 2. While algorithms are available that can be used to measure roundness or angularity [22,23], a new procedure is developed that suits the introduced shape model. It measures the size and roundness from stereoscopic images and is presented in Section 3. To verify this procedure, a simulation case study and an experimental case study using potash alum crystals are reported in Section 4.

2. Preliminaries–shape model

2.1. Basic principles

The geometric shape of faceted crystals can be described based on the following representation, called a $\mathcal{H}$-representation, that

* Corresponding authors.

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comprises a matrix of facet normals $\mathbf{A}$ and a vector of facet distances $\mathbf{h}$ so that the crystal shape is provided by:

$$C(\mathbf{h}) = \{\mathbf{x} \mid \mathbf{A}\mathbf{x} \le \mathbf{h}\}. \quad (1)$$

Each inequality $\mathbf{a}_i^T\mathbf{x} \le \mathbf{h}$ constrains the allowed points $\mathbf{x}$ for the shape $C(\mathbf{h})$ to a certain half space from which the name $\mathcal{H}$-representation originates. Given a specific crystallization process, the matrix of facet normals is fixed so that the crystal shape only depends on the facet distances in the vector $\mathbf{h}$. This shape representation is well studied in literature [2,3,4,33,40,45].

To model the rounded shape of abraded crystals, we will take on our earlier proposal that involves an operation performed on sets of vectors which is called Minkowski addition [33]. The Minkowski addition of two sets S_1 and S_2 is defined as [35]:

$$S = S_1 + S_2 = \{\mathbf{x}_1 + \mathbf{x}_2 \mid \mathbf{x}_1 \in S_1,\ \mathbf{x}_2 \in S_2\}. \quad (2)$$

In the scope of this work, only a crystal shape C according to Eq. (1) and a solid sphere of a certain radius are added. It is therefore sufficient to see that the Minkowski addition allows morphing the particle shape continuously between a polytope with sharp edges and a sphere, as it is shown in Fig. 1. The shape defining equation is:

$$C(\mathbf{h}_\mathrm{k},\ \lambda_\mathrm{r}) = C(\mathbf{h}_\mathrm{k}) + \lambda_\mathrm{r} B. \quad (3)$$

where $C(\mathbf{h}_\mathrm{k})$ is a non-empty polytope, called the kernel polytope or kernel crystal, and $\lambda_\mathrm{r} B$ is a solid sphere (or ball) of radius $\lambda_\mathrm{r} \ge 0$. Crystals with sharp edges are obtained for $\lambda_\mathrm{r} = 0$ (top left in Fig. 1) and a sphere is obtained for $\mathbf{h}_\mathrm{k} = 0$ (bottom right in Fig. 1).

The convex bodies obtained by Eq. (3) are known as parallel bodies in mathematical literature [14,34,35,41]. Their volume μ^vol, surface area μ^sur and mean width μ^mw can be computed through the corresponding measures $\mu_\mathrm{k}^\mathrm{vol}$, $\mu_\mathrm{k}^\mathrm{sur}$ and $\mu_\mathrm{k}^\mathrm{mw}$ of the kernel polytope $C(\mathbf{h}_\mathrm{k})$ and the radius λ_r according to the Steiner formula [35]:

$$\mu^\mathrm{vol} = \mu_\mathrm{k}^\mathrm{vol} + \mu_\mathrm{k}^\mathrm{sur}\,\lambda_\mathrm{r} + 2\pi\,\mu_\mathrm{k}^\mathrm{mw}\,\lambda_\mathrm{r}^2 + \frac{4\pi}{3}\,\lambda_\mathrm{r}^3 \quad (4)$$

$$\mu^\mathrm{sur} = \mu_\mathrm{k}^\mathrm{sur} + 4\pi\,\mu^\mathrm{mw}\lambda_\mathrm{r} + 4\pi\,\lambda_\mathrm{r}^2 \quad (5)$$

$$\mu^\mathrm{mw} = \mu_\mathrm{k}^\mathrm{mw} + 2\,\lambda_\mathrm{r}. \quad (6)$$

While the mean width is a commonly used measure in mathematical literature, this is not (yet) the case within the domain of particle modeling. However, the width is equivalent to the Feret diameter in a sense that the mean width matches the mean Feret diameter when it is averaged uniformly over all measurement directions.

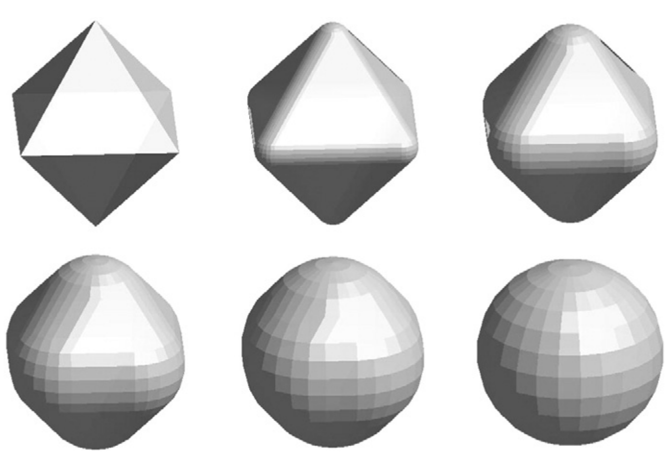

Fig. 1. Continuous transition of a crystal with sharp edges (top left) to a ball (bottom right).

2.2. Size and roundness

Eqs. (1) and (3) define how a rounded particle shape can be decomposed into a kernel polytope $C(\mathbf{h}_\mathrm{k})$ and a solid sphere of radius λ_r. These shape parameters are, however, difficult to observe directly so that we define measures for the particle size and roundness that can be identified easily from experimental imaging data by the procedure presented in Section 3.

For the quantification of the particle size, the mean width, Eq. (6), will be used. In this way, the dependence of the particle size on the shape defining variables $\mathbf{h}_\mathrm{k}$ and λ_r is piecewise linear, where the piecewise characteristic is caused by the behavior of the mean width functional $\mu_\mathrm{k}^\mathrm{mw}$ with respect to the vector $\mathbf{h}_\mathrm{k}$ [33,35]. Other measures, like the particle volume or surface area, are equally valid size measures but lead to more complicated equations. Additionally, the particle volume, that might be the most common alternative, is not measurable from 2-dimensional projections like those used in this work.

Abraded solids can be characterized by two distinct size independent measures, namely sphericity and roundness [42]. The sphericity expresses the compactness of the particle shape and is defined as the ratio between the surface area of a volume equivalent sphere and the real surface area. Since the general compactness does not vary significantly during abrasion, sphericity is not considered. The roundness on the other hand specifically addresses the local abrasion of the corners of the particle and is defined through the description of a measurement procedure [42]. Three perpendicular projections are taken, like the three images of Fig. 2. For each projection p, the radius R_p of a maximum inscribed circle (bold circle) and the curvature radii $r_{i,p}$ for all corners (thin circles) are measured. Corners are defined as regions of the boundary for which the curvature is less than or equal to R_p. The final roundness is then obtained by:

$$\text{roundness} = \frac{1}{N}\sum_{i,p}\frac{r_{i,p}}{R_p} \quad (7)$$

where N is the total number of measured curvature radii. Despite the definition of roundness by Wadell [42], other numerous definitions exist in the field of mineralogy or geology. This does also include definitions for the alternative measure: angularity. Most of these definitions do not separate well the overall shape information from the edge specific roundness or angularity [1,16,24,31]. According to this and given that the radius R_p of a maximum inscribed circle cannot be computed by our proposed framework for measure computation [33], we present a new roundness measure that reflects the inherent nature of the Minkowski addition based shape model. The difference of the total mean width and the mean width of the kernel polytope results in that part of the total size that is generated by the Minkowski addition of the solid sphere. Because of this relation, the new roundness descriptor μ^B is called additive roundness and will be used in the following:

$$\mu^B = 1 - \frac{\mu_\mathrm{k}^\mathrm{mw}}{\mu^\mathrm{mw}} = \frac{2\lambda_\mathrm{r}}{\mu_\mathrm{k}^\mathrm{mw} + 2\lambda_\mathrm{r}}. \quad (8)$$

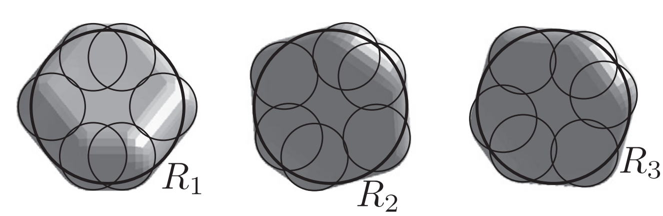

Fig. 2. Illustration of roundness by [42].