Advanced Powder Technology 23 (2012) 509–516

Contents lists available at SciVerse ScienceDirect

Advanced Powder Technology

journal homepage: www.elsevier.com/locate/apt

Original Research Paper

2-D sizing of sodium oxalate crystals by automated optical image analysis

A.V. Bekker, J. McShane, D. Vassallo, D. Bedell, T.S. Li, I. Livk *

CSIRO Process Science and Engineering, Parker CRC for Integrated Hydrometallurgy Solutions, PO Box 7229, Karawara, WA 6152, Australia

ARTICLE INFO

Article history:
Received 3 February 2012
Received in revised form 15 April 2012
Accepted 22 April 2012
Available online 23 May 2012

Keywords:
Sodium oxalate crystals
Crystal shape
Morphological filter
Aspect ratio
Rectangularity

ABSTRACT

A new automated static optical image analysis (SOIA) method was developed for characterising the shape of sodium oxalate crystals by separately measuring their length and width. The automated size analysis combines crystal image acquisitioning with a custom-developed numerical algorithm for morphological analysis of crystals. The automation of the two main measuring steps enables characterisation of a large number of crystals ensuring statistically meaningful results. As part of the numerical algorithm, a rectangularity-based morphological filter was implemented to eliminate overlapping crystals and crystal networks. The developed optical sizing technique has a low detection limit of 0.25 μm, which is close to the theoretical resolution of optical microscopy. It is demonstrated that the new automated SOIA sizing technique is very sensitive in identifying oxalate crystals of different shape. The measured crystal size distributions are compared to those obtained by an AccuSizer sizing instrument to confirm that they are consistent with the measurements obtained by a conventional crystal sizing instrument.

1. Introduction

In alumina refineries around the world, sodium oxalate emerges as a common impurity that needs to be appropriately managed to ensure efficient and stable operation of the Bayer process. Commonly, 5–10% of the organic carbon, which mostly enters Bayer process liquors in the form of humic substances [1], decomposes to sodium oxalate during the digestion stage [2,3]. When dissolved, sodium oxalate has no significant effect on precipitation yield or product quality [4]. However, through recycling of Bayer liquor the oxalate liquor concentration builds up resulting ultimately in precipitation of solid phase oxalate (SPO), which does affect the process in an undesirable manner [5]. Alcoa's nine refineries alone, for example, produce nearly 200 metric tonnes of SPO each day [6]. Sodium oxalate crystals in the Bayer precipitation circuit commonly exhibit an elongated needle-like shape as a result of different crystal growth rates of various oxalate crystal faces [7]. To enable an effective control of SPO in the Bayer process, it is essential to have available an accurate and efficient technique for characterisation of the size and shape of sodium oxalate crystals.

Measuring oxalate particle size by traditional methods, such as laser light scattering Malvern Multisizer or AccuSizer instruments, only provides information on the diameter of an equivalent sphere. Due to the irregular shape of SPO crystals, this information is insufficient, as it does not provide information about the crystal geometry. A 2-D sizing method, at least, is required in order to characterise crystal aspect ratio, and crystal length and width.

A 2-D technique for measuring crystal size was applied previously by Wang et al. [8] using an optical-microscopy in situ dynamic image analysis approach. This method, which is also described by ISO 13322-2:2006 standard [9], is mostly suitable for measurements in very dilute crystallisation systems with consistent and stable background. In addition, the in situ 2-D imaging suffers from problems associated with the random orientation of irregular crystals in a 3-D space [10]. This can significantly distort actual dimensions of needle-like suspended crystals. To avoid some of these issues, a 3-D in-line digital holographic microscopy was developed for crystal characterisation [11,12]. However, this method is restricted by a minimum size limit of 4–10 μm due to the holographic diffraction effects becoming prominent at that crystal size. Although potentially very powerful, this technique is currently not suitable for measuring a large number of crystals, as required in the case of SPO.

To overcome some of the restrictions mentioned above, a new automated static optical image analysis (SOIA) based sizing technique for measuring a 2-D size of individual oxalate crystals was developed. The novel crystal sizing technique, which is based on the analysis of optical microscopy images of oxalate crystals, has a low detection limit of 0.25 μm, which is close to the theoretical resolution of optical microscopy. The automated SOIA technique combines acquisitioning of optical images with a numerical algorithm for the morphological analysis of crystal images. The automation of the two main steps, image acquisitioning and image

* Corresponding author.
E-mail address: iztok.livk@csiro.au (I. Livk).

0921-8831/$ - see front matter
http://dx.doi.org/10.1016/j.apt.2012.04.013