Advanced Powder Technology 23 (2012) 191–198

Contents lists available at ScienceDirect

Advanced Powder Technology

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

Original Research Paper

Pentaerythritol crystallization – Influence of the process conditions on the granulometric properties of crystals

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ARTICLE INFO

Article history:
Received 24 September 2010
Received in revised form 21 January 2011
Accepted 2 February 2011
Available online 12 February 2011

Keywords:
Crystallization
Granulometric properties
Pentaerythritol
Seeding

ABSTRACT

The quality of crystals depends on many factors that determine their granulometric properties. In order to obtain crystals of desired size distribution, proper selection of the operating conditions is of a great importance. Commonly, the unseeded cooling crystallization is controlled by selecting the appropriate cooling profile. The crystallization process can also be controlled by adding a certain number of seed crystals of a uniform size in the crystallizer at the saturation temperature. This paper investigates the influence of the process conditions (mixing intensity, cooling profile, batch time, saturation temperature and seeding) on the granulometric properties of pentaerythritol obtained by batch cooling crystallization. All investigated process conditions influence the crystal size distribution (seeded and unseeded experiments). On the other hand, the shape of crystals was the same for all experimental conditions. Optimal cooling profile, lower retention time, higher mixing rate, and smaller initial seed surface area improves the final crystal size distribution.

1. Introduction

Crystallization is one of the most complex separation processes due to numerous simultaneous processes (momentum, heat and mass transfer, phase change, dissolution, agglomeration, breakage . . .). The obtained crystals have specific purity and granulometric characteristics (shape, size, and crystal size distribution) [1]. The crystal quality is very sensitive to cooling rate, mixing intensity, system geometry, type of crystallization process, and impeller used for mixing. Crystals of the desirable granulometric properties can be obtained only if the thermal and the hydrodynamic conditions are selected very carefully. In order to minimize excessive primary nucleation, crystallization has to be conducted within the metastable zone. Excessive nucleation yields too many small crystals and an unacceptable crystal size distribution. For the same reason, secondary nucleation (contact nucleation, breakage, mixing) has to be suppressed as much as possible [2]. At the optimal process conditions, regular shape crystals with the regular narrow size distribution will be produced. Hydrodynamic conditions have to provide good flow of the solution past the heat transfer surfaces and satisfactory handling of the crystals being formed [3]. In other words, to support crystal growth, it is very important that crystal surface area be accessible at locations where the supersaturation is generated.

Significant improvement in CSD and operation can be achieved by controlling supersaturation levels during the batch crystallization process. Cooling profile can be designed in a way that the optimum rate of supersaturation generation could be achieved at all stages of the batch run. Some of the results demonstrated that supersaturation control during a batch run was beneficial for increasing crystal size, reducing the batch cycle time and markedly narrowed the CSD [4].

The crystallization process (nucleation and growth) can be controlled by adding a certain number of seed crystals of a uniform size, L_s, in the crystallizer at the saturation temperature [4]. The process has to be conducted at low supersaturation level, within the metastable zone. Seeded crystallization process is influenced by the way in which the seeds are produced, seeding technique, seed quantity, and size. The size and mass of seeds influence the crystal size distribution and the rate of crystallization. Seeds must be very small and often are produced by milling. If the supersaturation is maintained below the metastable zone upper limit, then the spontaneous nucleation will be avoided and only the seed crystals will grow. Consequently, produced crystal size distribution will be narrow. Some materials tend to form agglomerates. In that case crystals with less uniform size distributions will be obtained.

Pentaerythritol is an organic chemical widely used in the production of high – quality alkyd resuis, lacquers, lubricant additives. Lots of researchers have studied the influence of different process conditions on the crystallization (seeded or unseeded) kinetics (nucleation and crystal growth) of pentaerythritol (batch or contin-

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doi:10.1016/j.apt.2011.02.001