Polyethylene vs. Stainless Steel Impellers for Crystallization Processes

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The importance of controlling the crystal size distribution of the product from an industrial crystallizer has long been recognized. To do this, some control over the nucleation and growth rates of crystals must be maintained. The crystal growth rate can be influenced by varying degrees of agitation, supersaturation, and temperature. However, these variables also have a major effect on the nucleation rate, so that it is seldom possible to change one of these quantities without simultaneously changing the other. It is the purpose of this note to discuss one means of substantially influencing the nucleation rate without affecting the growth rate.

The role of crystal impacts in determining the nucleation rates was established by Clontz and McCabe (1971) and Johnson et al. (1972). Ottens and deJong (1972) have presented data to indicate that the major source of nucleation in an industrial crystallizer is at the impeller, if there is internal mixing, or at the pump, if the mixing in the crystallizer is accomplished by external recycle. Johnson et al. (1972) also show that when a single crystal is impacted by a polyethylene rod, whose face is parallel to the crystal face being impacted, no new crystals are generated. This can be contrasted to the generous number of crystals produced by impact with a metal rod. Their system was magnesium sulfate heptahydrate.

On the basis of these reports an experimental plan to investigate the influence of the material of construction of the impeller in a stirred tank crystallizer was formulated. The crystallizer was a cylindrical Pyrex battery jar 15.25 cm in diameter and filled to a depth of 17.15 cm. Three equally spaced plexiglass baffles extending to within 3 cm of the bottom were attached to the crystallizer. A fitted plexiglass cover was constructed to accommodate the impeller shaft, a thermometer which could be read to 0.1°C, and seed crystal insertion. The entire apparatus was placed in a constant temperature bath. The impellers used were 5.1 cm in diameter, three bladed, with a square pitch, and located 9 cm from the bottom of the crystallizer.

In all of the experiments of this series, the impeller speed was held at 700 rev./min., a value which kept the seed crystal adequately suspended. The size of the seed crystals was held essentially constant at about 5 mm × 2 mm × 2 mm; when deviations from this size occurred the results were erratic. Supersaturation was kept at 2°C and the temperature at about 33 to 35°C. A typical run went as follows: A solution of $MgSO_4 \cdot 7H_2O$ was prepared, filtered, and placed in the crystallizer. The saturation temperature of the solution was determined by refractive index and the temperature of the solution was adjusted to 2°C above saturation. A seed crystal of the appropriate size and shape, and which had been recrystallized below 48.2°C to ensure the pure heptahydrate form, was inserted into the crystallizer and cured for 16 minutes with agitation. Shorter curing times gave excessively high crystal counts. Agitation was discontinued and the solution slowly cooled to the prescribed supersaturation of 2°C. When the system had stabilized at the temperature, the agitation was switched on for exactly 2 minutes; the solution was then allowed to sit for 90 minutes of growing time before the new crystals on the bottom of the tank were counted.

In blank runs following the above procedure, but with no seed crystal or with a seed crystal fixed in the tank, no new crystals were produced. The average crystal count for a polyethylene impeller was about 250 while for a stainless steel impeller it was about 1120, greater than a fourfold increase. The raw data are presented in Table 1.

The industrial significance of these data seems clear; if the nucleation rate in the crystallizer is too high, so as to yield a size distribution that is too small, converting from a metal impeller to one manufactured from a softer polymeric material may be advisable.

The probable reason for the reduced nucleation by the plastic impeller is related to the mechanism of contact nucleation. Energy is necessary to form nuclei by this phenomena. It is suggested that the plastic yields on contact with the crystal and some of the energy is absorbed by the impeller and a smaller amount is left for action on the growing crystal (Johnson et al., 1972).

Table 1. Data for Batch Crystallizer Runs

Run no.	Impeller	Crystal count
1	PE (polyethylene)	339
2	PE	302
3	PE	234
4	PE	164
5	PE	225
6	SS (stainless steel)	1,002
7	SS	947
8	SS	1,258
9	SS	1,265

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