H = average of h_0
g = gravitation constant
p = pressure
T = surface tension
R = tube radius
C_1, C_2 = dummy constants

Greek Letters

α = tube inclination to vertical
ν = kinematic viscosity
η = similarity variable = $\cot \theta/2 \exp(z \tan \alpha/R)$
ρ = density

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Effect of a Soft Impeller Coating on the Net Formation of Secondary Nuclei

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Recent work has confirmed the importance of secondary nucleation as the principal seed source in continuous crystallizers of the mixed-magma type (Clontz and McCabe, 1971; Johnson et al., 1972; Larson and Bauer, 1972; Randolph and Cise, 1972; Youngquist and Randolph, 1972; Cayey and Estrin, 1967; Ottens and DeJong, 1973; Bennet et al. 1973). These studies indicate that a dominant mechanism causing secondary nucleation is the high speed collision of macro-sized parent crystals with the vessel impeller or circulating pump impeller. Further, the number of crystals formed per impact appears to increase with parent crystal size and weight (proportional to impact stress). In a recent publication, Shah, McCabe, and Rousseau (1973) show that total nuclei formation is reduced in a mixed vessel when the agitator is constructed of a relatively soft material. In their experiment a single $MgSO_4 \cdot 7H_2O$ crystal was allowed to tumble in the vessel and the nuclei formed by collisions with the impeller were counted after a given time in the batch-operated vessel.

In the present experiments the effect of agitator coating on the formation of secondary nuclei in a continuous flow mixed-magma suspension was measured. The system studied was water/K_2SO_4. Identical runs were made and compared using a four-bladed stainless steel impeller before and after covering with 0.76 mm (0.03 in.) of a soft rubber coating of Uralane 8267. Similar runs were made with an aluminum impeller before and after coating with 0.10 mm (0.004 in.) of Teflon.

APPARATUS

The system used for this study consisted of a one-liter glass vessel with draft tube agitation provided by a variable speed axial flow impeller. Solution at a controlled saturation is continuously fed to the vessel at a known rate. Total supersaturation is controlled by controlling vessel temperatures. Under the short retention conditions of the test, supersaturation relief is negligible and suspension supersaturation is mainly determined by the ΔT drop from feed to crystallizer. Dry K_2SO_4 seed crystals of a known weight and size are introduced at the initial run time through an access port; these parent crystals are totally retained by a fine mesh (about 37μ) retaining screen through which the effluent discharges by gravity flow. A fine distribution of secondary nuclei is measured in the effluent using a multichannel Coulter particle counter. The operation of the apparatus is described elsewhere in detail (Cise and Randolph, 1972; Randolph and Cise, 1972).

RESULTS

The population data from the Coulter counter were processed as follows. Background-corrected population density data at a given crystal size were time smoothed with a second-order polynomial fit using a linear regression program. These smoothed data were then plotted on a semi-log plot versus particle size at specific run times. These data were again smoothed in the size domain with another polynomial fit. Figure 1 shows the smoothed data at identical run times of 40 min. for both the stainless and rubber-coated impellers in otherwise similar runs. The data points shown represent the original population measurements smoothed in time and particle size. The uncoated impeller consistently results in higher population densities in the crystallizer effluent under similar conditions of operation.

Figure 2 plots the ratio of population density for the stainless steel impeller to the rubber coated impeller as a function of crystal size for the same two runs shown in Figure 1. Note that the ratio of population densities increases uniformly with size and elapsed run time.

Figure 3 plots data similar to Figure 2 for two other comparison runs with the stainless and rubber coated impeller. These runs had a higher impeller rev./min. and lower supersaturation than the runs shown in Figure 2. Note that the ratio of population densities, stainless to rubber coated, is lower than in the first runs shown: however, the absolute value of population densities was higher. In the runs shown in the lower half of Figure 3 a step decrease was made in impeller rev./min.; an immediate increase in the ratio of population densities was observed.

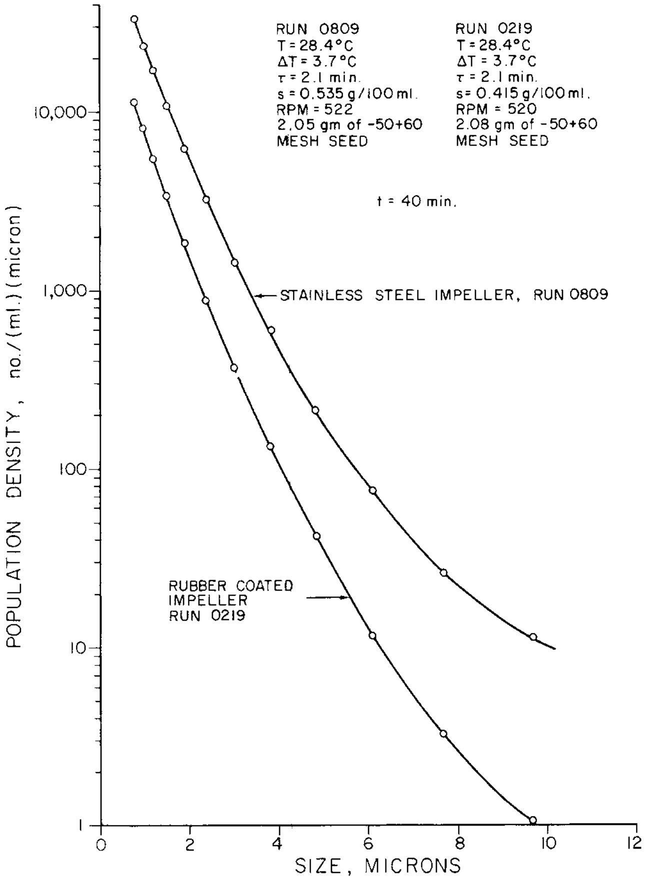

Fig. 1. Population density as a function of size for two impeller types.

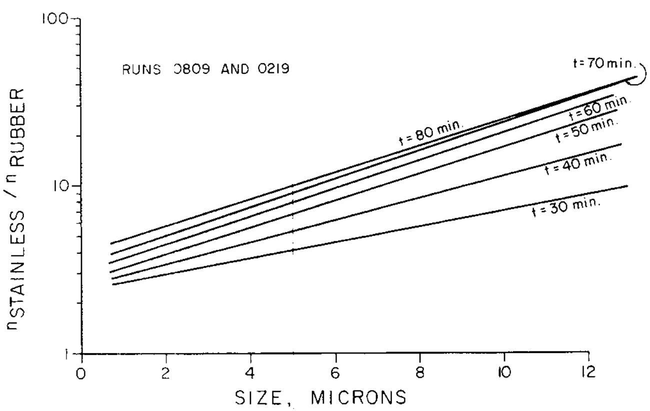

Fig. 2. Ratio of population density for stainless steel to rubber coated impellers vs. crystal size.

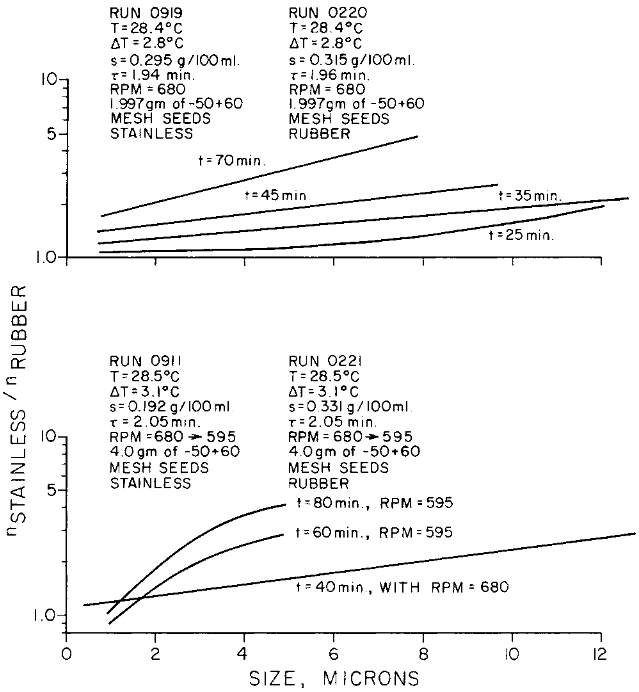

Fig. 3. Ratio of population densities for stainless steel to rubber coated impellers for various particle sizes.

Figure 4 plots time-smoothed population data similar to that obtained in the first experiment except the two impellers are aluminum and Teflon coated aluminum. The stirrer speed was increased from 780 to 822 rev./min. during the run. The uncoated aluminum impeller consistently resulted in 4 to 10 times higher population densities in otherwise similar runs. The step increase in stirrer rev./min. resulted in an abrupt increase in crystal population as previously observed in this system (Randolph and Cise, 1972).

The measured value of supersaturation is shown on the figures for each run. Although the ΔT between saturator and crystallizer was identical in each set of comparison runs, the measured value of supersaturation varied, either due to inaccuracy of gravimetric measurement of the very small concentration differences or to differing approaches to saturation in the packed bed. Recent measurements using a refractometer indicate that ΔT measurements are probably a more accurate indication of the supersaturation driving force than gravimetrically determined concentration differences. In any case no consistent bias in actual measured supersaturation between comparison runs is noted.

DISCUSSION

Figure 1 shows convincingly that the number of secondary nuclei in a mixed-magma suspension is reduced when a soft coating is applied to the impeller surface. As seen in Figure 2 the ratio of population density for the stainless compared to the rubber coated impeller uniformly increases with size. Thus, either secondary nucleation (at the sizes measured) increased with size at a greater rate with the stainless steel impeller or more of the existing nuclei survived. As the supersaturation driving force (and thus presumably growth rate) was held constant in these

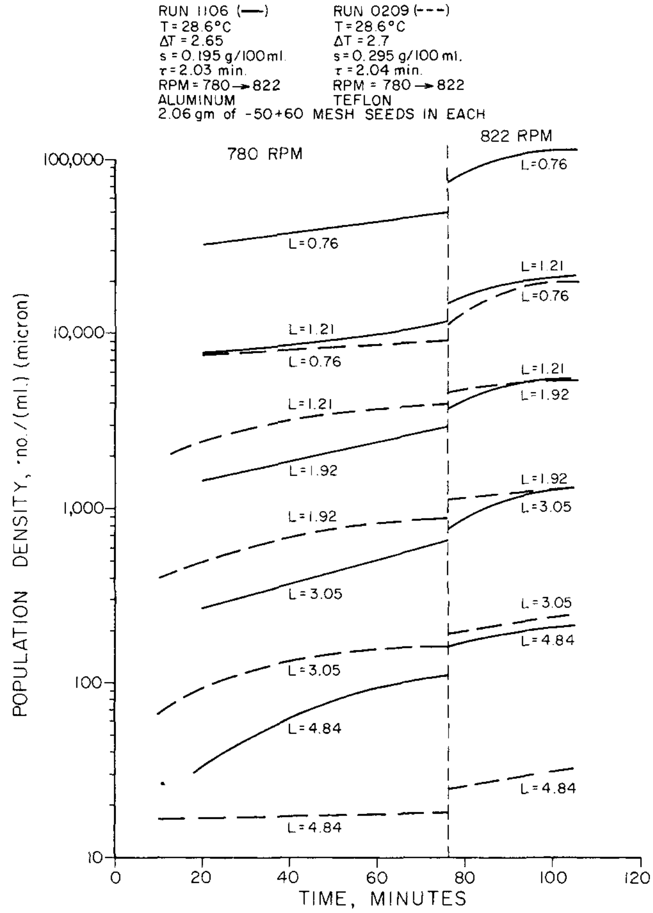

Fig. 4. Population densities for bare and Teflon coated impellers.

runs, the increased ratio of population density with size indicates an increased ratio of particle flux with size. Garabedian and Strickland-Constable (1972) postulate that the apparently slow growth rates of small crystals observed in this system (for example, Randolph and Cise, 1972) could result from the lack of crystal imperfections in the secondary nuclei. As the crystals grow, imperfections increase, resulting in the observed increase in growth rate. An alternate explanation of the apparent increase in population density ratio with particle size is that the rubber-coated impeller produced secondary nuclei with a higher degree of perfection and hence lower growth rate. This would result in a higher washout rate and decreasing particle flux with increasing size. However, the sudden increase in population density shown in Figure 4 as the impeller rev./min. increased [consistent with the work reported by Randolph and Cise (1972) and Youngquist and Randolph (1972)] indicates that the former explanation is correct; secondary nuclei are created over the entire size range of measurement and this rate increases more with size in the case of the harder impeller surface.

In either case the change in mechanism must be operative over the size range measured. The consistent increase in ratio of population densities with time is probably due to the increased breeding efficiency of the harder impeller surface as the parent crystals increased in size. Note that by 60-min. run time and at 12 microns size the population density was 35 times greater for the uncoated impeller than for the rubber coated impeller (Figure 2). A comparison of the decreased ratio of population densities in Figure 3 vis-a-vis Figure 2 indicates that there is a greater difference between the coated and uncoated impellers at low rev./min. and high supersaturation. It is likely that the difference between impellers saturates with intense agitation as crystal/wall and crystal/crystal impact mechanisms become operative in forming secondary nuclei.

The results from the bare and Teflon coated aluminum impellers are qualitatively the same as for the bare and rubber coated steel impellers. Again, a four-to-eight fold decrease in nuclei density under similar operating conditions was observed with the Teflon coated aluminum impeller.

SUMMARY AND CONCLUSIONS

1. A soft coating (for example, Uralane or Teflon) on the suspension impeller dramatically reduces secondary nucleation of K_2SO_4 crystals.

2. The ratio of population densities for the bare and coated impellers increases uniformly with size over the one-to-ten micron size range of measurement.

3. The increase in population density ratio with size can be explained by continued secondary nucleation over this size range (Randolph and Cise, 1972) or a difference in growth rate of the secondary nuclei caused by a difference in nuclei perfection (Strickland-Constable, 1972). If the former mechanism is correct, this would imply that the mean size of secondary nuclei increases with the hardness of the impellor, that is, the impact stress.

4. The difference between bare and coated impellers is increased at low rev./min. and high supersaturation.

5. The ratio of population densities for the bare and coated impellers increases with time as the seed crystals grow in size; such effects should be more noticeable in a crystal slurry of large average size.

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