Modeling of Coarse Particle Shape Evolution During Attrition in a Stirred Vessel

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Abstract

Particle breakage in stirred vessels is usually modeled using population balance equations (PBEs) to describe the evolution in particle size with time. Although shape has a strong effect on the particle properties and hence the product quality, changes in the particle shape distribution are typically neglected. To account for changes in both size and shape simultaneously, a multivariate population balance must be used. This multivariate PBE requires a multivariate breakage distribution function that accounts for the observed size and shape evolution in actual systems. A general mathematical framework that guarantees mass conservation and exchangeability for these breakage distribution functions has already been developed [1]. While the breakage distribution functions in this framework meet these constraints, they are not based on the fundamental physics of particle attrition. What is needed are breakage distribution functions based on fracture mechanics.

Previous research [2-3] uses fracture mechanics to predict the total particle volume lost due to attrition. This information is incorporated into the new model by including it in the breakage distribution function. Starting with the original coarse particle size and shape, the model accounts for the material removed from the parent particles due to attrition. The approach is to model the breakage distribution function as a bivariate function of the particle volume and the shape factor. A comparison of the model with experimental results for several systems is presented.

This work has wide applicability in that it directly affects any unit operation involving solids breakage in stirred vessels. This includes both stirred reactors with solid particles as well as crystallizers. Not limited to batch processes, this can also be used for continuous processes.

Previous Work

Gahn and Mersmann [2, 3] developed a physical attrition model based on a set of assumptions and on the material properties of hardness, fracture resistance, and the quasi-isotropic elastic constant. This work was based on the impact of a crystal with a hard surface such as an impeller. With this method, the number of fragments generated could be estimated as well as the minimum and maximum particle sizes. In addition, the volume of material abraded from a single particle, V_a, was predicted from the Vickers' hardness (H_v), the shear modulus (□), the fracture resistance (□), the efficiency factor (K), and the plastic deformation work for a given particle (W_{pl}). In later work [4] this attrition model was applied to a crystallizer where both growth and breakage were occurring simultaneously. While these models quantitatively address the size distribution, they do not address particle shape.

If the original volume of the parent particle is known, then the change in actual parent particle volume can be calculated by subtracting the volume lost due to attrition. This volume loss due to attrition changes the shape factor of the parent particle. Since the theory considers the contact of a crystal corner with a hard surface such as an impeller and since it considers materials that are more prone to attrition than to fragmentation, the experimental tests will focus on crystals with corners that are prone to attrition.

The purpose of the experimental work is to provide validation for the modeling work. Even if theoretical models can be tested by using analytical solutions, it is imperative to prove that the models can represent real systems. Since previous papers give very little information on shape factors due to attrition and breakage, it is necessary to perform experiments to collect this data.

Experimental Method

Crystallization experiments were performed in a LabMax automated laboratory reactor system manufactured by Mettler-Toledo. This system includes a 1 liter jacketed glass vessel with an upflow impeller stirrer, a temperature control system, and a data acquisition system. A Julabo chiller provided cooling fluid for the LabMax. The computer included with this system controled the temperature of the vessel contents by controlling the temperature of heat transfer fluid circulated through the vessel jacket. The temperature of the vessel contents was measured by a PT-100 temperature probe and recorded.

Since many size analysis methods such as sieving only give a single particle dimension, image analysis was used to measure the aspect ratio of individual crystals. Image analysis was performed using a polarized light microscope with a data acquisition system. This system included a camera mounted on the microscope that connected to a computer with image analysis software.

Since the objective was to study the effects of breakage in a stirred vessel, steps were taken to avoid primary nucleation and growth as well as dissolution. For these experiments, a saturated solution was used to minimize growth and nucleation. The saturated solution was created by heating a mixture above its saturation temperature to make sure that all of the solids had dissolved, and then lowering the solution temperature to the saturation temperature. During the tests, the temperature was held constant at the saturation temperature. The sieve fraction of crystals to be studied was added to the saturated solution. To prevent changes in the crystal size, the samples were rapidly filtered using vacuum filtration.

Experiments were conducted over a range of conditions with the sodium chloride/water system. Tests were run at several temperatures, impeller speeds, and magma densities as shown in Table 1. Test data was also taken at different residence times, τ. For comparison purposes, image analysis was run on unbroken crystals as well.

Table 1. Conditions for sodium chloride experiments.

T, °C	Agitation rate, rpm	M_T, g crystals/100 g sol'n	Time Elapsed, min
20	200	10	10
20	200	10	20
20	150	10	10
20	150	10	20
20	200	5	10
20	200	5	20
20	150	5	10
20	150	5	20

Results

Changes in the particle size distribution for NaCl can be seen after short residence times of 10 to 20 minutes. An example of this is shown in Figure 1. In this figure the number fraction in a size interval is plotted at the maximum particle size in the interval. The curves are used to guide the eyes. Examination of the particles under the microscope shows that the unbroken particles are very close to being an ideal cube. Due to the regular shape of the unbroken crystals, it is easier to detect changes in particle shape.

Concluding Remarks

Work is continuing with different systems such as potassium chloride/water. The theories will be tested with several systems under different conditions to determine the range of applicability of the theories.

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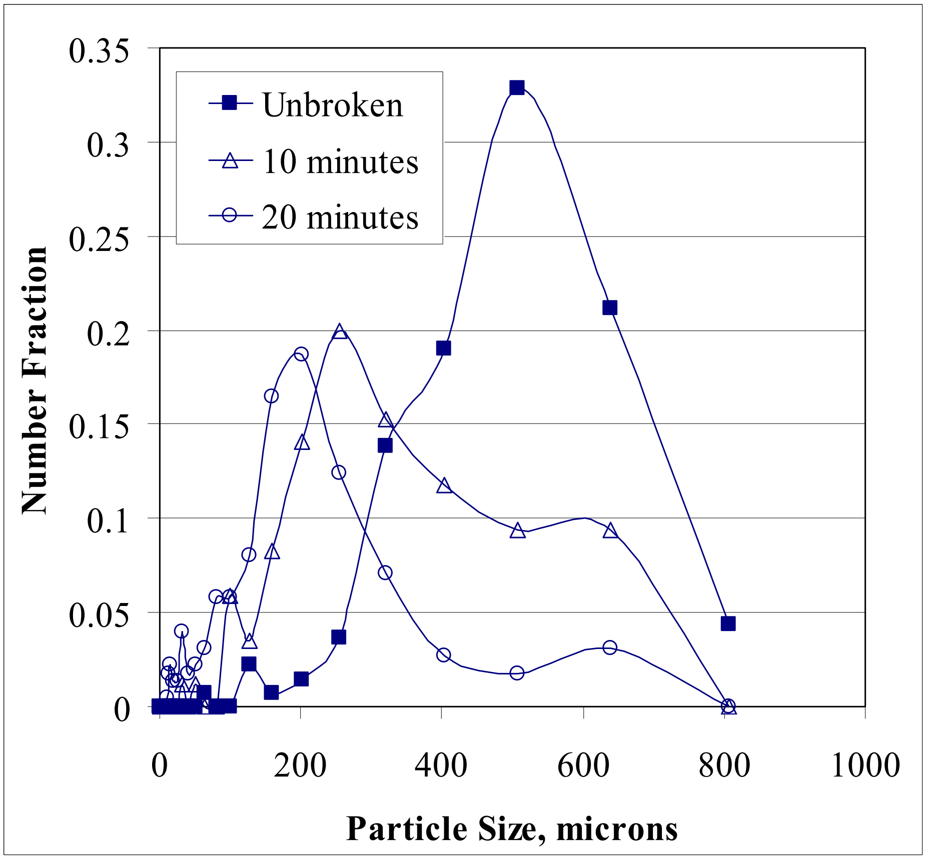

Figure 1. Particle size distributions for sodiumchloride at 20 ^{o}C, 150 rpm, and a magma density of 5 g crystals/100 g solution. Number fractions are plotted at the maximum length in the size interval.

References

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