

A Course in Particle and Crystallization Technology

Priscilla J. Hill¹

Abstract – The traditional chemical engineering curriculum is based on vapor-liquid processes with little discussion of processes involving solids. In typical courses attributes such as temperature, pressure, and composition are emphasized, while attributes such as particle size are omitted. For solids, however, product quality often depends on the particle size. Industry has stated that they need more engineers with training in particle technology [Ennis, 6; Nelson, 9; Nelson, 10]. In spite of this, few U.S. chemical engineering departments offer courses in particle technology.

In response to this, a new split-level elective course in Particle and Crystallization Technology (PACT) was developed. This survey course, first offered during the Spring 2007 semester, was a blend of theory and practical applications where the applications emphasized both unit operations and the interaction of operations in solids processing. This course differed from many particle technology courses due to its emphasis on crystallization as a particle formation process.

Keywords: *particle technology, crystallization*

INTRODUCTION

Particle technology has been important to mankind for thousands of years. The uses range from the production of early Egyptian pottery to milling of flour for bread [Ennis,6]. Not limited historic times, it is well known that particle technology plays a key role in the chemical processing industry. Recognizing this, Japan and Germany have powder technology departments in a number of universities. In spite of the importance of particle technology, as of 1994, the U.S. had largely ignored particle technology education [Ennis,6; Nelson, 9]. Some have observed that the U. S. has lost competitiveness in this area due the fact that it isn't included in engineering training [Nelson, 9].

After these observations were published in 1994 and 1995, some U.S. chemical engineering departments have added elective courses in particle technology. Faculty at the University of Akron [Chase, 2], New Jersey Institute of Technology [Dave, 3] and the University of Florida [Donnelly, 5] reported on their courses in 1998. Industrial researchers discussed the development in particle technology (PT) education in 1998 [Nelson, 10] and acknowledged the development of academic research centers at the University of Florida, Clarkson University, and Pennsylvania State University. More progress has been made with other courses.

There have been a number of developments since the 1998 review. Other U. S. universities such as Purdue University have added PT elective courses [Sinclair, 15]. While some faculty had developed entire courses, others developed other educational resources such as instructional modules [Donnelly, 5]. One item is the development of a PT textbook with a CD containing video sequences of laboratory demonstrations of interesting particle technology phenomena [Rhodes, 14]. Some educational resources included teaching examples and focused on fluid-particle systems [Fan, 7; Fan, 8]. There continues to be interest in particle technology. As noted by German academics, it is crucial to provide students with the fundamentals based on physical properties [Peukert, 11].

One limitation of these approaches is that they do not include crystallization, although crystallization is a key particle formation process. Crystallization is often neglected or only taught at a superficial level. It was recently

¹ Mississippi State University, Box 9595, Mississippi State, MS 39762, phill@che.msstate.edu

noted that crystallization could and should be incorporated in existing courses [Doherty, 4]. Specifically, it is important to teach the fundamentals of crystallization.

PARTICLE AND CRYSTALLIZATION TECHNOLOGY COURSE

To prepare students to face challenges in particle technology, it is essential to provide instruction in fundamental concepts. This was accomplished in a new split-level elective course in particle and crystallization technology (PACT). Specific educational objectives for this course are 1) to introduce students to fundamental concepts in crystallization and particle technology, and 2) to train students to apply these concepts to real world problems.

This new elective course in particle and crystallization technology was offered for the first time at Mississippi State University (MSU) during the Spring 2007 semester. This split level course enrolled 15 undergraduate students, juniors and seniors, and 2 graduate students for a total of 17 students. Split level classes are common for elective classes in the chemical engineering department in MSU. The graduate level was justified by having the graduate students perform work beyond that required for the undergraduate students. This extra work can include extra homework problems that are more advanced or more advanced test questions. The prerequisites were to have completed the first semester junior level classes in chemical engineering or equivalent. No text book was required for the course, although the recommended reference book was *Introduction to Particle Technology* by Martin Rhodes [Rhodes, 13]. Since a textbook was not available, many handouts were given to the students.

Since there was not a laboratory section for this course, it was necessary to find other means to visually demonstrate PT concepts. A DVD entitled *Product Properties and Process Engineering* available from BASF was used. Although originally produced in German, the DVD also has a version dubbed in English. One of the demonstrations involved the hiding power of two pigments suspended in a base, where the only difference between the two pigments was the particle size.

Since there are many aspects to particle technology, one semester is insufficient to cover the area. Therefore, this was a survey course that covered selected topics. These topics are summarized in Table 1. Topics 1 and 2 are background for both the particle technology and crystallization sections of the course. The particle technology focus includes Topics 3 through 7, and crystallization is covered as the last major topic. In many cases, the theoretical concepts were introduced and then followed by applications to actual equipment.

Table 1. Course Topics

<ol style="list-style-type: none">1. Introduction2. Particle characterization<ol style="list-style-type: none">a. Size and shape measurement techniquesb. Data representation3. Cyclones and hydrocyclones4. Settling<ol style="list-style-type: none">a. Single and multiple particles in a fluidb. Batch settlingc. Continuous settlingd. Real thickeners5. Filtration<ol style="list-style-type: none">a. Theory and calculations – constant rate and constant pressureb. Equipment, and equipment selection6. Particle size reduction7. Particle size enlargement8. Crystallization<ol style="list-style-type: none">a. Fundamentalsb. Extractivec. Fractionald. Polymorphs

Introduction

The purpose of the introductory lecture was to define particle technology, to explain the significance of particle technology and crystallization to the chemical industry, to motivate the students, and to provide course expectations. The significance was emphasized by including safety concerns such as the possibility of dust explosions due to particle size; and facts about the prevalence of solids in the chemical industry, e.g. 62% of Dupont's 3000 products are powders, crystalline solids, granules, slurries, dispersions and pastes [Ennis, 6].

Particle Characterization

Particle characterization and data representation were discussed because they are fundamental concepts for both PT and crystallization. Since many of the subsequent course topics use these fundamental concepts, these were discussed first.

Characterization included methods of size analysis ranging from earlier techniques such as sieving, imaging techniques such as microscopy, and more recent techniques such as light scattering and laser diffraction. Advantages and disadvantages of the various techniques were discussed. Much of the information was adapted from *Particle Size Measurement* [Allen, 1].

Data representation was also discussed. This included continuous and discrete size distributions, and population or number density functions. Common used continuous distributions such as Rosin-Rammler and log-normal distributions were emphasized. Other attributes such as particle shape were discussed and various particle shape factors were presented.

Cyclones and Hydrocyclones

Cyclones and hydrocyclones are good examples of separators that separate streams based on particle size. They demonstrate that using an average particle size is not sufficient for modeling some process units. Since these units often do not have sharp splits, the lectures include a discussion of the grade efficiency curve. Lectures also include scale-up considerations, types (e.g. high efficiency cyclones), and design configurations.

Settling

This topic was introduced with basic transport phenomena concepts including Stokes' Law and terminal velocity correlations. Various correlations for the drag coefficient are introduced. To extend the single particle theory to multiple particles in a solution, the concept of hindered settling was introduced. These fundamental concepts provided the basis for the design of batch and continuous settlers. Methodology was presented for designing settlers and thickeners.

Filtration

Filtration was discussed since it is a key operation in many processes involving solids. Theory included discussions of pressure drop and cake resistance. Theory was applied to constant rate and constant pressure filtration.

Regarding practical application, many types of filters were discussed along with their advantages and disadvantages. Other capabilities of filters were discussed including whether or not cake washing was possible. Comparisons were made between batch and continuous filters. Since there are so many types of filters, equipment selection heuristics were discussed primarily based on the charts developed by Purchas [Purchas, 12].

Particle Size Reduction

Theory included energy requirement correlations such as Rittinger's, Kick's, and Bond's Laws. Population balance modeling with breakage distribution functions and specific rates of breakage was also discussed. Various types of equipment were described and categorization of equipment according to particle size was included. Process synthesis was incorporated by discussing a milling circuit.

Particle Size Enlargement

Enlargement mainly focused on granulation and the physics of granule growth. Population balance models with agglomeration kernels were introduced. Various granulator types were discussed along with their benefits and limitations. Since individual equipment units are not usually used in isolation, a size enlargement circuit was presented.

Crystallization

Since many students have not been introduced to crystallization, it was necessary to cover the fundamentals. One key set of fundamentals is based on thermodynamics, specifically solid-liquid equilibrium. This is key since it determines whether or not crystallization can occur. Solubility concepts included theoretical models and empirical correlations. It also included data representation of various phase diagrams including isothermal cuts projected onto ternary diagrams. Various expressions for supersaturation are also introduced. Determining supersaturation is essential since it is the driving force for crystallization.

Process synthesis of crystallization systems was included for both fractional and extractive crystallization. These are based on equilibrium thermodynamics and do not provide sizing of equipment, but do provide a feasible equipment configuration for separating multiple solutes in a solution.

To size the equipment, it was necessary to introduce the crystallization kinetics. Therefore, simplified models of crystal growth and nucleation were introduced. It is emphasized that crystallization is not completely governed by equilibrium thermodynamics. Therefore, the metastable zone can be significant.

While thermodynamics is one focus, the other is structural. Polymorphs and enantiomorphs are defined and examples are given. The possible variations in properties of different polymorphs were presented.

DISCUSSION

From the discussion of topics, it is clear that this course was built on core studies in chemical engineering including thermodynamics, transport phenomena, and kinetics. Although the kinetics are crystallization kinetics rather than reaction kinetics, many of the concepts are the same. While it was impossible to cover everything about particle and crystallization technology in one semester, it was possible to cover theory and applications for selected topics.

The students' response to the course was quite positive. Some senior students who were graduating and going into industry at the end of the semester expressed that they took the class because they knew that they would have to work with particulate processes in their new jobs. Overall, the students' evaluations of the course were quite high.

Since this course has only been taught once, it is still a work in progress. One limitation of teaching population balance models is the mathematical difficulty of the solution technique for these partial integro-differential equations. The plan is to develop software with an MS Excel interface that students can use to study these models, so that they can focus on the model characteristics rather than on the equation solution technique. Another limitation is the lack of a laboratory demonstration of concepts; although this was addressed by showing videos of laboratory demonstrations. One possible future development is to laboratory exercises for this class.

This course is currently scheduled to be taught for the second time during the Spring 2009 semester. Due to the rotation of elective courses in the MSU chemical engineering department, it is expected that this course will be taught every other year during the spring semester.

ACKNOWLEDGMENT

This material is based upon work supported by the National Science Foundation under Grant No. 0448740.

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Priscilla J. Hill

Priscilla Hill is currently an Associate Professor in the Dave C. Swalm School of Chemical Engineering at Mississippi State University. She received her B.S. and M.S. degrees in chemical engineering from Clemson University; and her Ph.D. in chemical engineering from the University of Massachusetts at Amherst. She has research interests in crystallization, particle technology, population balance modeling, and process synthesis.