AC 2012-3763: PACT: A COURSE IN PARTICLE AND CRYSTALLIZATION TECHNOLOGY

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PACT: A Course in Particle and Crystallization Technology

Abstract

Although many products produced by chemical manufacturers today are solids or contain solids, the training in many chemical engineering departments has little discussion of solids beyond eutectic diagrams in a materials class or a limited discussion of filtration in a chemical engineering class. Specifically, while there is much discussion of a material's composition, temperature, and pressure, other attributes such as particle size and shape are neglected. When size is discussed, frequently it is only the average size. The difficulty is that the size distribution affects the performance of many equipment units including filters, hydrocylones, and crystallizers. While some chemical engineering departments have developed particle technology courses, others have not.

To address this deficiency, a new survey course in Particle and Crystallization Technology (PACT) was developed that blended theory with practical applications. The goal was to provide instruction that was not being covered in the core curriculum that would be helpful for students in their future careers. This course was offered twice as a split-level elective course with undergraduate and graduate students. Topics included characterization of particle size and shape distributions, filtration, continuous and batch settling, cyclone and hydrocyclone design, particle size reduction and enlargement, and crystallization. This course is novel in its inclusion of crystallization, ternary solid-liquid equilibrium phase diagrams, and population balance models in a particle technology course. As part of the course development, an initial version of a computer program was developed to aid students in studying particle breakage modeling by using population balance equations. This presentation will discuss the topics presented and resources used, as well as informal assessment of the course.

Introduction

Particle technology has long affected human culture and technology from early times when people were milling flour for bread and Egyptian pottery was being produced[1], to current usage in the chemical processing industry (CPI). Although Japan and Germany recognized the importance of particle technology and responded by creating powder technology departments in several universities, prior to 1994 the U.S. had few educational efforts in this area[1, 2]. Once it was realized that this lack of particle technology education was hurting U.S. economic competitiveness and that industry needed engineers with more particle technology training, a number of U.S. chemical engineering departments added elective courses[3-6], and academic research centers were developed[7]. In addition to courses, various educational resources were developed including instructional modules[5], a textbook[8], a CD with videos of laboratory demonstrations[9], and teaching examples for fluid-particle systems[10, 11]. It was noted that it is essential to teach students fundamentals based on physical properties[12].

Although many topics are routinely covered in these classes, crystallization is usually neglected despite the fact that it is a key process for forming particles and controlling their properties. In 2006, it was noted that crystallization can be incorporated into existing courses to address the lack of crystallization education[13].

Course Implementation

A new course in Particle and Crystallization Technology (PACT) was taught during the Spring 2007 and Spring 2009 semesters as a split level elective class. During the Spring 2007 semester there were 15 juniors and seniors, and 2 graduate students; and in the Spring 2009 semester there were 21 juniors and seniors, and 1 graduate student. The graduate level course differed from the undergraduate course in that extra assignments were required. The first time the course was taught no textbook was required, but the first edition of the Rhodes textbook[8] was used as a reference book. In 2009 the textbook by Rhodes was required[14]. Since a textbook was not required the first time, the students were provided with lecture notes. The second time the course was taught a supplemental text on crystallization was used[15].

Similar to the core courses, this was taught as a 3 credit course. In 2007 the class met for two sessions of 75 minutes every week, and in 2009 the class met for three sessions of 50 minutes each week. The class was designed for juniors and higher level students to satisfy an elective requirement. For this course, it was necessary for students to have completed the initial calculus courses, the mass and energy balances course, and the fluid flow course. Therefore, students who were in the second semester of their sophomore year would have the prerequisites to take this elective. Other elective courses were taught in the chemical engineering department each of these two semesters, and students were free to choose the elective course that they wanted to take. As in the core courses, the students' mastery of the instructional objectives was assessed through performance on homework and tests. This generally consisted of approximately six homework assignments and three tests during the semester.

The goals of the course were to teach students basic concepts regarding particle technology and to have the students be able to apply the concepts to unit operations involving solids. Given that this was a survey course, only selected topics could be covered in one semester.

The first day of class was used to get the students excited about particle technology and to help them understand its importance to the CPI. Specifically, it was emphasized that 62% of Dupont's products either were solids or contained solids in 1994[1]. Since particle size was used through much of the class, particle size and particle size characterization methods were presented along with data representation methods. Other attributes such as measurements of particle morphology were discussed along with representations of morphology such as aspect ratio.

Other topics included design of unit operations such as cyclones, hydrocyclones, settling systems, filtration systems, particle reduction operations, particle enlargement operations, and crystallization. A detailed list of selected instructional objectives is given in Table 1. From these it can be seen that both theoretical aspects and design of solids handling units were included. To better explain the concepts, selected segments of a video of lab demonstrations were shown. The videos were from the DVD *Product Properties and Process Engineering* produced by BASF[16].

Table 1. Selected detailed instructional objectives for the PACT course.

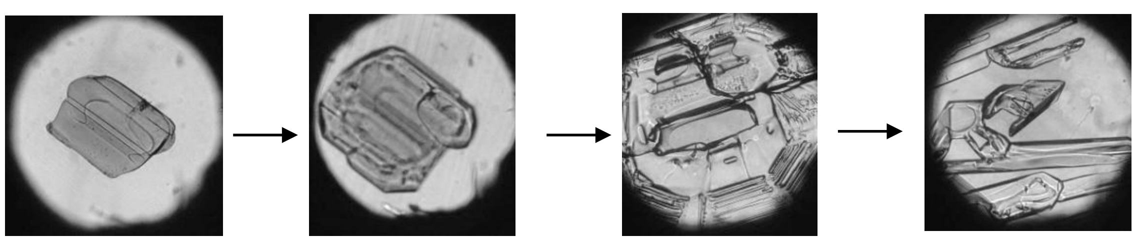
The students should be able to do the following items.

- Calculate an aspect ratio given the major and minor axes of a particle
- Choose an appropriate size analyzer given particle and system properties
- Relate volume to number distributions
- Calculate the equivalent volume sphere diameter and the equivalent surface sphere diameter
- Determine the diameter and number of cyclones needed for a given system and a given cutoff size
- Determine the total efficiency of a cyclone
- Determine a grade efficiency curve based on feed and coarse size distributions and quantities
- Determine the coarse size distribution given the feed distribution and a grade efficiency curve
- Provide a flowsheet for multiple classifiers for functions such as thickening or clarification
- evaluate R_m and α from constant pressure or from constant rate filtration data
- calculate the filtration time required for one batch filtration cycle for both constant pressure and constant rate filtration
- find the terminal velocity U_T given the size, x, for a single particle falling in a fluid,
- calculate the interface velocity in sedimentation in a vertical cylinder given the height-time plot and the concentrations
- calculate the concentration of the final sediment for sedimentation in a vertical cylinder given the height-time plot and the initial concentration
- determine feasible filter types based on operating characteristics, settling characteristics, and the cake growth rate.
- calculate the energy required to crush material using Rittinger's law, Kick's law, and Bond's law given the original and final particle sizes
- explain why it might be necessary to have several crushers/mills in series
- determine the number of particles expected to be produced in several size intervals given the number and size distribution of the parent particles, the specific rates of breakage, and the breakage distribution functions
- identify the birth and death terms in the breakage and agglomeration equations
- calculate solubility using the ideal solution model given the heat of fusion and the melting temperature of the solute
- construct a polythermal phase diagram and an isothermal phase diagram for solid liquid equilibrium
- design a process to completely separate two solids given the feed composition and a <u>polythermal</u> ternary phase diagram of the two solutes and a solvent
- design a process to completely separate two solids given the feed composition and an <u>isothermal</u> ternary phase diagram of the two solutes and a solvent
- determine if a mixture at a given overall composition and temperature will have a component or components coming out of solution based on a phase diagram.
- explain the difference between homogeneous and heterogeneous nucleation
- list methods and choose a method for determining the polymorphic form
- know the difference between mass flow and funnel flow in a hopper
- list some problems with hopper flow
- determine if particles of a given size form a combustible dust

The crystallization lectures began by discussing solubility and the different units used for reporting solubility in the literature. This included discussing sources of solubility information such as technical articles on specific systems, handbooks, prediction from thermodynamics, and experiments. Students were cautioned to be careful with literature data since some compounds form hydrates and data can be reported as g hydrate/100 g solvent or as g anhydrous compound/100 g solvent. Thermodynamic models were presented for both ideal and non-ideal solutions to predict solubility. Freezing point depression was presented as well as the simple eutectic diagram. A procedure was provided for creating a binary simple eutectic diagram given the data and a thermodynamic model. Both isothermal and polythermal ternary phase diagrams were presented and explained.

The next lecture focused on non-equilibrium conditions where there was supersaturation. It was explained that supersaturation is the driving force for crystallization to occur. It was emphasized that not all materials that come out of a solution in a solid form are crystals. Crystals have a lattice structure, whereas amorphous materials are unstructured.

In the lectures on polymorphs, polymorphism was defined as a substance crystallizing into more than one structure. Examples were provided and it was stated that the different polymorphs often have different properties. One of the classic examples is carbon forming graphite and diamond. Students readily recognized that the properties and value of the two substances were very different even though they both are carbon. It was emphasized that at a given temperature and pressure, only one polymorph is stable. To help students understand how common polymorphism is, examples of polymorphs were provided. When discussing polymorph transitions, changes in potassium nitrate in a saturated solution were shown as in Figure 1.



10 hours 16 hours 24 hours

Figure 1. Potassium nitrate disintegrating into needle shaped crystals in 2 days when aged in saturated solution at constant temperature[17].

Selected basic mechanisms of nucleation and growth were discussed, as well as the concept that impurities can sometimes have a strong impact on nucleation and growth rates.

One key point was to help students understand why they should learn some of the theory and why they should be able to read solid/liquid phase diagrams. Specifically, when theory guides the experiments it shortens the development time, results in a faster time to market, and the company makes more money. Without theory it is a trial and error approach. For example, without an understanding of phase behavior, engineers can spend much trying to operate in regions where it will require very low temperatures to crystallize the product, or where it is impossible to obtain a pure product. Therefore, it is necessary to understand theory.

Concluding Remarks

The students thought the course was useful both times the course was taught. During the class, students who had cooperative education experience often commented that they had seen some of these equipment units in industry, but prior to this course they didn't understand what the units did or how they worked. Some students had worked in plants that had a crystallization process, but didn't understand the process before taking this course.

In the standard university course evaluations that the students fill out at the end of the semester, students evaluate the course by responding to statements with a rating on a scale from 1 to 5. A rating of "1" indicates that the student strongly disagreed with the statement and a rating of "5" indicates that the student strongly agreed with the statement. Table 2 shows average responses to three selected questions. The responses were more positive in 2009 than in 2007, but this could be partially due to students not having textbooks for this course in 2007. In both 2007 and 2009, none of the students gave a 1 or 2 rating to any of these three questions. Given that all of the averaged ratings are above 4.0, the students had a favorable view of the class. The responses on the second question in Table 2 indicate that the students thought that they had learned new material in this class.

Table 2. Responses to selected course evaluation statements. Ratings can range from 1 to 5 where 1 = Strongly Disagree and 5 = Strongly Agree.

Statement	**2007 Average**	**2009 Average**
1. The instructor conveyed the course content in an effective manner.	4.1	4.4
2. I learned a great deal in this class.	4.1	4.3
3. I would recommend this instructor to other students if they wanted to learn this subject.	4.4	4.6

Student comments such as "… course should be offered again", "I think this course is relevant and should be offered again" and "PACT was a great class!" indicated that students thought the course was useful and that it should be taught again. Other comments such as "The overall material was interesting as I could relate it to previous work experience", "Overall, I feel this was a very informative course full of applicable knowledge", and "This class is needed! Most co-op jobs revolve around this class!" indicate that the students understood that the material presented was relevant to real world problems. Overall, the students were positive about the class and thought that they had gained useful knowledge and skills from the course.

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