On Laboratory Development for a Curriculum in Particle Technology

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Abstract

This paper discusses the development of laboratory facilities for use with an on going NSF-CRCD project that will establish a three-course concentration in particle technology at NJIT, offered across the engineering curriculum. The main objective of the NSF funded project is to address the urgent need for undergraduate and graduate education in this vital field. The experimental and simulation facility which is being developed will be used mainly for the third course, "Experiments and Simulations in Particle Technology" which is intended for upper-level undergraduates and first-year graduate students, but will also become an integral part of other curriculum activities. This paper describes the equipment and the experiments that are being developed for this purpose, and will also describe the faculty and student experiences with a trial course (Experiments in Particle Technology) that is offered during Fall 1996 semester by the first author.

1. Introduction

Particle technology is concerned with the characterization, production, modification, flow, handling and utilization of granular solids or powders, both dry and in slurries. This technology spans a host of industries including chemical, agricultural, food products, pharmaceuticals, ceramics, mineral processing, advanced materials, munitions, aerospace, energy and pollution. As a consequence of an NSF Combined Research and Curriculum Development (CRCD) grant, an interdisciplinary concentration of new courses in particle technology is now being created at the New Jersey Institute of Technology by professors in several of its departments[1]. It is believed that these courses cover material which is substantially absent in the established engineering curricula. In this paper, ongoing efforts towards this curriculum development are described.

The main objective of this project is to establish a concentration in particle technology, see Fischer etal.[1] for the details. The proposed concentration consists of mainly three courses. These are: (1) "Introduction to Particle Technology" which is intended for upper-level undergraduates and first-year graduate students, (2) "Current Research in Particle Technology" which is intended for graduate students, and (3) "Experiments and Simulations in Particle Technology" which is intended for upper-level undergraduates and first-year graduate students. In addition, several experiments from the third course will be integrated into core undergraduate laboratory courses in Mechanical and Chemical Engineering departments. In the earlier paper[1], the introductory course was described in detail. This paper mainly concerns with the third course, and in particular the development of several experiments and related laboratory facilities. In the next two sub-sections, we address the need for education in particle technology followed by a summary of major accomplishments of this project to date.

1.1. Why Particle Technology?

It is highly likely that most engineering graduates, in particular the graduates of mechanical, chemical, and civil engineering, will encounter products or processes involving granular or powder like materials at some stage of manufacturing. For example, E.I. du Pont de Nemours & Co. estimated that of the 3,000 products that it sold, 62% were powders, crystalline solids, granules, flakes, dispersions, slurries and pastes[2]. A further 18% of the products incorporate particles to impart key end-use properties. In the chemical process industries, a minimum estimate of 40% or $61 billion of the value added by the chemical industry is linked to particle technology. There are major problems encountered in handling and utilization of granular solids or powders because unlike pure fluids or gases for which reliable theories and scale up laws are available, there are few reliable techniques that allow for scale up and design of particle processing equipment. Moreover, although these materials often appear to flow like a liquid which may infer that particle mechanics can be handled by the methodologies which have been developed for analyzing the behavior of fluids, such attempts have often led to inadequate or even disastrous design of such equipment. For example, it was recently reported in *Chemical Engineering Progress* that "Plants handling solids anywhere in the main process train perform more poorly than those processing only liquids and gases. Recently built plants perform no better than those built in the 1960's. Average plants with solid feedstock operate at only 50% of design capacity. One-fifth fail to attain more than 20%, and although startup time for a plant processing raw solids is usually estimated to take 3.5 times as long as liquid/gas processing plants, the actual startup times are 6 times longer!"[2]. In summary, the chances of our graduating engineers encountering problems related to particle technology in their future careers are very high. Therefore it is important for them to be exposed to this subject during their education. It is hoped that our CRCD project helps in overcoming the current deficiency in the engineering curriculum in this vital area.

1.2. Significant Accomplishments

1. Formation of an advisory board with particle technology experts from industry, academia and research laboratories. It comprises representatives from 12 industrial companies, 5 universities and a US National Laboratory. The board meetings are held every March, starting from March 1995.
2. Development and running of the undergraduate lecture course (the first course), “Introduction to Particle Technology: Research Fundamentals and Applications”, during the Fall ‘95 semester by Dr. I. Fischer, for which approximately 20 students, both undergraduates and graduates, were enrolled.
3. The development and running of an advanced graduate course* in Summer ’95 by Dr. J. Luke covering the basics of fluid-particle flows at low Reynolds number, including the hydrodynamics of sedimentation and flow around suspensions. Six students took the course for credit, and a similar number audited the course. Some of the materials developed were used in the graduate lecture course run during Spring ’96.

4. Running of a special graduate course[*] during the Fall ‘95 was taught by Dr. R. Dave, entitled “Image Analysis Applications in Particle Technology,” for which eight students were registered.
5. Offering of the graduate course (the second course) “Current Research in Particle Technology: Micro-Level Modeling”. This was taught by Dr. Dave during the Spring ‘96. Nine students took this course for credit, and one student audited the course. Several external guest lecturers gave presentations during the semester. As a part of this course, a special one day workshop was organized on March 30, 1996.
6. Renovations and expansion of the space in the Mechanical Engineering Center for the laboratory course (the third course), “Experiments and Simulations in Particle Technology” to be offered in the Fall 1996 semester by Dr. R. Dave, were completed.

2. Experiments

The main objective of a CRCD program is to bring current research into engineering curriculum. Incorporation of research material into curriculum is not an easy task for a highly interdisciplinary field such as particle technology. The subject matter of particle technology encompasses several fields and includes a large number of topics. Often, in order to incorporate even one research topic, one may need to introduce the students to many other background topics. This makes it extremely hard to cover the intended material in a limited amount of time. A laboratory course poses even more challenge, since the class time must be split into teaching the basic concepts as well as performing experiments. The time constraint dictates that only a limited number of topics and/or experiments are covered in a single semester, three credit, course. This constraint as well as the expertise/interest of the group of PIs are the major factors in deciding which and how many experiments are selected. Private communications with the Advisory Board also helped in this selection process. For example, several industrial members suggested that we need to include experiments that deal with characterization of particulate materials. This may be in terms of its size, or other properties such as flowability.

During Fall 1996, a trial course involving experiments in particle technology was offered by Dr. Dave. The original objective of the course was to include classical experiments, research related experiments, and also computer simulated experiments (utilizing various available computer simulation codes and animation techniques). However, the trial course only included a selected number of classical experiments and research related experiments. The course development is ongoing, and the next offering of the course is planned in Spring 1997. It is anticipated that the laboratory development will be completed by that time and the course will also include simulated experiments. In the following sub-sections, several experiments are described.

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[*] These courses, offered as special topics, are in addition to the three courses proposed under the current grant and are intended to help the development of the proposed curriculum and instructional laboratory.

2.1. Angle of Repose

One of the most fundamental difference between liquids and powders is that unlike liquid, when a powder is poured, it forms a heap. The heap makes an angle with the horizontal axis. This angle can be of any value, but by definition, can not exceed a maximum value called the angle of repose. If the angle of inclination of the heap increases beyond the angle to repose, the heap will collapse to form a heap with sides which are less steep. It is generally believed that the angle of repose (AOR) is related to the angle of internal friction for the powders, and may be used to predict the flowability of the material out of a hopper[3]. Although the AOR is somewhat related to material flowability, it alone cannot be used to design a hopper (this will be further explained in section 2.5)[4]. However, it does give indication of how big a pile one can expect from a material, and thus for example helps in conveyor belt size determination when the powder is poured onto a conveyor for transportation.

There are four standard methods to measure the angle of repose. These methods are: fixed height cone, fixed base cone, tilting table and the rotating cylinder (see Fig. 1). In the first two methods, the material is poured through a funnel into either a fixed height or fixed base size heap. All the four methods depicted in the figure are utilized in a given laboratory session. Digital camera and image analysis are used to measure the angles of repose. It was observed that each method results in a somewhat different value of AOR for the same material, and within each method, there could be a large scatter in the data obtained. We learned during the course of this experiment is that the manner in which the experiment is conducted, the results can vary, accounting for a good amount of scatter due to “human” factors.

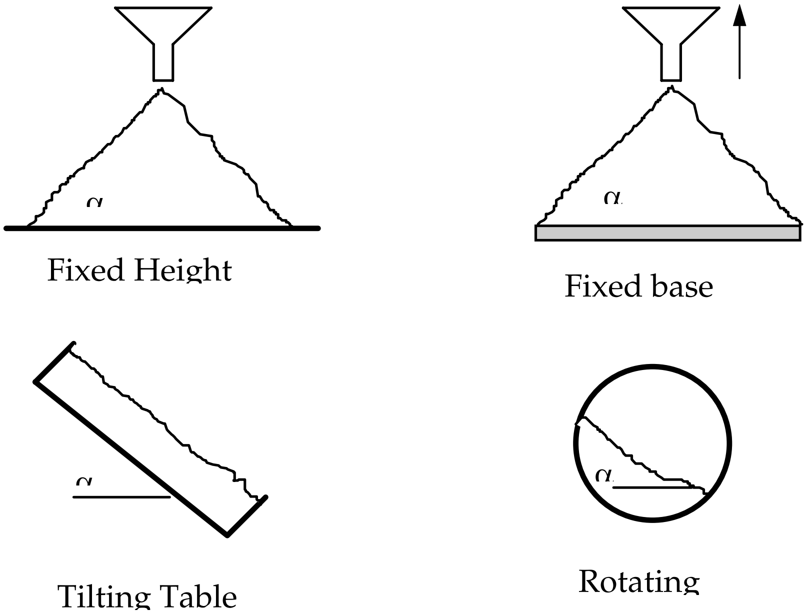

Figure 1. Four different methods to measure the angle of repose.

Material such as sand, flour, corn starch, and powdered sugar were used by different groups of students, and it was observed that more cohesive material had a higher AOR, and the scatter of data for AOR was also higher.

2.2. Particle Size Analysis Using Sieves

Sieving is one of the simplest and oldest methods of determining particle size distribution and is probably used in industry more than any other method[5-8]. It is also one of the most inexpensive methods. Sieving has been considered along with optical and electron microscopy as a method which classifies particles according to geometric similarities. The sieves may be used for particle sizes down to about 38 microns. The sieving apparatus used in our experiments is an Octagon 2000 Vibrated Siever, with a set of sieves ranging from 250 microns (mesh # 60) to 4.0 mm (mesh # 5). By stacking a set of sieves with the sample placed in the top sieve, one could obtain a size distribution curve by weighing the residuals at each sieve. The data can be also used to plot a cumulative distribution. The students analyzed samples of coarse play sand to obtain these results.

Although sieving is relatively inexpensive, the necessary calibration materials are not. The sieves, whether new or not, need to be calibrated against standards to improve accuracy of the results. Due to high cost of calibration standards, our sieves were not subjected to periodic calibration. Besides, while there are many kinds of calibration methods and they all can do a reasonable job on measuring the sieve openings, it is still very difficult to determine the effective sieve opening very accurately. Some of the problems faced in determining the effective sieve opening are: (1) the sieve openings are not uniform, (2) due to some under size particles always remaining on the sieve, sieving is never supposed to be complete, (3) effective sieve opening is not a constant, as it will change according to the manner in which it is vibrated. The second problem listed here relates to the rate of sieving. It is true that in theory, it may take an infinite amount of time for all the undersize material to pass through the sieve. In other words, even after a long period of shaking some undersize material may stay on top of the sieve. There are simple rate models proposed to estimate the reasonable amount of time the material should be shaken. The rate of sieving process basically depends on the probability of the particles of passing through the sieves , and this probability will be affected by the factors such as, the size of the sieve openings and the particles, the shape of the sieve openings and the particles, the properties of the particles, and the loading on the sieve. For the samples that were tested, the students also collected data to study the sieving rate. This was done by measuring the amount of material remaining in the sieve after each time interval and then plotting the rate of discharge curve.

2.3. Particle Size Analysis Using Laser Diffraction Technique

There are many different methods to perform particle size analysis, and the proponent of each method usually considers his/her method to be the best. In practice, however, each method measures an “equivalent” size of the particle, i.e. the effective particle size that is based on the method of measurement. For example, sedimentation based methods would measure the size as the equivalent hydrodynamic diameter, while the sieve analysis method would measure the smallest particle diameter such that the particle would pass through the sieve aperture. This also means that although a particular method may be highly repeatable, it may not give the same result as another equally repeatable technique. Laser Diffraction System (LDS) is considered a quick and reliable method to determine particle size distribution, and is claimed to be based on the “first principles”. However, the results obtained are dependent on the assumptions made regarding the scattering theory as well as the scattering properties of the particles being analyzed.

In our laboratory, we have a Malvern Mastersizer X Laser Diffraction particle size analyzer. It had a good dynamic range, and has interchangeable lenses as well as particle feed systems. The students performed size analysis of samples obtained through the grinding experiment. The samples that they analyzed had a size range from a few microns to about 100 microns. During the course of the experiment, the students learnt about sample collection, preparation, and the use of the Mastersizer. The most basic task of sample collection was perhaps the most difficult, and it was realized that a better sampling scheme would be required. The Mastersizer software allows for selecting different scattering theories and the refractive index models. Students utilized both Fraunhofer and Mie scattering theories, and changed the model used for the refractive index. For each case the results such as the Mode (of the size distribution) and the Residual (of the fit of measured and computed scattering data for all the detectors) were recorded. Several different scattering models were used for each sample, and they were: Generic Solid (20HD), Fraunhofer (2$$D) which corresponds to opaque materials, Glass (20AD), which corresponds to transparent materials, and Titanium Dioxide (2THD). Mark Bumiller of Malvern Instruments was a guest lecturer who presented material on various methods of particle size analysis.

2.4. Size Reduction/Grinding with a Ball Mill

Ball mills are frequently used in industry for particle size reduction. It is estimated that the efficiency of grinding process is very low, perhaps as low as 1 %, thus it is a process that requires much energy, most of which is wasted. For the basic ball mill experiment, we have used a ball mill (Paul Abbe Ball Mill) with a ceramic cylindrical jar and cylindrical Burundum Alumina as the grinding media. The students were asked to perform a simple grinding experiment to study the rate of change in the particle size distribution as a function of time. There are many other variables that can affect the results, such as the amount of charge, the amount, shape, and size of the grinding media, and the rotation speed, but the time constraint of a laboratory session prevents from exploring these factors. In fact, it is a challenge to find a suitable test material that demonstrates the main features of the grinding process in a period of about 2 to 3 hours. For the sake of demonstration, soft gravel like material of size 250 microns to about 4 mms was utilized.

2.5. Material Testing by Jenike Shear Cell for Design of Mass Flow Hoppers

As mentioned in the introduction, the fluid-mechanics hydrostatic model is inadequate for dealing with most particle flow situations, including the mechanics of emptying a supply hopper. For instance, the hydrostatics gives no insight as to what the slope of the hopper should be or about the size of the outlet. Core flow, and rat-holing are frequent problems encountered in industrial hoppers. Also, the outlet can clog, forming an arch-like dome of material above it within the hopper. Typical industrial practice when confronted with this predicament is to whack the hopper with a sledge hammer to provide a shock which will initiate flow. While the results may be satisfactory, very often instead of no flow there is a condition of excessive flow called flooding. In that situation the discharge through the outlet becomes uncontrollable, usually resulting in spillage sometimes to the extent of workmen being buried. Such unsatisfactory design of bins and hoppers has resulted in part, from engineering graduates being unfamiliar with methods developed for considering the handling of particulate materials such as the Jenike methodology[9] for selecting hopper slope and outlet size.

The main issue in hopper design is the material testing procedure that somehow provides the information about flowability and cohesiveness of the material so that one can decide on the hopper slope and minimum outlet size. There are many different methods to test the flowability of the material[10], one of them being the measurement of the angle of repose, which as mentioned in section 2.1 is not adequate for designing a hopper. The method due to Jenike is still considered the most reliable and is perhaps the most widely used technique in industry[9]. The purpose of this experiment is to calculate parameters of a mass flow hopper for a given test material.

There is a detailed standard procedure for using the Jenike apparatus, since the variability and the scatter in the test data is found to be very wide if careful testing is not performed[11]. The data obtained using the Jenike apparatus is used to generate the Jenike Yield Locus (JYL). Each locus is formed by plotting the normal stress (shear weight) verses the pro-rated shear stress. According to Jenike, these yield loci contain all the information needed to characterize the "flowability" of the studied powder. A Jenike shear-cell based material tester was purchased for this experiment. Students tested materials such as flour, powdered sugar, and corn starch. Highly cohesive material such as corn starch poses difficulties in obtaining reliable results. It was found that the test apparatus is not very user friendly, and the task of complete testing is tedious and would require 5 to 6 hours. In the future, we plan to use a flexible hopper to verify the results obtained using the Jenike device to design a mass flow hopper.

The experiments described till now are classical experiments in particle technology. Next three section describe the experiments that are closely related to the research interests and expertise of the PIs.

2.6. Study of Rise of a Single Large Sphere in a Vibrated Granular Bed

Size segregation is often an undesirable outcome of handling and/or processing operations of bulk solids. In a vibrated bed of monodisperse dry granular material, observed behavioral regimes (e.g., heaping, compaction, convection, fluidization, surface waves or arching) are highly dependent on the level and form of the external excitation, and are likely to influence the process of size segregation in a polydisperse bed. In general, a large ball placed at the bottom of a vibrated bed will rise to the surface. In order to understand why a large ball (or intruder) rises, experiments and simulations have aimed at identifying possible mechanisms. Two main driving processes for rising have been proposed. One is the formation of a bulk convective flow of the bed particles which carry at the same time as the intruder. The other is related to local rearrangements of the bed geometry under gravity, as supported by numerical simulations, and is possibly controlled by collective effects like arching or bridges. Experimentally, a "non-convective" and size dependent rising has been observed, but only in a two-dimensional bed. Our recent experiments[12] show three different regimes based on the frequency and amplitude of excitation, and show in three dimensional beds the dependence of rise time of the large particle on its size. In the laboratory sessions, the students are asked to examine various regimes and make observations at different operating conditions.

2.7. Measurement of the Minimum Sintering Temperature of Fluidized Solids using a Dilatometer

Fluidized beds are used in a variety of industrial processes including the catalytic cracking of petroleum to make gasoline, aviation fuel and home heating oil, the conversion of mineral ores into useful products, and the coating and granulation of pharmaceuticals. Many of these processes operate at high temperatures, which cause softening and/or partial melting (sintering) of the solids' surfaces, thereby requiring higher gas velocities to keep the bed in the fluidized state. This behavior is due to the fact that energy imparted to the particles by the fluidizing gas must overcome not only the weight of the bed, but also particle-particle interaction forces due to surface stickiness.

These cohesive forces become significant for a given fluidized material above a characteristic temperature level, usually well below the particle's melting point. This temperature is referred to as the minimum sintering temperature T_s, and is defined as that temperature at which thermally induced surface softening and sintering begins. Fluidized beds, moving beds, and pneumatic pipelines operating above the minimum sintering temperature can result in uncontrolled particle growth, the formation of agglomerates, and, in some cases, total defluidization, as well as excessive wall deposits with subsequent blockage of pipes, orifices, etc. The precise measurement of this temperature, which is an intrinsic property of the solid particle surface, is therefore very important and should be performed prior to any industrial application.

An obvious way to determine the minimum sintering temperature is to perform fluidization-defluidization experiments in a pilot fluidized bed using the granules of interest, and to correlate the minimum fluidization velocity as a function of temperature. At the point where permanent agglomerates form which shows up experimentally as a sudden increase or decrease in pressure drop across the bed, and/or the minimum fluidization velocity no longer corresponds to the values calculated from the well known Ergun correlation, the minimum sintering temperature is obtained. The drawback of this procedure is that it requires the use of a well instrumented pilot fluidized bed and, in addition, a large number of time consuming tests at different temperatures have to be performed.

A relatively simple procedure - developed by professor Robert Pfeffer, his colleagues, and students - to estimate the minimum sintering temperature without the necessity of performing high-temperature fluidized bed experiments, is to use constant rate dilatometry[13-15]. Since, at the point of sintering, the granule's solid surface deforms, a dilatometer can be used to obtain the elongation-contraction versus temperature curve for a porous rod composed of the granular material in question. In this technique, a small sample of powder - about 1-2 grams - is heated at a constant rate in a Theta Industries - Econo I dilatometer which is depicted schematically in Figure 2. The instrument can be used to measure elongation or contractions of the sample while the temperature is increased, decreased or held constant at any rate of up to 15°C/minute in a quartz glass holder situated in an electric heater capable of temperatures of up to 1600°C. The sample holder is constructed so that granular material of particle size down to about 30 μm can be poured into it. The instrument can be operated in a horizontal as well as vertical position and a constant force of up to 0.5 kg can be applied to the sample by adjusting an arrangement of weights in the apparatus.

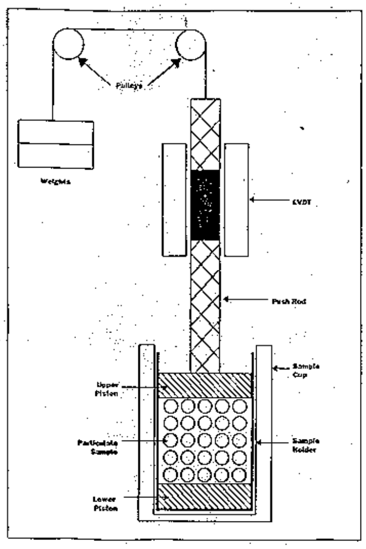

Figure 2. Schematic diagram of the dilatometer

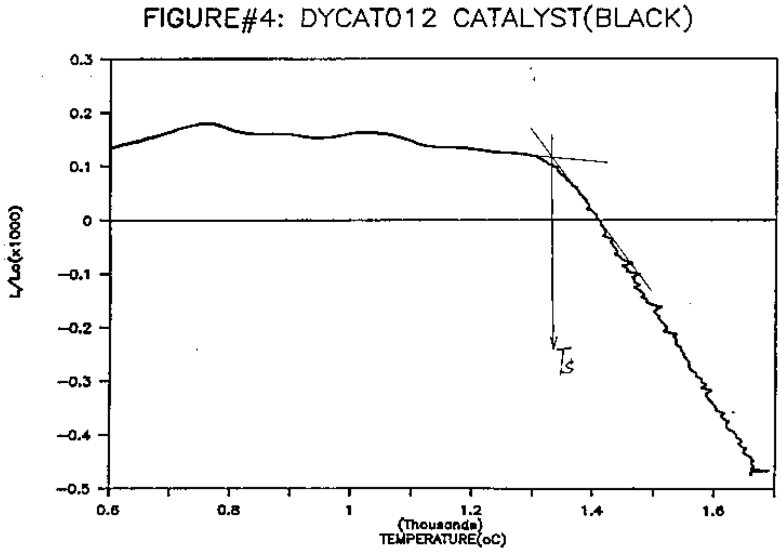

Figure 3. Dilatation-temperature curve

It is found that the general shape of the elongation-contraction curves for many particulate materials such as various polymer granules, coal powders, coal ashes, glass beads, ores and alumina-based catalysts are quite similar. As the heating begins the sample usually dilates due to thermal expansion which may or may not be followed by a plateau region where the dilatation is counter balanced by a contraction as the sintering temperature is approached. At T_S, the surface of the particles begins to soften and the sample contracts at a very fast rate. If the sample is removed from the dilatometer shortly after the minimum sintering temperature (or onset of the rapid contraction) is reached, the particles are always found to be strongly agglomerated.

After a classroom lecture in which the students were introduced to the subject of sintering of powders in fluidized beds and shown dilatometry results for a wide variety of different powders, as well as a video of an experiment showing the actual defluidization of a cracking catalyst at about 1330°C in a fluidized bed, they were given a known alumina-based catalyst sample and asked to measure the minimum sintering temperature of the powder using constant rate dilatometry. The experiment was set up by the students and the dilatometer was programmed to operate overnight at a constant heating rate in a Nitrogen atmosphere. The next morning the students were asked to analyze the results. The dilatation-temperature curve obtained for the alumina-based catalyst is shown in Figure 3. The dilatometer predicted minimum sintering temperature of 1300°C is within 50°C of the actual defluidizing temperature measured in a fluidized bed.

2.8. Study of Particle Sedimentation

The falling ball viscometer is the first in a sequence of experiments concerning suspensions and sedimentation. The experiment familiarizes student with issues in sedimentation. The apparatus consists of a glass cylinder (roughly 100 cm long and 10 cm in diameter) containing a viscous fluid (Ucon fluid 50-HB-3520 manufacture by Union Carbide). Small numbers of particles placed in the fluid at the top of the cylinder may be observed as they sediment along the axis of the cylinder. A funnel and valves at the bottom of the cylinder facilitate recover of the particles. In a lecture student receive instruction on the basic theory of sedimentation at low Reynolds number and its application to size segregation and characterization. Using the manufacture's statement of the physical properties of the fluid, a collection of particles, a balance, a thermocouple based thermometer, a meter stick and a stop watch, students are asked to investigate some of the physical properties of the device. Issues investigated include include particle-wall and particle-particle interactions, inertial effects and thermal effects. A given group of students is asked to focus on a single issue. Particular emphasis is placed on having student explain variability in observations. Reports will be kept on file and future students will be asked to analyze there reports and reconcile their results with those of groups in previous years.

2.9. Other Experiments

During the first offering of the course, only the above mentioned experiments were ready for the use of the students. At present, we have equipment available for more experiments, that will be made available next time the course will be offered. This includes, Patterson-Kalley BlendMaster Blender/Mixer and Armfield Fluidized Bed Experiment for classical experiments. A flexible hopper is being fabricated for use along with the Jenike shear tester. We also have research related equipment that include a Particle Collision Apparatus that utilizes Kodak EktaPro 1000 High-Speed Video Camera to measure pre and post collision velocities of a pair of colliding spheres to study their collision properties, and a magnetic induction based Non-Intrusive Tracking System which allows us to track a single sphere in a mass of other flowing spheres. The latter can be used in a variety of experiments. We have also recently acquired a magnetic mediated dry particle coating apparatus, which will be used to demonstrate dry particle coating process.

3. Discussion

The task of establishing a laboratory facility has been highly challenging, yet it has been equally rewarding. The subject of particle technology is indeed quite broad, and it is apparent that a single person or even a team PIs may not have full expertise required to develop a comprehensive program of education. However, we believe that our efforts will go a long way towards improving the current situation. In our curriculum development efforts, providing the right background material for the wide variety of subjects have been more demanding as compared with delivering the material related to the research of the PIs. The introductory course and the laboratory course are an attempt to provide the broader background, while the graduate level lecture course provides the flavor of the PIs research. As mentioned before, the task of laboratory development is ongoing, and it will require much closer collaboration with the advisory board in the coming year.

Acknowledgments: We are grateful for the financial support from National Science Foundation grants EEC-9420597 (CRCD) and EEC-9354671, as well as Exxon Teaching Aids grants. We also appreciate help from all the students who participated in this course, in particular, Krishnendu Roy and Bodhi Chaudhari. Thank are also due to Mark Bumiller of Malvern

Instruments for presenting a lecture to the students on particle size analyzers. We are also very grateful for useful discussions with many advisory board members, in particular, Karl Jacob of Dow Midland. Lastly, we deeply appreciate help from Rachel Anderson of Dow Midland for spending time with us to help with Jenike test procedure and for providing very useful background material.

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