Principles of Particle Technology: Philosophy, Topics & Experiments

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Introduction

Generally there are key basic experiments that define the development of the subjects that we teach. For example, the experiments of Joule and Reynolds underpin thermodynamics and fluid mechanics. In the classroom we attempt to convey the results and conclusions of fundamental experiments in a period that is a minute fraction of the time in which the original experiments were done and the corresponding concepts developed. The philosophy of the lecture-laboratory course is to enable students the run basic experiments for themselves with the intention that they will develop a deeper understanding of fundamental concepts and relationships from their "hands-on" experiences.

Such is the approach in "Principles of Particle Technology", a junior-level 3-credit class in Chemical Engineering that has two lecture periods and one 2-hour laboratory period per week during a 15-week semester. Particle technology is particularly amenable; key basic experiments can be done within the laboratory session and the apparatus can be simple. In addition, the chronologies of the lecture and laboratory sessions are arranged to be in step with each other. Students work in pairs on the same experiment in a single laboratory session; each experiment is completed within a single session. Typically, the enrollment is in the range of 30-36 students corresponding to 3 laboratory sections. The requirements for the course are admission into the upper division of Chemical Engineering, completion of the two-semester lower-division physics sequence, and at least co-enrollment in Fluid Mechanics. There is a single midterm and a final exam.

Students complete 8 experiments in 8 laboratory sessions and there are 3 homework assignments in addition to the written work associated with each laboratory. The Chemical Engineering Department is an undergraduate-only program, so no graduate-student assistance is available. The instructor-of-record is responsible for setting up each lab, tearing down each lab as well as the grading; another responsibility is the implementation of "continuous process improvement" by improving the experiments and their descriptions. This has been the same person since the course's inception; it was first run in 2003. It was developed from an elective lecture-only course that this individual gave in 2000; it was found that particle technology is a rich subject for class-demonstrations and the experiments to be described grew out of these. The apparatus for the experiments was constructed with readily available components and simple bench-top or hand tools; no machine-shop work was required.

Two texts have been used over the history of the course, viz. **"**Introduction to Particle Technology" (1) and **"**Fundamentals of Particle Technology" (2). The latter one is currently in use; it has the advantage of being free and on-line, and the course sequence follows the first seven chapters. Sections of "Perry's Chemical Engineers' Handbook (3) are also additional sources that the students are expected to read. Normally in course work, students' homework comprises problems at the end of chapters in the assigned text. Here, these problems are essentially replaced by doing equivalent calculations with data that students have generated in the lab coupled with the plotting of good graphs with their data. The experiments are robust in the sense that poor experimental technique will often still lead to data that can be used to do the calculations, but the final results may be poor.

The students' written work from the lab generally comprises: (i) plotting their data – in a linear form where applicable, (ii) doing the basic calculations and these usually involve using quantities derived from model fits to their data, (iii) answering questions which, where possible, involve using their plots. In addition to grading the calculations and the answers to the questions, students' graphs are graded with a scheme set up for ensuring correct axis titles and units, correct numbers of significant figures on axis labels, use of unconnected markers for data, suitable fits to the data with consistent behavior at limits (particularly, the origin) represented by a smooth curve or line, and tick marks on the axes.

Lab 1. Particle-size analysis by sieving
Lecture topics (2-3 classes) include the types of distribution, the bases of describing distributions, methods of measurement, sieve designations, the Tyler & US Sieve series and particle shape with equivalent diameters. The lab comprises: (i) determinations of mesh sizes and openings for samples of screen for which microscopes and rulers are available, and (ii) sieve analysis of −5/8″ pea gravel (Menards), and (iii) sieve analysis of −20 mesh household sugar (Flavorite). Two spinning rifflers of different capacities are used to obtain representative samples as demonstrations at the start of the lab. The importance of representative sampling for particulate samples can not be overemphasized. The larger material is sieved using standard 8″ sieves in the US Sieve Series and a mechanical shaker (Ro-Tap RX-29). The smaller material is sieved is using a nest of hand sieves (Mini-sieve micro sieve set, Scienceware Cat. No. F37845-1000). The purpose of sieving two materials is to convey a sense of the size ranges that are amenable to sieve analysis.

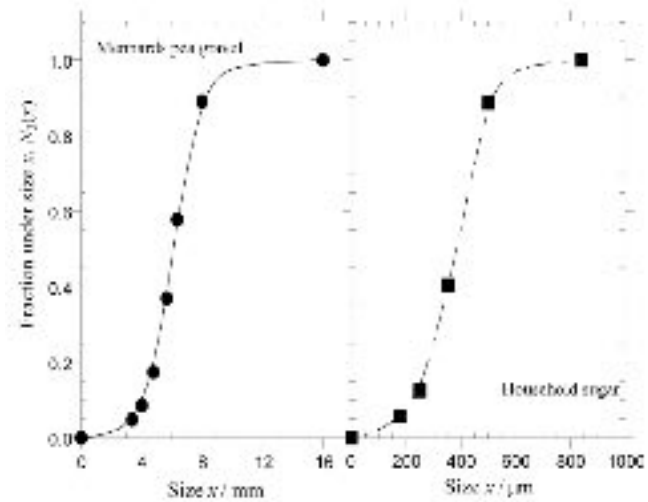

Figure 1. Typical particle-size distributions obtained by sieve analysis
Fig. 1A shows results on a representative sample of −5/8″ Menards Pea Gravel.
Data ●; smooth curve ————————
Fig. 1B shows results on a representative sample of −20 mesh Household Sugar.
Data ■; smooth curve —— —— ——

Students construct size distributions from masses of material retained on each sieve. From their distributions, they determine: (i) medians and modes, (ii) expected fractions below and above certain sizes that do not correspond the screens used, and (iii) fractions within certain ranges. Fig. 1 shows typical results.

Labs 2 & 3. Fluid flow through porous media – water and air through sand.
Lecture topics (4-5 classes) include the characterization of the packed bed (porosity, volumetric concentration, bulk density), superficial and interstitial fluid linear velocity, Darcy's law, permeability, the Reynolds number for flow through a packed bed, empirical correlations for friction (Kozeny-Carman, Carman, Ergun) and the Sauter mean diameter.

Figure 2 contains a schematic diagram of the apparatus used the measuring the flow of water through beds of sand[1] (Sand, White Quartz, −50+70 mesh; Aldrich Cat. No. 27,473-9) in Lab 2. The lab period starts with a brief discussion of the application of Bernoulli's equation to the setup and how the standard form of Darcy's law, usually cast for horizontal flow, should be modified. Students do three separate experiments in which they determine static head-volumetric flow relationships (ΔH vs. Q) for the flow path: (i) in the absence of sand, (ii) with a bed of sand, and (iii) with a second bed containing double the mass of sand. They also measure the corresponding bed heights and the internal diameter of the column. Figure 3 contains typical results.

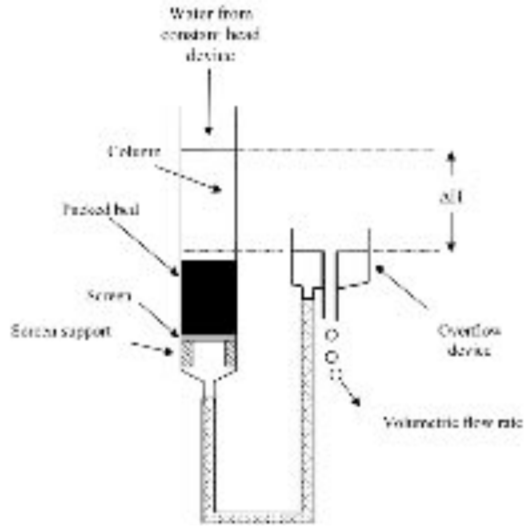

Figure 2. Apparatus for experiments with water flowing through sand

In their analysis of the data, students (i) correct the packed-bed data for the frictional effects of flow through the apparatus, (ii) calculate the beds' porosities, and (iii) manipulate Darcy's law and Kozeny-Carman equation so they then calculate the mean particle size of the sand – they then reconcile their answers with the size description of the sand.

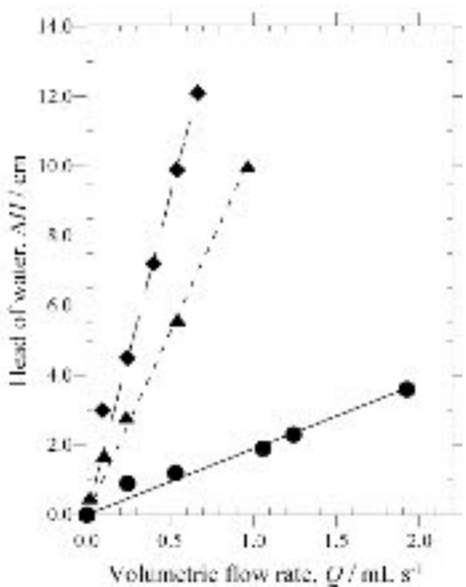

Figure 3. Typical data for the flow of water through sand
Data for flow through the apparatus in the absence of sand ●;
Best-fit line ————————;
$\Delta H = 1.89Q$ R^2=0.97
Data for flow through the apparatus in with a bed of sand, mass ≈ 15g, ▲
Best-fit line – – – – – – – ;
$\Delta H = 10.4Q$ R^2=0.99
Data for flow through the apparatus in with a bed of sand, mass ≈ 30g, ♦
Best-fit line — — — — — ;
$\Delta H = 18.3Q$ R^2=0.98

Figure 4 contains a schematic diagram of the apparatus used the measuring the flow of air through beds of the sand in Lab 3. The period starts with a brief discussion of the application of Bernoulli's equation to the setup and how conditions differ from the previous experiment with water (Do we need to measure a static head term?). Students do three separate experiments in which they determine pressure-volumetric flow relationships (ΔP vs. Q); they are for the flow path: (i) in the absence of sand (mostly they find that this too small to measure, so they do not need to correct their bed data), (ii) with a bed of sand, and (iii) with a second bed containing double the mass of sand. In their analysis of the data, students (i) calculate the beds' porosities, and (ii) manipulate Darcy's law and Kozeny-Carman equation so they then calculate the mean particle size of the sand – they then reconcile their answers with the size description of the sand. Figure 5 contains typical results.

[1] The same sand is used throughout.

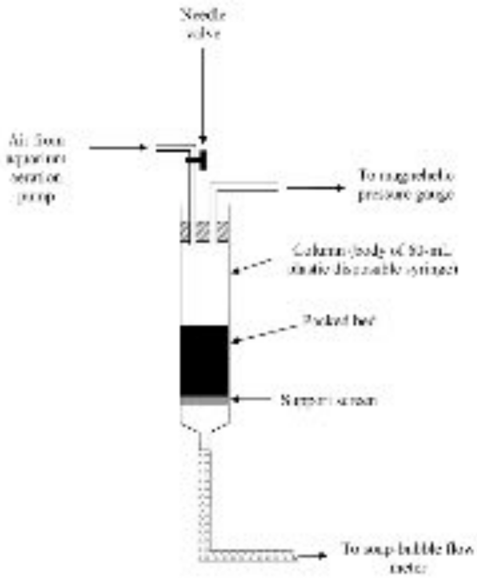

Figure 4. Apparatus for experiments with air flowing through sand

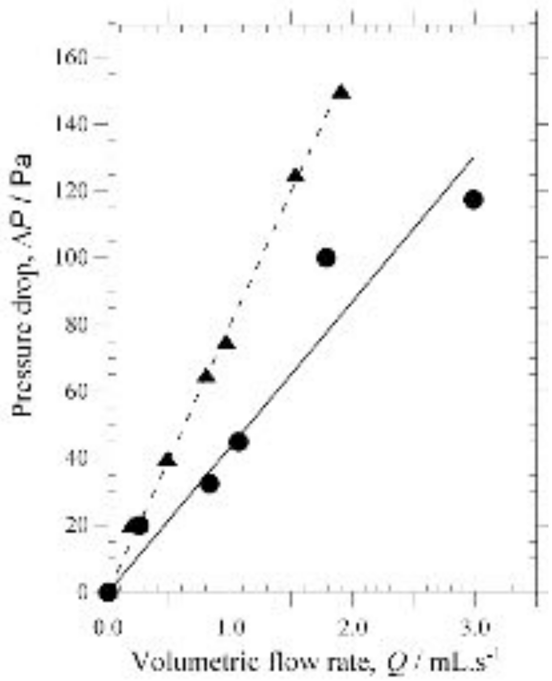

Figure 5. Typical data for the flow of air through sand
Data for flow through sand, mass 37.4 g, bed height 4.1 cm ●
Best-fit line ———————————; $\Delta H = 43.6Q$; R^2=0.93
Data for flow through bed of sand, mass 79.1 g, bed height 8.6 cm ▲
Best-fit line — — — — — — — — ; $\Delta H = 80.0Q$; R^2=0.998
(The data for the flow of air in absence of sand is not shown; the correction is negligible in this case.)

The assignments associated with Labs 2 & 3 are submitted together; in the event that the students recognize that the mean-particle sizes from the two experiments disagree, they should then be stimulated to check their calculations. Discrepancies are often attributable to the use of incorrect values of the viscosities of water and air, and, less often, the density of air. A tacit objective of the two labs is to get the students to remember the magnitudes of the densities and viscosities of air and water.

Lab 4. Filtration rates of −200 mesh limestone
Lecture topics (3-4 classes) include the application of Darcy's law to filtration, terms (medium & cake resistance, specific resistance to filtration, moisture ratio), various cases (constant rate, constant pressure) and the design of constant-pressure systems from batch studies (area & time requirements).

The apparatus comprises: (i) 2-L filter dome that sits atop a round plastic block (Kontes) to which a vacuum pump is attached, (ii) a calibrated vessel inside the filter dome for the collection of filtrate, and (iii) a glass filter funnel with a glass frit inside (the filter medium) whose outlet passes through a rubber bung into the filter dome. Students do three experiments, viz. a drainage experiment (no applied pressure drop) with the filter medium alone and filtration experiments at two pressure drops.

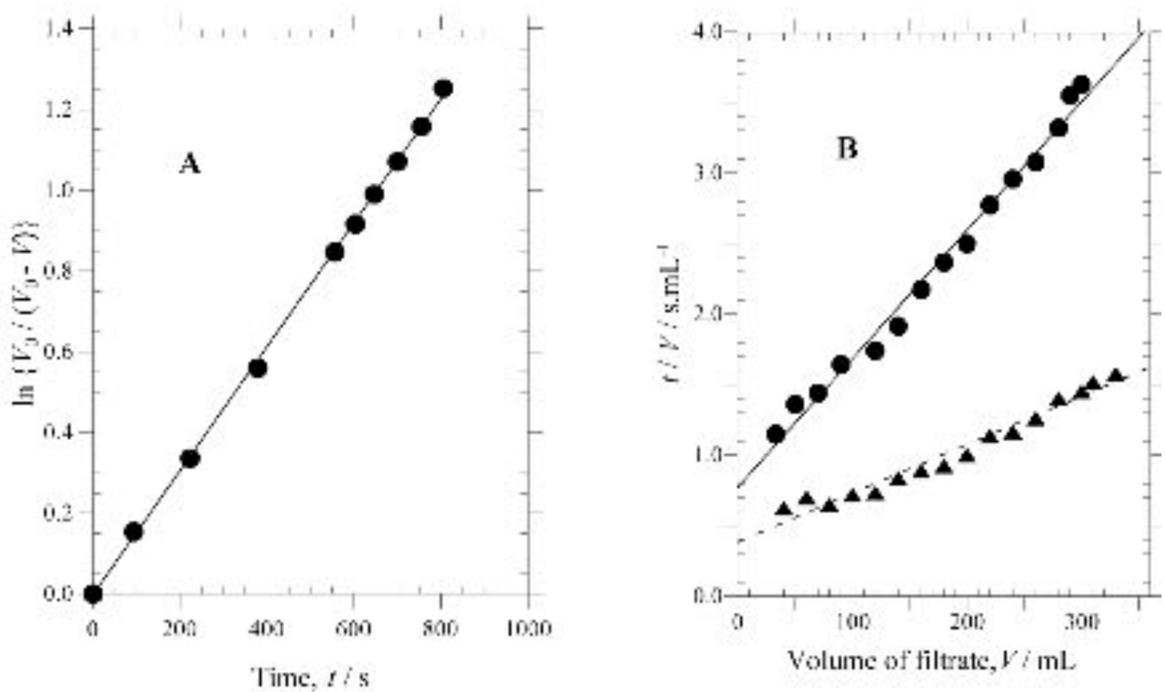

Figure 6. Typical data from the drainage of water (A) through the filter medium and for the filtration of −200 mesh limestone out of water (B).

Fig. 6A shows results from a drainage experiment.

Data ●; Best-fit line ———————————;

$\ln\{V_0/(V_0 - V)\} = 1.53\times10^{-3}t$ R^2=0.999

V_0 is the initial volume of water charged to the filter medium (350 mL) and V is the volume of filtrate at time t.

Fig. 6B shows results from the filtration experiments at two different pressure drops.

Data at ΔP = 8 in Hg ●; Best-fit line ———————————;

$t/V = 9.10\times10^{-3}V + 0.776$ R^2=0.99

Data at ΔP = 16 in Hg ▲; Best-fit line — — — — — — — — — —;

$t/V = 3.40\times10^{-3}V + 0.387$ R^2=0.97

They calculate the following quantities: (i) the resistance of the filter medium (from the plot of the drainage data - see Fig. 6A), (ii) the moisture ratios of the cakes, (iii) the volumes and height the cakes (to be compared with their measured values), and (iv) the porosities of the cakes. From standard plots (see Fig. 6B), they calculate the specific resistance to filtration and the resistance of medium. Finally they calculate the mean particle size of the limestone; they are asked to reconcile their answer with the description of the limestone. They compare their values of the medium resistances from the drainage and the filtration data, the porosities of their two cakes, and they are expected to offer explanations for significant differences in these two quantities.

Lab 5. Single-particle sedimentation

Lecture topics (5-6 classes) include single-particle sedimentation, Stokes' law, the single-particle Reynolds number, the need for empirical correlations to calculate terminal velocity, the drag coefficient, the drag curve and its tabular representation (Heywood tables), applications of the drag curve and the design of sedimentation basins. Students attempt to measure terminal velocities of nine different particles from which they calculate the drag coefficient and the

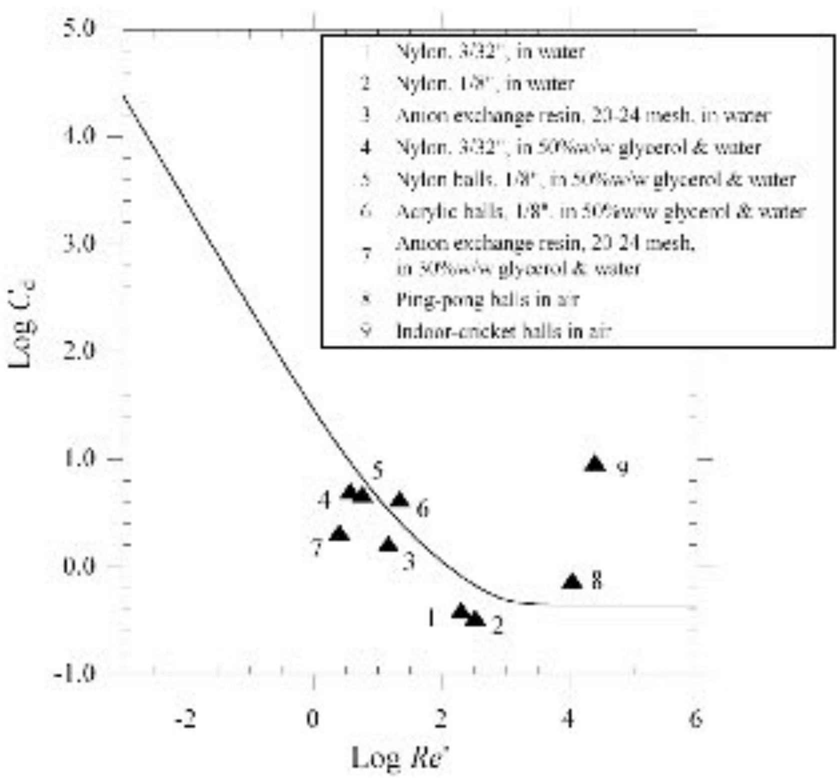

Figure 7. Typical data from the measurement of terminal velocities of single particles in various media.

Data ▲; the drag curve ———————

C_d is the drag coefficient and Re' is the single-particle Reynolds number.

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single-particle Reynolds number; they also plot their own drag curve from the Heywood tables. Figure 7 contains typical results; students are expected to offer explanations for significant deviations from the drag curve.

Lab 6. Particle-size analysis by hydrometry of −250 mesh limestone
This lab requires the direct application of Stokes' law to calculate sizes of particles that have fallen through various distances at particular times after a well-mixed suspension is allowed to settle. They measure the specific gravity of the suspension after various settling times with a hydrometer, requiring the direct application of Archimedes' Principle. The experiment follows that of the ASTM procedure (4) except for a major modification; purpose-built hydrometers are

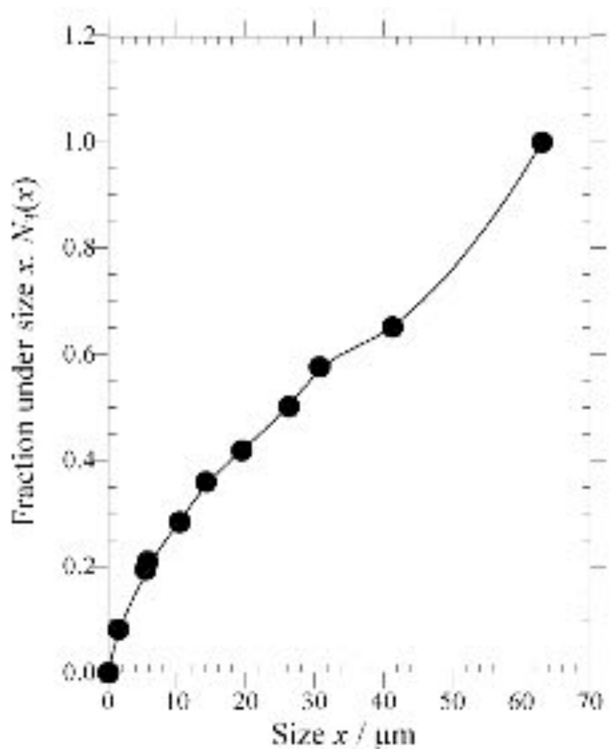

Figure 8. Typical results from the particle-size analysis of −250 mesh limestone by hydrometry
Data ●; smooth curve ————————————

used with a specific-gravity range of 1.000-1.010 so much diluter suspensions are employed than those specified in the ASTM procedure (4). Figure 8 contains typical results. Students use their particle-size distributions to answer the similar questions to those posed in Lab 1.

Lab 7. Batch settling of +325−200 mesh limestone
Lecture topics (3-4 classes) include the modification of single-particle equations to treat hindered settling and the development of the equations for the describing volumetric fluxes of particles. The design of continuous thickeners (area requirements) from batch-settling studies is the concluding topic. In the lab students measure the velocities of the interfaces between settling particles and "clear" liquid after the suspensions, of various compositions, have been well shaken. Students calculate the particle fluxes from the interfacial velocities; they then design a continuous thickener with the best-fit equation of their flux data. Figure 9 contains typical results.

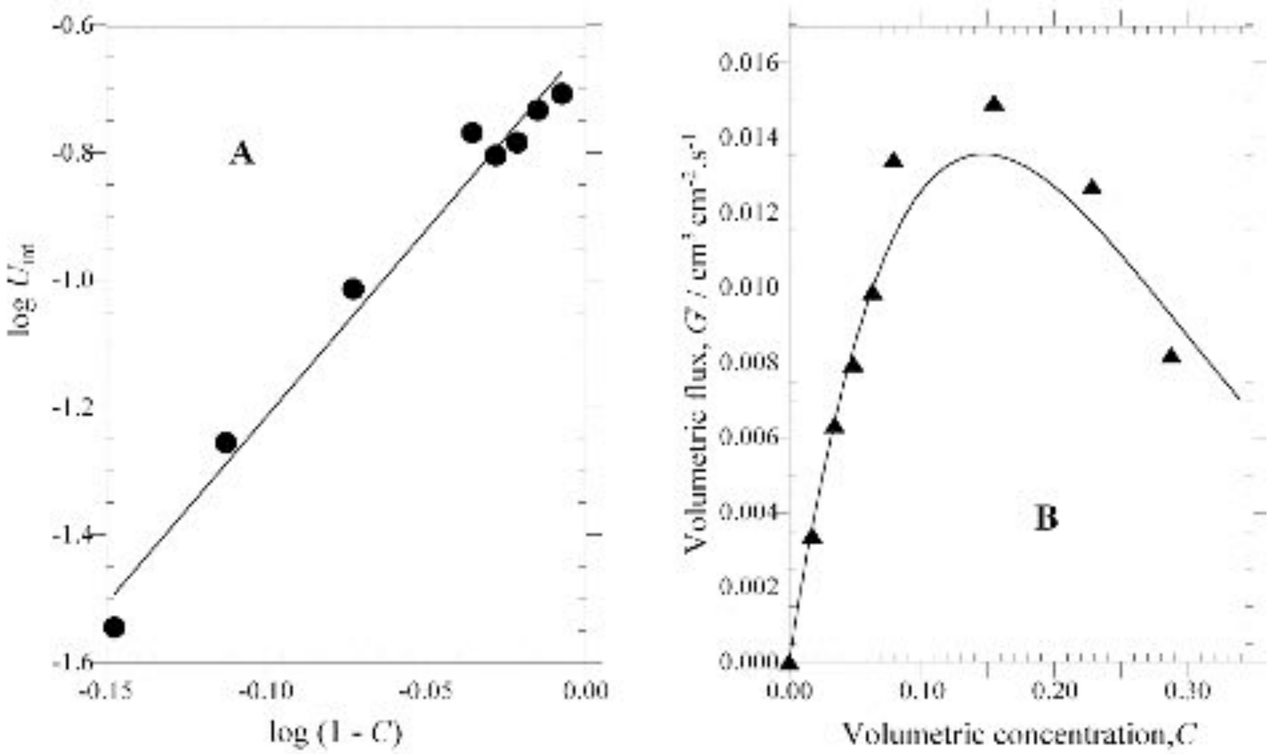

Figure 9. Typical results from batch settling experiments with +325 −200 mesh limestone

Fig. 9A displays the data for the interfacial velocities between "clear" and settling particles, U_{int}, plotted against the initial volumetric concentration of particles, C.

Data ● ; best fit line ————————

$\log U_{int} = 5.86\log(1-C) - 0.628 \equiv n\log(1-C) + \log U_t$ $R^2 = 0.98$

U_t is the single-particle terminal velocity.

Fig 9B displays the volumetric particle fluxes, G', derived from Fig 9A.

Data ▲ ; best fit curve ———————— with the function: $G' = U_t \times C \times (1-C)^n$

Lab 8. Fluidization of sand with water

The treatment of the up-flow section in a continuous thickener in lectures leads naturally into the topics relating to fluidized beds (2-3 classes). These include the fluid linear velocity of minimum fluidization, fluid linear velocity and bed expansion (the Richardson-Zaki equation) and types of fluidization. Figure 10 contains a schematic diagram of the apparatus used in the lab. Students measure the bed height (L) and the superficial linear velocity of the water in the column above the bed. They fit their data to the Richardson-Zaki equation from which they calculate the average single-particle terminal velocity of the sand; with this they estimate the mean particle-size and reconcile their answers with description of the sand. (The size and density of the particles puts the terminal velocities outside the Stoles' law regime, so the use of the Heywood Tables is required.) Figure 11 contains typical data.

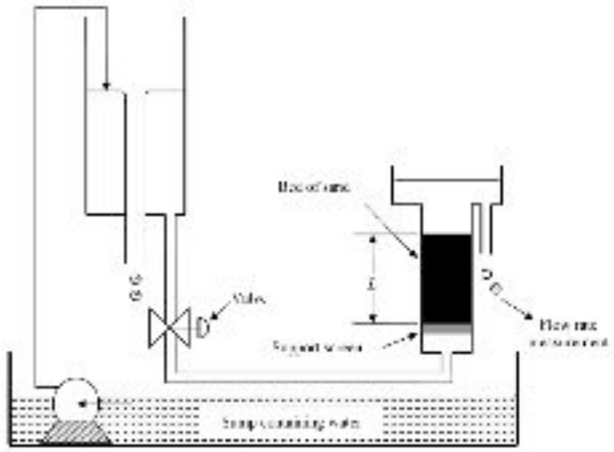

Figure 10. Apparatus for the fluidization of sand by water

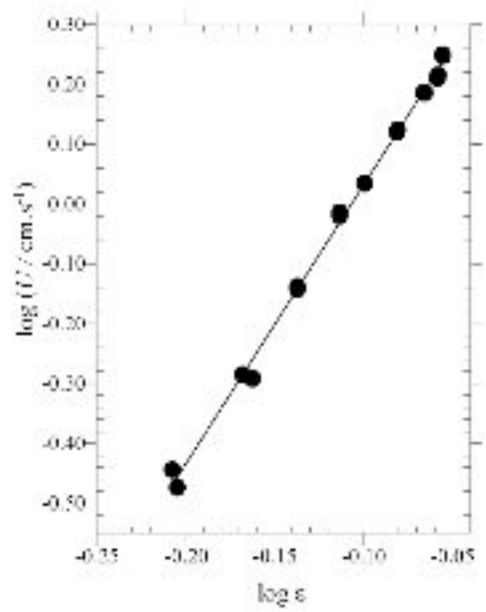

Figure 11. The Richardson-Zaki plot with typical results for the fluidization of sand with water

Data ●; best-fit line ———————————

$$\text{Log } U = 4.65 \log \varepsilon + 0.497$$
$$R^2 = 0.997$$

U is the linear velocity of the fluid above the bed and ε is the voidage (porosity) of the expanded bed.

Lab 9. In-class experiment: fluidization of InsulAdd[2] with air

The experimental work in the course ends with an in-class experiment with air and InsulAdd; this is to demonstrate the different features of gas and liquid fluidization. Before the experiment starts, we estimate the volumetric flow rate for the onset of fluidization (Q_{mf}). InsulAdd fluidized by air corresponds to Group A in the Geldart classification. Figure 12 shows a schematic diagram of the apparatus and Fig. 13 contains typical results.

[2] This is a solid material that contains hollow spheres with density of about 0.8 g/mL. Originally, it was developed to help insulate the exterior surface of the space shuttles – see http://www.nasa.gov/topics/nasalife/green_paint.html. It is now sold as additive for paint to increase its insulating properties – see http://www.nasa.gov/topics/nasalife/green_paint.html

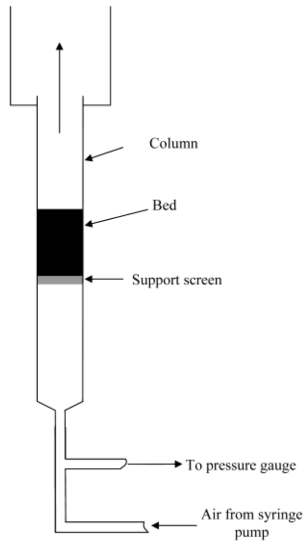

Figure 12. Apparatus for the fluidization of InsulAdd with air

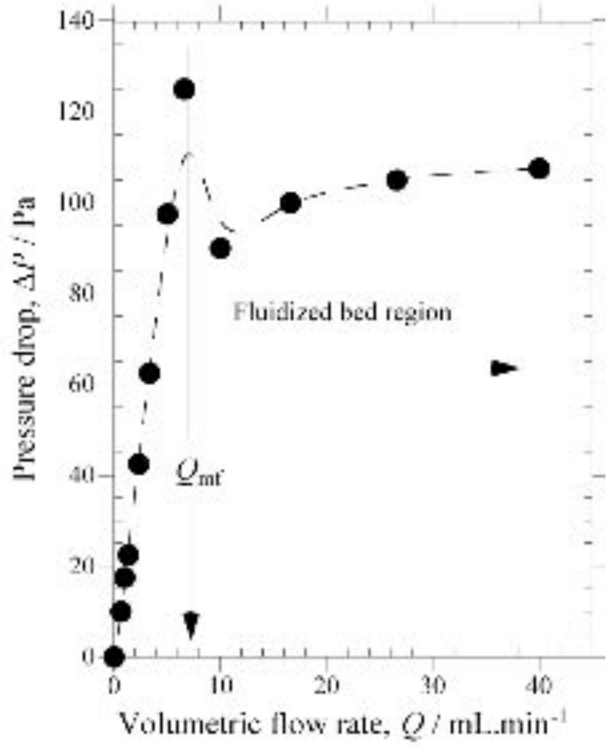

Figure 13. Fluidization of InsulAdd with air – typical results from an in-class experiment

Data ● ; smooth curve —————————————

The vertical line indicates the demarkation between the fluidized and packed bed regions. Q_{mf} is the volumetric flow rate at the onset of fluidization as the flow is increased from the packed-bed region.

In conclusion

The consequence of replacing a single class period in the week for a 3-credit course with a laboratory is fewer topics are covered in comparison to the 3-credit lecture-only version. However, the philosophy and hope behind "hands-on" work is that a deeper and a longer-lasting understanding of the basics will result from the experience; this course is designed to cover the basics of particle technology. Topics introduced are met again in later courses, such as Separations (packed columns) and Chemical Reaction Engineering (fluid-solids reactors). In the capstone design sequence, Chemical Engineering Design I & II, there are at least one or two projects that require particle technology.

The lab component provides additional benefits; students plot up to 24 graphs and the grading is designed to get them into good habits. They are exposed to various methods of measuring flow rates and pressures and well as to the basic technique of determining particle-size distributions. As a whole the course provides the opportunity to reinforce the basic laws and concepts of physics, e.g., Newton's laws of motion, Archimedes' Principle, pressure and friction. In addition the course is an ideal compliment to fluid mechanics using its basic principles throughout, especially those involving friction and the empirical correlations involving dimensionless groups.

Acknowledgement

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(4) ASTM, D422-63 Standard Test Method for Particle-Size Analysis of Soils [Reapproved 1998].

Biographical Information

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Keith Lodge has developed two laboratory-based courses, one in process control and the other in particle technology. He also teaches heat and mass transfer in which he brings a hands-on approach to the class. His general research interests include Thermodynamics, Physical Chemistry & Particle Technology in Chemical Engineering, Environmental Engineering & Science, and Partition Coefficients & Activity Coefficients. Recent publications describe his work concerned with fugacity measurements (*Fluid Phase Equilib.* **2007**, *253*, 74-79), octanol-water partition coefficients (*J. Phys. Chem. A.* **2010**, *114*, 5132-5140) and porosity measurements of taconite pellets (*Powder Technol.* **2010**, *204*, 167-172).