FLUIDIZED BED POLYMER COATING EXPERIMENT

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The fluidized bed polymer coating process is a unique experiment that can have a large impact on student learning and retention. It was first developed for a National Science Foundation Novel Process Workshop[[1,2] and is a highly visual experiment in chemical engineering processes and experimentation. In addition, the coating process is environmentally benign because it has essentially no volatile emissions. The object of the experiment is to place a protective coating on a metal object by first heating it in a hot-air stream and then dipping it into a fluidized bed of thermoplastic powder. The powder is contained within a clear plastic cylinder (acrylic) which allows the students to see and feel the fluidization. At the end of the experiment, students are able to take home a metal object of their choosing, covered with a brightly colored polymer.

The experiment can be used throughout the engineering curriculum. For recruitment at the precollege and freshman level, the fluid motion of the gas and the brightly colored particles attracts the attention of everyone in the laboratory. The brightly colored powders contained within the clear plastic walls of the fluidized bed gives this experiment the professional look of an executive desk amusement. Prospective students and freshmen also are given the opportunity to *feel* the water-like quality of the bed using a rod or a ruler. This is done by asking the prospective student to move the ruler around in a slumped bed (bed without air flow) and then in a fully fluidized bed. The resulting look of amazement is one of those uplifting moments in a professor's life!

Freshmen use the fluidized bed as an example of the engineering measurements of flowrate, temperature, pressure, and coating thickness. They design an experiment to determine the desired coating thickness by varying the dipping time and temperature of the object. Simple Excel plots are produced from their experiments. Sophomores measure pressure drop through the distributor plate to determine the relationship between flowrate and pressure drop. In an advanced fluids class, the fluidization regimes can be identified from a pressure-drop-vs.-flowrate plot. For transport phenomena, the combined heat and mass transport of the coating process can be examined. Many other advanced experiments can be conducted using this apparatus if pressure transducers are placed in the bed. These measurements allow the students to determine the bubble size and frequency,[3] the minimum fluidization,[4] and to characterize the gas-solid flow regimes.[5,6]

This experiment is compact and cost-effective; the cost of fabricating the equipment is about $830. The colored polymer powder makes the experiment enjoyable to watch and to collect data. Student feedback has been extremely positive.

INTRODUCTION

The business of polymers is a major component of the process industry and represents a significant area of opportunity for the chemical engineering profession. The field encompasses many technologies, ranging from polymerization processes used for chemical production of materials to fabrication processes needed to transform the materials into usable products.

The use of polymeric material continues to expand. Advanced polymers are being developed for use in emerging areas of technology such as medical devices, smart packaging systems, fuel cells, and electronic device fabrication. Conventional plastics find extensive use as a material of construction for many products common in daily life. Their low

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weight, resistance to weather and wear, and economical production, make them attractive alternatives to glass, metal, and wood for use in products ranging from food and beverage containers to recreational equipment to automobile components to building materials.

Coating processes fall in the area of polymer fabrication technologies along with molding, extrusion, casting, forming, and calendaring. In parts that must be constructed of metal for structural reasons, a plastic coating may be applied for decorative and/or functional purposes such as electrical insulation, corrosion protection, and abrasion resistance.[7-10]

Fluidized bed coating is a commercially important process in many industrial fields. The main uses of fluidized bed coating techniques are in the pharmaceutical industry for controlled-release coatings on drugs and for microencapsulating drug components;[11,12] protective coatings for glass containers and other components;[13] coating of particles as small as 50μm;[14] fluidized-bed electrostatic coating;[15] chemical vapor deposition in a fluidized bed for metal-coated microspheres for inertial confinement fusion targets;[16,17] and fluidized bed coating of aluminum.[18] Additional examples of fluidized bed coating of materials are available (a recent search in chemical abstracts yielded over 800 references).

The major advantage of powder coating a substrate is that these processes use no solvents and thus provide an *environmentally friendly* alternative to older techniques such as dipping, brushing, and spraying.[19,20] Fluidized bed coating is a novel process that offers the advantages of efficient use of materials (near 100%), the ability to coat irregular shapes, high coating rates, simple and inexpensive equipment requirements, process automation, and smooth and continuous coating applications.

TABLE 1
Industrial Applications of Fluidized Beds

Polymeric Materials	• Gas-phase polymerization of polyethylene • Production of silicon for the semiconductor industry
Biochemical	• Cultivation of microorganisms for the food and pharmaceutical industries
Chemical Synthesis	• Phthalic anhydride • Fischer-Tropsch synthesis of hydrocarbons • Acrylonitrile, maleic anhydride, activated carbon, calcination, roasting of sulfide ores, chlorination, reduction
Petroleum Processing	• Fluid catalytic cracking (FCC) for production of gasoline from oil • Coal gasification • Thermal cracking of naphtha petroleum fractions to produce ethylene and propylene • Fluid coking
Combustion	• Coal combustion • Solid waste incineration • Steam raising
Physical Operations	• Coating metal objects • Drying of solids • Adsorption of solvents

FLUIDIZATION FUNDAMENTALS

Fluidization finds application in many important industrial processes. Examples of fluidization are given in Table 1. In fluidization, a gas or liquid is passed through a bed of solid particles that is supported on a perforated or porous plate. In fluidized bed coating, air is passed through a bed of polymer particles. These particles become fluidized when the frictional force of the air on the particles equals or exceeds the weight of the bed. The minimum velocity required for fluidization can be determined by equating the weight per unit area of the bed to the pressure drop of air through the bed. At this minimum fluidization velocity, all of the powder particles become suspended and the bed exhibits liquid-like behavior. This can easily be observed through the clear plastic wall of a laboratory fluidized bed. As shown in Figure 1, at gas flowrates less than the fluidization velocity, the bed is a fixed bed and there is no movement of particles. At flowrates above minimum fluidization, this bed of Geldart class A particles[21] first expands and then at higher velocities, bubbles appear.

For a given system, minimum fluidization velocity can be determined from a pressure drop vs. air velocity diagram as shown in the flow regimes figure. The value of the minimum fluidization velocity can be compared with correlations presented by Wen and Yu[22] to get an effective particle size of the powder mixture

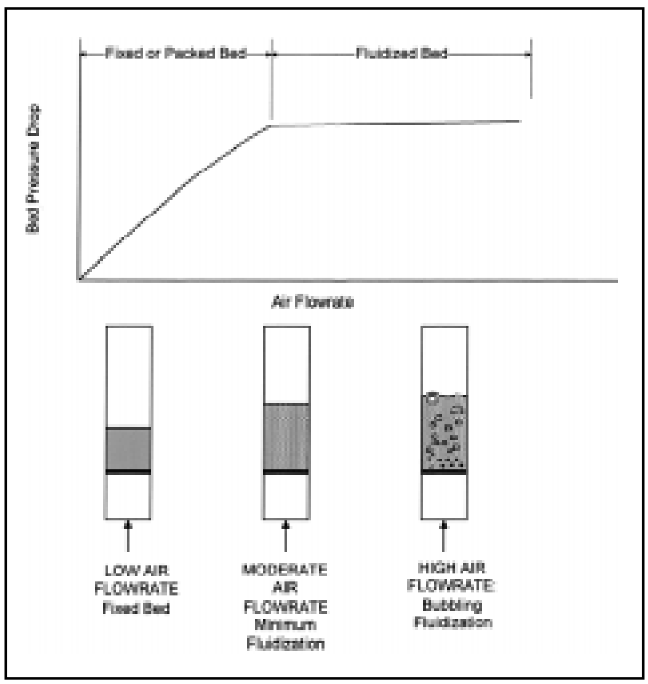

Figure 1. *Fluidization regimes.*

$$u_{mf} = \frac{\mu_g}{d_p \rho_g}\left[(33.7)^2 + 0.0408\left(\frac{d_p^3 \rho_g (\rho_s - \rho_g) g}{\mu^2}\right)\right]^{0.5} - 33.7$$

where

u_{mf}	superficial velocity at minimum fluidization, m/s
μ_g	gas viscosity, kg/(ms)
ρ_g	gas density, kg/m^3
ρ_s	particle density, kg/m^3
g	9.81 m/s^2
d_p	particle diameter, (m)

This paper does not intend to review the theory and equations governing the fluidized bed process. Instead, the reader is referred to the many texts on the subject for additional information.[21,23-25]

As air flow is increased above the minimum fluidization velocity, the bed may exhibit behaviors ranging from smooth fluidizaiton to bubbling fluidization to fast fluidization and pneumatic transport, in which the particles are transported by the air stream. Smooth fluidization is desirable for optimal performance in the powder-coating process.

The liquid-like nature of the fluidized powder bed allows a metal object to be easily dipped into it. The metal object is preheated to a temperature above the melting point of the polymer prior to being dipped. As the hot metal object is dipped into the bed, the polymer particles contact and melt on the hot surface. Additional heat is transferred from the object through the initial polymer layer to additional layers of polymer. After the coated metal object is removed from the bed, it is then allowed to cool. In many of the dipping operations, the outer layer of polymer powder has not completely melted on the object. To give a smooth texture to the surface coating the object may be reheated, allowing this outer layer of particles to melt and become incorporated with the coating. For a given object, the thickness of the coating is dependent on two process variables: preheat temperature of the object and the amount of time it is submersed in the powder bed.

SCOPE AND OBJECTIVES

The purpose of this experiment is to introduce students to basic measurements of temperature, pressure, flowrate, and film thickness using a fluidized bed coating unit. By conducting this experiment, they will also be introduced to the chemical enigneering operation of fluidization. The experiment is broken into two parts. The first part is a demonstration of the basic fluidization regimes. Students operate a laboratory fluidized bed and take measurements to generate a classical pressure-drop-vs.-flowrate diagram to determine the minimum fluidization flowrate for the system. During this part of the exercise, students get a chance to observe the behavior of the fluidized bed over a wide range of air flowrates. In the second part of the experiment, the participants are charged with conducting coating trials to determine process conditions (preheat temperature and dip time) necessary to achieve a specified coating thickness on sample objects.

This experiment is designed to be a cost-effective bench-scale experiment that can be easily integrated into lower-level courses like other experiments we have developed at Rowan University.[26-29] Our goal is to excite lower-division undergraduate students in chemical engineering while at the same time imparting some of the process aspects of emerging technologies. We recognize that larger pilot-scale processes in fluidization or polymer processing have a role in senior courses and unit operations laboratories and are presented in the literature. The use of the experiment described in this paper will have the added effect of generating excitement in students for these subsequent experiences.

FRESHMAN ENGINEERING LABORATORY EXPERIMENT

The following is the laboratory experiment developed for a freshman engineering laboratory course that we call the Freshman Engineering Clinic.[30-31] The experimental system is shown in Figure 2. A team of students (typically three to four) conducts the experiment, and it can be easily conducted in a 3-hour lab period with minimal assistance. The experiment was developed with the support of the National Science Foundation for a workshop on teaching novel processes in the curriculum (see Figure 3).

Freshman Laboratory Objectives

- Using a calibration curve, convert the rotameter readings in mm to a flowrate in mL/min
- Measure the temperature of an object using a bare wire thermocouple.
- Measure the pressure of the inlet air stream using a Bourdon gauge.
- Measure the pressure difference across a fixed and a

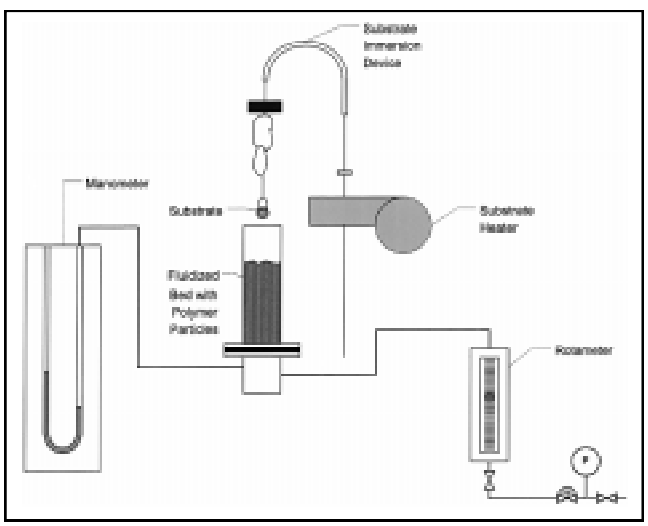

Figure 2. *Fluidized bed apparatus.*

fludized bed using a liquid filled manometer.

- Estimate the thickness of a polymer coating from knowing the surface area of an object and the masses of the coated and uncoated objects.
- Determine the optimum temperature, dipping time, and fluidization regime to obtain an average coating of 0.025 inches.
- Explain the effect of temperature and dipping time on the coating thickness of an object.

Safety Considerations

Specific hazards of this lab include the heating of metal objects to very high temperatures. Students are asked to wear appropriate gloves and to use tongs when possible when handling these hot objects. This is especially true with the use of the heat guns. Safety goggles or glasses are required since there is a possibility of fine powder, hot objects, or line breakages entering the eyes of the participants. The polymer powder used in the coating process is very fine and will produce dust. Loading of the fluid bed column should be done to minimize exposure of the particles. A cloth or Kimwipe should be secured over the top of the column when operating at high air-flow rates where entrainment of the powder can occur.

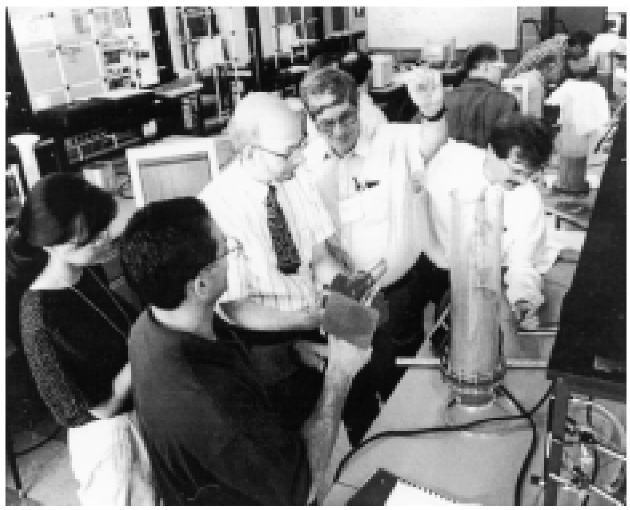

Figure 3. *Fluidized bed experiment being conducted for NSF Workshop on Novel Processes. Drs. Carolyn Lee, Robert Ybarra, C. Stewart Slater, Dave Kauffman, and Robert Hesketh (left to right).*

Relevant Data

The coating material is functionalized polyethylene copolymer-based powder (Polyarmor Powder-PFS Thermoplastic Powder Coatings, Big Spring, TX). It has a particle density of ρ =0.934 g/cm^3. The particle size distribution is not given by the vendor, but a recommended literature value for the average polyethylene particles size is 75 μm.[10] The polymer melting point is 105°C (221°F). The polymers can be obtained in a variety of interesting colors, such as safety yellow, safety green, safety red, safety orange, and light blue. The use of the various colors and mixing of them adds visual fun to this experiment. The fluidized bed is made of Plexiglas and the coated substrates are easily seen in a laboratory. The metal substrate used is a steel washer (Hillman #270067) 1/2-inch nominal size. The exact dimensions are OD 1.376 in (3.495 cm), ID 0.563 in (1.430 cm), and thickness 0.117 in (0.297 cm). The substrate surface area is calculated to be 3.19 in^2 (20.6 cm^2). Any size washer or metal may be coated, and trial runs are suggested to optimize the experiments. We recommend that the students do not dip their team member's car keys or residence hall keys into the fluidized bed!

Required Equipment

The fluidized bed can be fabricated from clear plastic (acrylic) tubing and sheets. The clear plastic tube is glued to a flat sheet flange and a rubber gasket material is used to seal the distributor plate to the unit. The distributor plate is a polyethylene porous sheet manufactured specifically for heat treating fluidized beds. This plate can be obtained from POREX Technologies. The drop mechanism for the metal samples was fabricated by bending stainless steel tubing into a U shape and running a thin metal cable through the center of the tubing. An attachment device is placed at one end to hook a wire loop to it and the other end has an adjustable stop. The wire is weighted using washers to obtain a fast drop into the fluidized bed. The remaining components shown in Figure 2 are standard laboratory units given in Table 2.

Experimental Procedure

Part 1: Investigation of Fluidization Regimes

The first experiment in the freshman laboratory is to have the students investigate the flow regimes of the fluidized bed. In this experiment they identify the equipment and identify the point of incipient fluidization. They are asked to place a ruler into the fluidized bed and feel the difference between a slumped bed (no air flow) and a fully fluidized bed. Students always marvel at the fluid-like behavior of particles. The next step is to obtain a fluidization curve of bed pressure drop as a function of air flowrate shown in Figure 4. In addition, in advanced courses students can make a plot of bed height as a function of air velocity to determine the Geldart particle classification of the powder.[21] In this experiment the freshmen use several measurement devices: air pressure gauge, rotameter, ruler, and a U-tube manometer. This helps fulfill one of the objectives of our Freshman Clinic, which is to introduce process measurements. This experiment also serves a role of reinforcing graph preparation from experimental data using spreadsheet tools such as Microsoft Excel.

At the end of the laboratory the students

1. *Submit a fludization chart (graph). This includes bed pressure and height vs. flowrate.*
2. *Show the value of the minimum fluidization velocity that is determined on the graph (Figure 4).*
3. *Submit a laboratory notebook yellow sheets containing data and a sample calculation of the flowrate.*
4. *Submit sample calculation of step 2 in the next experiment.*

Experiment

Part 2: Polymer Coating

The next part of the experiment is where students coat the metal samples. They are told that a metal part is to be used in an application where the rate of heat transfer through the part is critical. This part will be exposed to a corrosive environment and a coating is required for protection. Increasing the coating thickness increases corrosion protection, but decreases the heat transfer rate. Initial calculations indicate that a coating thickness of 0.025±00.001 inches (0.0635±0.0025 cm) will maximize corrosion protection while allowing for an adequate heat transfer rate.

Using this problem statement, the students conduct a series of pilot runs in the fluidized bed coating system to determine values for the process variables (pre-heat temperature and dip time) that will produce the desired coating thickness. To examine the behavior of the coating process, they conduct runs of constant temperature and constant time. They are given a range of temperatures that start below the melting point of the polymer (105°C, 221°F) and extend it to 450°F (232°C). The dip time ranges between 2 and 10 seconds.

The students determine an average coating thickness from the formula $m_c = \rho At$, where m_c is the mass of the polymer coating, ρ is the density of the coating, A is the area, and t is the desired thickness. The mass of the coating is determined by difference using the electronic balance. A wire is attached to the sample and placed on the hook to dip and remove the sample from the fluidized bed. Using the heat gun, the sample is heated to a temperature approximately 10-15°F above the desired temperature. The sample is then dropped into the fluidized bed for a given time, then removed. After the sample has cooled, the wire is removed and the coated sample is weighed using the electronic balance. To give the sample a more attractive finish, it can be reheated to obtain a smooth finish.

An example of the student data is shown in Figure 5. They find that the coating thickness can be increased by increasing either the coating temperature and/or the time. Many students also find out that if they use a temperature near or below the melting point of the polymer, the polymer particles do not coat the metal object!

At the end of this laboratory the students

- *Submit a summary graph of data from the coating experiment.*
- *Submit a summary paragraph on the effect of temperature and dipping time on the coating thickness.*

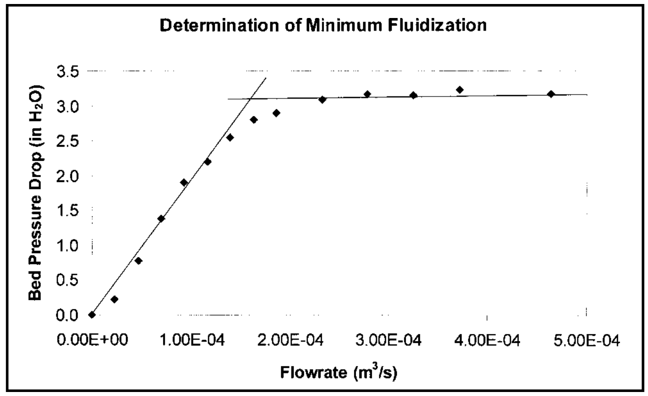

Figure 4. *Determination of minimum fluidization.*

TABLE 2
Parts List for Fluidized Bed

Fluidized bed unit: fabricated by Pemm Corp., Chelsea Industrial Park, Brockway Road, Wappingers Falls, NY 12590. Phone 914-831-5828; (includes polkyethylene porous distributor plate, fine sheet 15-45 µm, 0.25"X42"X44", part no. 4902 from POREX Technologies, 800-565-8777, www.porex.com)	$140
Rotameter, Cole-Parmer, NO44-40, SS float, 41,512 mL/min max of air	$220
Heat gun, Wagner TurboCool H13000, PN 0503835, 1200W	$139
Handheld thermometer, Omega HH-26K	$ 99
Ring stand	$ 47
Polyarmor powder (3-lb sample)-PFS Thermoplastic Powder Coatings, 3400 W. 7th, Big Spring, TX 79720; www.powder-coating.com or e-mail pfs-a@xroadstx.com; Telephone 800-753-5263	$ 40
Three-prong extension clamps (two) - each	$ 20
Tilt stand (for thermometer)	$ 15
Stopwatch	$ 12
U-tube manometer 0.24" H_2O, McMaster-Carr, #3985K25	$ 40
Castaloy clamp regular holder	$ 10
Plastic tubing for air lines	$ 25
Electronic balance, OHAUS Explorer toploading, E1B120, Max 2100±0.01g*	$1,500
Metal samples and disposable hanging wire	

* *Not included in total experiment cost.*

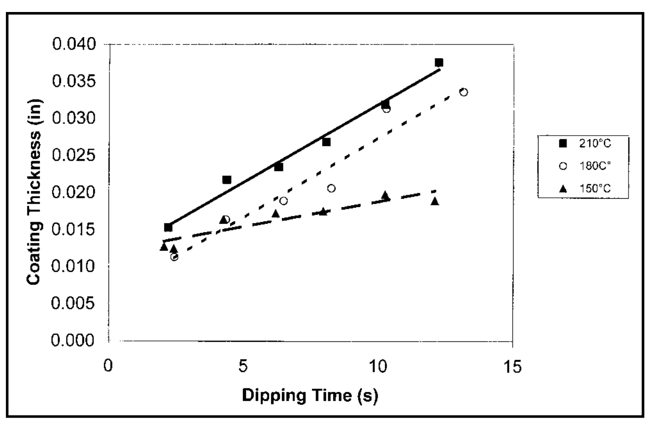

Figure 5. *Example of student coating thickness results.*

- *Predict, based on the data, the time and temperature required for a coating thickness of 0.05 inches.*
- *Submit laboratory notebook yellow sheets containing data and sample calculations (showing units).*

STUDENT FEEDBACK

This laboratory is both meaningful and fun. Students practice principles of measurement and learn about fluidization, coating, and environmental principles. They also have fun coating objects of their own choosing. In addition to the standard samples, students have coated keys and flashlights made in an earlier lab. Student feedback on the fluidized bed experiment has been extremely positive. Representative comments are "one of the best experiments in the Freshman Clinic," "this got me excited about process measurements and chemical engineering," and "I like the visual coating part." One of the course evaluation questions, *Was the experiment interesting and information*, got a 4.71/5.00 score, making it the second highest rated of all freshman laboratory experiments.

CONCLUSIONS

The experiment helps in student recruitment and retention and provides a focal point of laboratory demonstrations to pre-college students. Beyond the visual nature of the experiment are some key engineering objectives that students accomplish. Some of the process measurements performed include: use of a rotameter to measure air flowrate, measuring the temperature using a bare wire thermocouple, measuring the pressure of air stream using a Bourdon gauge, and measuring the pressure difference across a fixed and fluidized bed using a liquid filled manometer. Students also do some problem solving by estimating the thickness of a polymer coating from knowing the surface area of an object and the masses of the coated and uncoated objects and determining the optimum temperature, dipping time, and fluidization regime to obtain the desired coating thickness. Finally, they explain the effect of temperature and dipping time on the coating thickness of an object. We believe that this experiment is both motivational and an excellent learning experience.

ACKNOWLEDGMENTS

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