ChE *laboratory*

POLYMER PROCESSING

For The Undergraduate Unit Operations Laboratory

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In recent years polymers have assumed a commanding position in the chemical industry. According to a recent survey in the 1994 Annual Technical Conference of the Society of Plastics Engineers (SPE ANTEC), the volume of plastics manufactured in 1993 approached 70 billion pounds. With polymers playing an ever-greater role in industry, polymer processing becomes an even more important component in chemical engineering education.

Polymer processing is an engineering specialty concerned with the operations carried out on polymeric materials or systems to increase their utility.[1,2] Typical industrial processing operations include extrusion, blowing, injection molding, and reaction injection molding; each of these operations can involve chemical reactions, flow, or a permanent change in physical property.

The objectives of an experiment in polymer processing are twofold: understanding the governing principles of the operation, and appreciation of the process as applied in industry. Two unit operation experiments involving polymer processing have been developed and incorporated into the unit operations laboratory curriculum at Louisiana State University. One, an experiment involving a single-screw extruder, emphasizes the former, and the second, an experiment involving an injection molding machine, primarily focuses on the latter. A different approach was followed in developing each of the experiments according to the level and scope of the students performing the experiments.

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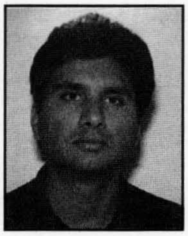

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In the experiment for senior-level students that involves extrusion through a capillary die, the students learn the operation and principles of a single-screw extruder. They are asked to determine the viscosity of a polymer melt (a non-Newtonian fluid) from the experimental data of pressure drop across the capillary tube and the corresponding flow rate. They are asked to infer the relationship between pressure drop and backmixing in the extruder. The emphasis is on understanding the concept of viscous flow of a non-Newtonian fluid. The students are also asked to observe the die swell and attempt to correlate it with the operating conditions. Since die swell is not well understood in the literature, this demonstration generates an appreciation for the complexity of the flow.

In the junior-level experiment, the students learn about the controls and operation of a state-of-the-art injection molding machine. They are asked to find the best operating conditions for producing a part with the given material on the injection molding machine. Emphasis is on the design of optimal experimental planning, statistical variation of the properties of the parts, and sensitivity of the product to different sets of operation conditions.

Groups comprised of three students perform each of the experiments and three periods of three hours each are allotted. The students prepare a preliminary report after the first meeting and then prepare and present a full report on the experiment at the end. The report consists of a description of the experiment's goal, the experimental plan, a description of the apparatus, a discussion of the theory behind the experiment, presentation of experimental data, a discussion of the results, and finally, any suggestions which might improve the experiment and a discussion of the sources of error.

EXPERIMENT 1: Extrusion

Apparatus

The extruder assembly (shown schematically in Figure 1) consists of an extruder equipped with a motor and a die. The extruder is a single-screw extruder, 3/4" in diameter with an L/D ratio of 20, manufactured by Siescor. It is driven by a 3/4-HP DC motor manufactured by G.E.C. The motor is provided with a single reduction worm gear reducer, 321-c series. The diagram also shows the points of measurement of temperature and pressure.

Figure 2 shows the cross section of the capillary die used in the experiment. The diameter of the capillary is 2 mm and the length is 12.5 mm. A Dynisco TPT 232 transducer is used to measure the pressure and temperature of the polymer melt.

A charge of pellets is put in the hopper and the unit is heated up. The temperature profile, including the operating temperature for the die and the temperatures for zones 1 and 2 of the extruder depends on the material of choice. Typical die temperature for polypropylene (PP) and polyethylene (PE) are in the range of 190-230 °C and 150-200 °C, respectively. The screw is not rotated until the temperatures have been stable for sixty minutes (called heat-soak). Zone 1 temperatures are within ± 3 °C of the melting temperature of the polymer. Zone 2 temperature is set between the zone 1 and the operating temperature. The students are asked to find the melting points for the material in a standard reference such as *Polymer Handbook.*[3]

Theory

Shear stress for a Newtonian fluid is a linear function of shear rate ($-dV_x/dy$):

$$\tau_{rz} = -\mu \frac{dv_z}{dr} \tag{1}$$

In a plot of shear stress vs. shear rate, the slope of the resulting straight line is equal to the viscosity (μ) and only dependent upon the temperature. For a non-Newtonian fluid, the shear stress is a function of the shear rate, and the viscosity is dependent upon both the temperature and the rate of shear. Several mathematical models are applicable to describe the stress and strain rate response (constitutive equations) of viscous fluids. For the melt flow of a typical thermoplastic material such as PP or PE, as is used in this experiment, the Power Law model is the most suitable constitutive equation.[2] In cylindrical coordinates, according to the model,

$$\tau_{rz} = -m\left|\frac{dv_z}{dr}\right|^{n-1}\frac{dv_z}{dr} \tag{2}$$

The model contains two empirical constants: the consistency or modulus of viscosity, m, and the Power Law index, n.[4] While m is a strong function of temperature, n varies with shear rate—but for the range of the shear rates used in this experiment n can be treated as a constant. The above relation holds good at a given temperature. Notice that for n=1 the Power Law model reduces to Newton's Law (Eq. 1), where m is the same as the viscosity of the fluid.

Experimental

Material

The material chosen for the following experiments was Linear Low Density PE from DOW, type LLD 2. The reported Melt Index (ASTM D1238) was 1.5. The melting point from DSC analysis was determined to be 128°C. The operating temperature range was from 145°C to 175°C for the experiment.

Part 1 • For this part of the experiment, the students are asked to determine the values of m and n that best characterize the material chosen for extrusion (PP or PE). As mentioned above, n should be a constant and m a function of temperature. Assuming that the cylindrical die on the discharge end of the extruder may be approximated as a cylinder of uniform radius R and that the polymer melt maintains a constant fluid density, a differential balance for the transport of momentum yields

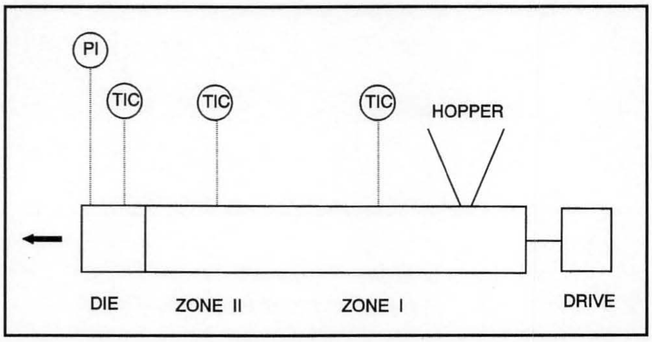

Figure 1. *Schematic of the extruder assembly.*

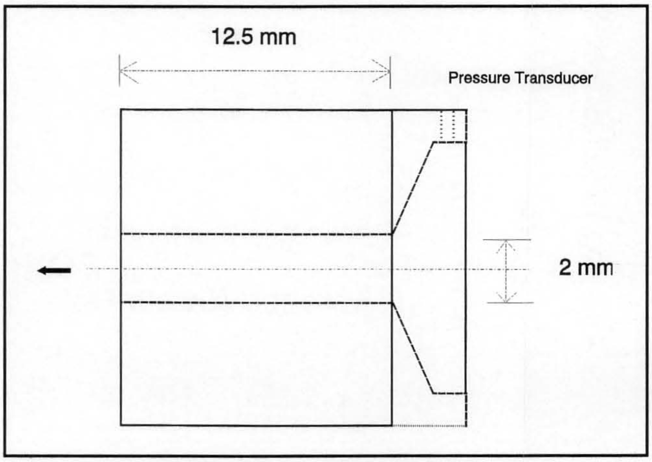

Figure 2. *Schematic of the die.*

$$Q = \left(\frac{n \pi R^3}{1 + 3n} \right) \left(\frac{\Delta p R}{2 mL} \right)^{\frac{1}{n}} \tag{3}$$

where

Q = volumetric flow rate of the extrudate
L = length of the cylinder
ΔP = pressure drop across the cylinder

The students are encouraged to refer to standard textbooks[4,5] to gain an understanding of the shell balance technique. They are required to derive this relationship beginning with a differential shell balance written in cylindrical coordinates.

The data collected in the experiments should consist of volumetric flow rates and polymer melt pressures for varying values of melt temperatures. When the extruder is operating at a steady state (stable pressure drop for a given flow rate), the effects of the speed of screw rotation, the temperatures at the various points in the units, and the pressure drop across the capillary should be made. The volumetric flow rate or mass flow rate can be determined by periodically cutting off the extrudate and weighing the extrudate mass exiting between measured time intervals.

Equation 2 can also be expressed in terms of the wall stress τ_W and apparent strain rate $\dot{\gamma}$, which are expressed as

$$\tau_W = \frac{\Delta p R}{2 L} \tag{4}$$

and

$$\dot{\gamma} = \frac{4Q}{\pi R^3} \tag{5}$$

respectively.

Equation 2 can be alternatively written as

$$\tau_W = m \dot{\gamma}^n \tag{6}$$

The data can now be used to determine the parameters n and m by regressing the linearized form of the momentum equation.

Figure 3 shows a log-log plot of wall shear stress vs. strain rate for PE at different temperatures, and Table 1 shows the values of the parameters m and n determined from the data. The data are then compared with that obtained from a commercial rheometer at Louisiana State University. A Rheometric Advanced Capillary Extrusion Rheometer (ACER) and a Bohlin CS VOR cone and plate rheometer have been used for that purpose.

Another important goal of this experiment is to highlight the difference between the results of the students' experiment and the analysis from the commercial instruments. The students learn that the L/D ratio is an important factor in the accurate determination of viscosity through capillary rheometers. A lower L/D ratio (6.25 for this experiment) results in an incorrect higher viscosity as the flow is not fully developed. The students are expected to comment on the differences between the two results and to point out other possible sources of error.

Part 2 • Another aspect of the experiment is to examine the behavior of the screw extruder as a volumetric pump. The amount of polymer melt delivered per rotation varies with the operating conditions. The screw does not function as a constant discharge device. Data for pressure drop vs.

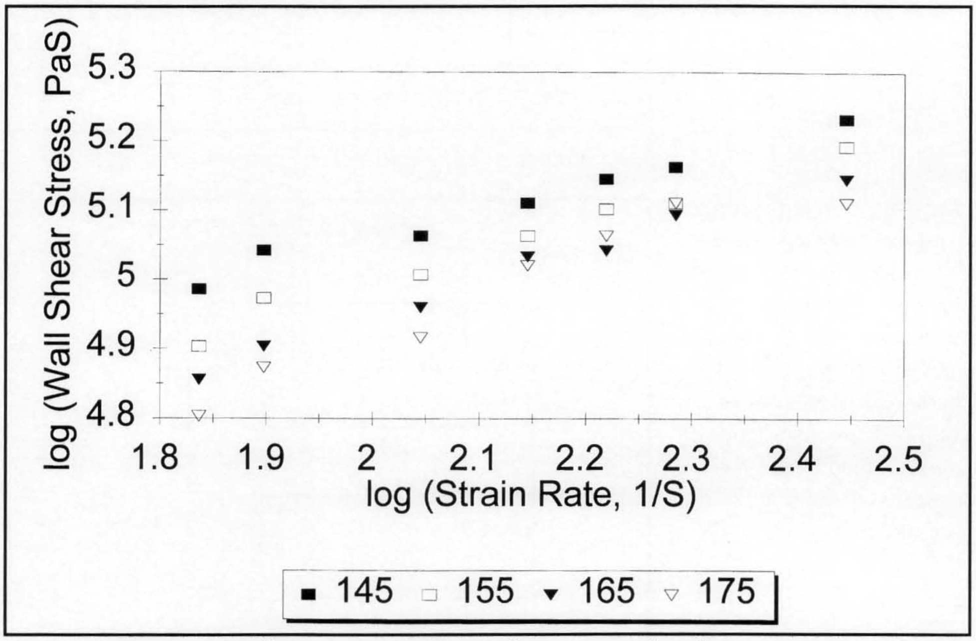

Figure 3. *Wall stress vs. strain rate for a range of melt temperatures.*
■ - 145°C; □ - 155°C; ▼ - 165°C; ▽ - 175°C

TABLE 1
Values of n and m as functions of temperature

T °C	n	m (Pa S^n)
145	0.38	17200
155	0.47	10100
165	0.51	7420
175	0.53	6190

screw rpm at different temperatures is collected. Since the polymer is virtually incompressible in the accessible operating range, a deviation from a straight line will provide a measure of the degree of slippage and back-mixing in the extruder. The data is then correlated with the operating conditions. Figure 4 shows the results obtained from PE at several operating temperatures.

To give the students an appreciation of the complex phenomena of die swell, they are asked to determine the percent increase in diameter. Only a qualitative comment on the phenomena is expected, as further analysis is beyond the scope of the unit operation experiment.

Comments

The temperature range in this experiment is intentionally kept on the lower side of the typical operating temperatures, primarily to observe a considerable difference in viscosity with increasing temperature. Polystyrene would be a good choice as an alternative material for the above experiment. It is readily available and its hardening mechanism is governed by vitrification rather than by crystallization. Rheological properties would change differently as the glass transition temperature is approached rather than as the melting point is approached.

The students are graded on their ability to collect good data, their application of theory to interpret results, their understanding of the limitations of the experiment, and the presentation of written and oral reports.

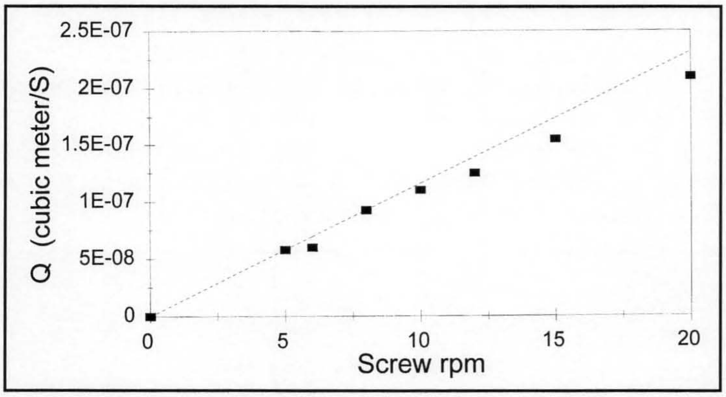

Figure 4. *Flow rate vs screw rpm: T-165°C*

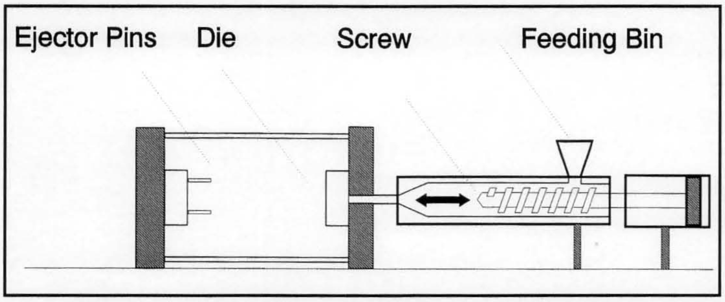

Figure 5. *Schematic of injection molding machine.*

EXPERIMENT 2: Injection Molding

Apparatus

The injection molding apparatus consists of a state-of-the-art Allrounder 170 CMD fully hydraulic injection molding machine manufactured by ARBURG. The machine consists of a melt chamber with a screw that is capable of both rotary and translatory motions, a motor-drive assembly for the screw, and a die that splits in two. The part of the die at the screw end is stationary, and the other half is moved back automatically to remove the part with the help of the ejector pins. Figure 5 shows a schematic of the machine.

The polymer is fed from the bin. At the beginning of the cycle the screw rotates and moves back, taking in a measured amount of the material. During the next phase of the cycle the screw moves forward, building up the pressure, and injects the melt into the die. The screw holds the pressure for a preset holding time, and the material in the die is then cooled for a preset cooling time. The mold opens and the part is ejected out in the last phase of the cycle.

Even though the whole cycle is fully automatic, there are over one hundred variables that can be set individually from the control panel. The variables correspond to operating conditions such as temperature and pressure, and the three phases of the cycle—metering, injection, and cooling. In this experiment, five of the most relevant variables are selected for study. They are dosing volume, injection speed, injection pressure, holding pressure time, and cooling time. To maintain simplicity of experimental design and uniformity among the experiments, all other variables are kept constant.

Theory and Scope of the Experiment

As mentioned earlier, the goals and emphasis for this experiment are different than the one on extrusion. Since this experiment is designed for junior-level students, the detailed mechanism of the injection molding process (which involves nonlinear differential heat and momentum transfer equations describing the fluid flow) is beyond its scope. The goal is to educate the students on the importance of good experimental planning when there are a large number of variables present in a process. They learn to isolate the significant variables in the process.

The students have to understand the basic steps involved in the injection molding operation and learn how the machine is operated. They are asked to plan a series of experiments to collect data that will enable them to determine the set of variables that produces the best part. They have to identify the effect of each variable on the final product and assess the sensitivity of each of the variables under study.

A "dogbone" shaped ASTM D638M 91-A standard tensile test part is formed in the mold for this experiment. The parts are analyzed using the following criteria:

1. Visual Inspection • As is the practice in industry, the part is scrutinized for the following defects:

Flash •	Extra material on the edge of molding which has to be cut off
Bubbles •	Trapped air in the part
Surface Marks •	Marks of the flow lines on the surface that damage the finish
Short Molding •	Incomplete sample
Shrinkage •	Indentation along the mold length

2. Weight of the sample

3. Tensile Strength • The sample was tested on a tensile testing machine (Instron 4301) to determine the yield strength and the modulus of the sample. The gauge length was 50 mm, the grip length was 115 mm, and the crosshead speed used was 500 mm/min according to the ASTM test mentioned above.

Experimental

Material • Polypropylene HGZ 030 (manufactured by Phillips) is used for this experiment. Its melting point was determined to be 168°C using the DSC technique. The reported melt index was 30, and the operating temperatures for the barrel and the screw are fixed at 200°C.

Operating Conditions • The following critical operating conditions are selected for optimization:

1. *Dosing Volume*: Total volume (cc) of the material metered in the barrel. The whole charge is injected in the mold.
2. *Injection Pressure*: The pressure (bars) that is generated by the screw to charge the material in the mold.
3. *Injection Speed*: The volumetric speed (cc/s) at which the screw charges the material. The barrel is divided into five sections and the screw translational speed in each can be controlled individually. In this experiment, only the speed in the final section is allowed to vary; the speeds in the other sections are kept constant.
4. *Holding Time*: The time (s) for which the pressure is maintained by the screw at the mold after all the material is charged.
5. *Cooling Time*: Total time (s) between the instant when the charge is complete and when the part is ejected out of the mold.

The students optimize the properties of the "dogbone" sample by systematically varying the operating conditions. The optimum sample is a complete sample with the least defects requiring the shortest cycle time. The students are required to select twenty samples for tensile test from the samples that they visually analyze. A constraint on the number of the samples curbs haphazard runs and necessitates careful planning of experiments. Of the twenty samples, five must be made with identical conditions so that a statistical deviation among the samples of the same batch and among different sets can be compared.

A summary of the effects of the operating conditions on the product is given in Table 2. The first column lists the operating variable that was studied, and the second and third columns describe, respectively, the effect of decreasing and increasing the value of the variable on the sample.

Comments

Grading is based on the students' ability to plan the experiments, to identify the effect of the operating conditions, their interpretation of a large amount of data to arrive at the optimum condition, and the general organization and presentation of their oral and written reports.

The experiment was carried out on a fully automatic assembly, which is otherwise used for research work. A frequent complaint from the students was the inability to make a "bad" sample. A semiautomatic Newbury injection molding machine (Model H375-RS) is being considered for use only in the undergraduate unit operations laboratory.

TABLE 2

Effect of Operating Conditions on the Properties of the Polypropylene Sample in the Injection Molding Machine

VARIABLE	*LOW*	*HIGH*
Injection Speed	Sample weight is higher Flow marks visible on the surface	Sample weight is lower Produces flash Increased minimum pressure required for molding
Injection Pressure	Needs lower injection speed Produces short shot	High injection speed is possible Needs longer holding time
Dosing volume	Produces short shot	Produces Flash
Holding time	Shorter cycle time Material shrinks back	Longer cycle time
Cooling Time	Difficult to handle sample Deformation of sample as the sample remains soft	Longer cycle time

ACKNOWLEDGMENTS

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ChE book review

THE COMPLEAT CHEMICAL ENGINEER: A Guide to Critical Thinking

by Robert B. Barat, Norbert Elliott

Kendall/Hunt Publishing Company, 111 Purina Drive, Dubuque, IA 52001 (1993)

Reviewed by
Donald R. Woods
McMaster University

The main focus of this book is to improve critical thinking and communication skills, especially in the context of the senior laboratory. The book is founded on the four principles that the authors describe in an essay for instructors: take an interdisciplinary view, think critically, learn to communicate effectively, and consider the impact of technology on society.

The authors produce a framework for introducing and integrating these principles. The model that they use is one of seeing the situation from four different points of view—independent thought, intellectual breadth, cultural breadth, and ethical awareness. They illustrate their application of the model to the process of solving a mass and energy balance, designing a process, and performing a laboratory experiment. The seven-step strategy for doing the laboratory (plan, execute, convert and integrate data, look for patterns, reflect on the quality of the results, argue your results and conclusions, and translate) is well illustrated by excerpts from various laboratory experiments. Although this is a broad framework for thinking critically, few details are given about how to actually do it. The premise is good; the details are missing.

The topics address in the various chapters are:

Chapter 2, Interpreting the History of Chemical Engineering, introduces the heuristic of "particle, wave, field" and asks us to apply these different viewpoints to a study of the several historical decisions important to chemical engineers.

Chapter 3, Working in the Laboratory, describes the purpose of experimentation, provides an 11-step strategy, lists the usual safety regulations, gives seven very good guidelines for experimenting (*e.g.,* penetrate the heart of the experiment), gives checklists to troubleshoot experiments, discusses collaborative work, and provides assessment checklists. On the assessment forms that are given, I would have liked to have seen the criteria given explicitly as well as some items that assess critical thinking.

Chapter 4 on The Uses of Argument in Chemical Engineering focuses more on error bars than on evidence, claims, and qualifications. I would have liked more on the latter.

Chapter 5, Conducting the Literature Search, describes the usual resources and strategies.

Chapter 6 on Ethics, gives a good but brief overview. Some of the professional engineering association's Codes of Ethics could have been given and applied to different cases. The authors' tendency was to encourage the reader to create his or her own code.

Chapter 7, Planning the Laboratory Environment, An Architectural View, discusses the layout of a lab.

Engineers and the Environment, Chapter 8, uses a case study to briefly illustrate the principles.

Communicating Information in Chemical Engineering, Chapter 9, outlines the principles of writing to the audience, and Chapter 10 describes the formats to use for different types of reports and lecture notes. Chapter 11 illustrates how to write lab reports and has a rich set of examples. The marking of the communication is given. I would like to have seen more assessment of critical thinking.

The last part of the book, "An Essay for Instructors," provides excellent suggestions about how to use the topics and assignments in a variety of courses. The problems at the end of each chapter are imaginative and illustrate the four principles upon which the text is based. Some basic feedback forms are given; no index is given.

The book introduces a starting framework for independence, breadth of viewpoint, and ethics. I wish there was more explicit development of the themes. The book gives a convenient collection of material on how to work in the laboratory and how to write laboratory reports, but little is given to develop critical thinking. ❐