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Demonstration of the Power Law Model through Extrusion

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Overview:

Polymer melts are often extruded into simple shapes ("extrudate"), such as cylindrical pellets, flat sheets, or pipe.[1] Polyolefins are among the most common extrudable polymers. With a simple lab extruder we can examine the effect of operating conditions on polymer output and pressure drop, correlate the resulting data using the "Power Law" model for flow of polymer melts and solutions, and use that model to scale up the process to more complex extruders. We can determine the relationship between operating conditions and deviations from theoretical displacement behavior ("slippage") and extrudate shape ("die swell").

Extrusion involves transporting and melting the solid feed, sometimes mixing it with non-polymeric materials, and the pressure build-up and transport of the melt or mixture (**Figure 1**). It is applied to thermoplastic polymers, which deform when heated and resume their earlier "no-flow" properties when cooled. Extruders exist in both single and twin-screw designs, the latter being more commonly used in industry. Extrudable polymers include PVC, polyethylene, polypropylene, olefin copolymers, and ABS (acrylonitrile-butadiene-styrene). Thinner shapes such as films or thin walls (*e.g.*, milk bottles) are normally formed by blow molding. Complex thick shapes such as car body parts are normally formed by injection molding. However, extruders are still used to feed polymer into the molds.

A typical thermoplastic polymer such as a high-density polyethylene (HDPE) copolymer (of ethylene + a longer chain olefin) is used here. It is ExxonMobil Paxon BA50. The operating temperature for the die and zones (**Figure 1**) depend on the material. The flow rate can be determined by timed weighing of the die output, and all other necessary data (screw speed, zone temperatures, pressure entering the die) can be read from the instrument panel.

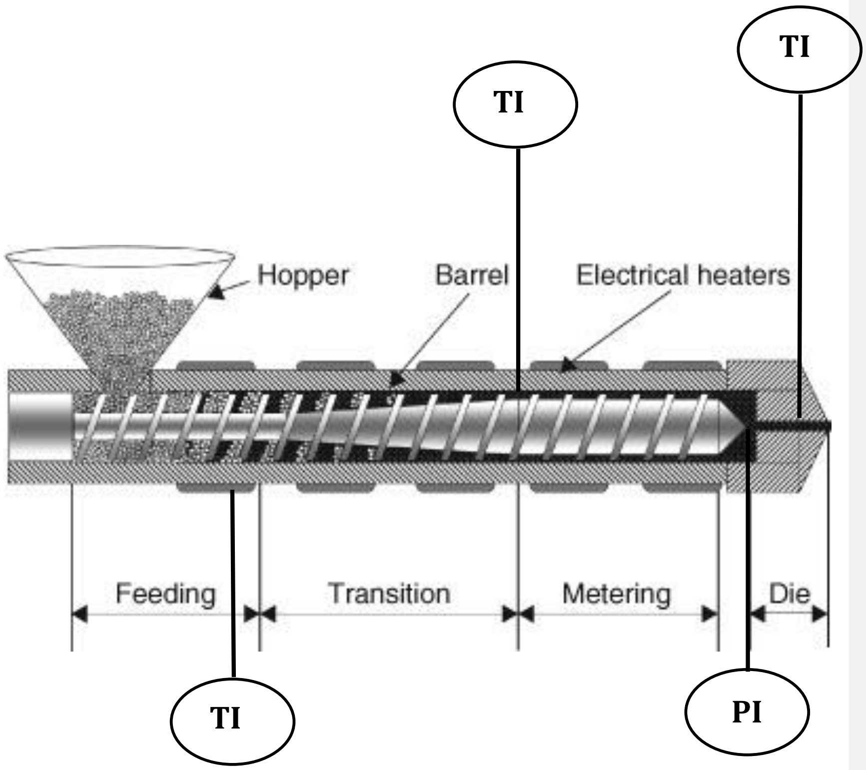


Figure 1. Schematic of the extruder assembly. TIC = temperature-indicating controller, PI = pressure indicator. The die is cylindrical, 12.5 mm long by 2 mm inside diameter.

Principles:[2]

The extruder is comprised of a cylindrical chamber (the "barrel") with resistive heating elements and a helical screw which rotates along the center-line inside. The screw's channels (between the flights) are wide at the feeder end to promote mixing and melting but their widths decrease along the length, to promote pressure buildup into the die. The flights also increase in height such that the clearance between flight and barrel is small. The screw is designed to ensure steady transport from the feeder, allow for reduction in volume as the pellets melt, build up pressure, and transport the melt through the die.

The flow behavior of a polymer melt changes with shear rate, temperature, and pressure. The fluid viscosity decreases with both increasing shear rate and temperature — it is NOT

Newtonian. This property ("viscoelasticity") is important in terms of processing and design.[2-3]

The viscoelastic behavior of polymer melts is described by the Power Law model, which contains two empirical constants, the modulus of viscosity *m* and the index *n*. The parameter *m* is a strong function of temperature. The parameter *n* may vary with temperature. The parameters can also vary with shear rate, if the range is very large.

Procedure:

1. Turn the exhaust "ON" when you are ready to power up the extruder.

2. Fill the hopper and extruder with polymer pellets.

3. Make sure that the motor switch is "OFF". Then turn the main switch "ON".

4. For this experiment, a typical thermoplastic copolymer (ExxonMobil Paxon BA50, melt temperature ~204 °C) of high-density polyethylene (HDPE) plus a longer chain olefin is extruded through a cylindrical die. Typical die temperatures are 220-250 °C.[4] The zone 1 temperature should be 5-20°C above the melt temperature; the zone 2 temperature is set between the zone 1 and the die temperatures. Change the 3 temperature controllers to the correct set points, using the up/down keys on their panels.

5. After the temperatures of all heated zones reach their set points, wait for a minimum of 1 h to melt the polymer inside the extruder. This is called the "heat-soak"; it is important because any solid left in the melt will exert an excessively high pressure on the die and result in unsteady flows.

6. Turn the motor "ON". There are two switches. Set the desired speed, starting with a low RPM and gradually increasing it as polymer is seen exiting the die, until the lowest desired speed is reached. A speed range of 10-100 RPM usually works, but is highly temperature-dependent. Do not exceed 3,000 psi die pressure under any circumstances, and try for <2,500 psi.

7. Run for ~10 min after reaching the desired speed before collecting data. Check the hopper periodically to ensure it has resin pellets.

8. Measure the flow rate by cutting (with scissors) and weighing the extrudate mass exiting between measured time intervals. The die is very hot and should not be touched without safety gloves. The pans being used can be pre-and post-weighed to collect samples; the polymer will not stick to the weigh pans. Measure the diameter of the extrudate ribbon with a micrometer.

9. When switching to a new speed again wait ~10 min before collecting data. If instructed to work at more than one die temperature, wait 15 min after the new die temperature is reached before collecting data. Lower the speed initially if you raise the die temperature to avoid wasting polymer during the transition.

10. After collecting all desired data, turn "OFF" the extruder motor switches.

11. Make sure to turn "OFF" both motor switches. Then turn "OFF" the main switch.

12. Calculate the volumetric flow rates Q from the mass rate and the melt density of the polymer (specific gravity = 0.949).

13. The Power Law model for the shear stress (flow in the z-direction, stress propagation in the r-direction) in the die is:

$$\tau_{rz} = -m\left|\frac{dV_z}{dr}\right|^{n-1}\frac{dV_z}{dr} \qquad (1)$$

When this equation for the stress is substituted into the z-direction equation of motion, and only the τ_{rz} viscous stress and z-pressure derivative retained (the left-hand side inertial terms are negligible for most polymer flows because the viscosities are so high), there results an ordinary differential equation that can be solved to yield:

$$Q = \left(\frac{n\pi R^3}{1+3n}\right)\left(\frac{\Delta P \quad R}{2\ m\ L}\right)^{1/n} \qquad (2)$$

Where ΔP is the pressure drop through the die, and L and R are die length and radius, respectively. Using **Equation 2**, you can determine the values of m and n that best characterize the material at a given die temperature. A template spreadsheet may be provided. You can linearize Equation (2) and then use both linear and nonlinear regression to find m and n, and compare the results. If you have data at two temperatures, you would also perform separate regressions on the two sets of data (why?).

Commented [ASW1]: This is another place where the manuscript needs to be updated from a lab experiment into a manuscript fitting our format.

Commented [KMD2R1]: Don't understand this. There are no instructions in your format on data workup, but in any advanced lab that's an important part of the lab, whether you would include it under "procedures" or "results". I explained in the e-mail sent with the old draft that the spreadsheet is a template file to make the calculations easy for someone who has not taken fluid mechanics yet. Normally what we do is to supply the template if the lab is performed by students who have had fluid mechanics. We don't supply it if they have had it, since they should be able to figure out how to regress the data themselves.

14. Critically examine the data to see how well they are fitted by the power-law model, and whether portions of the data are in fact even consistent with the model.

15. If you understand the concept of propagation of errors, you can estimate the one-sigma confidence limits on m and n at each temperature from the one-sigma confidence limits of the regression parameters.[6]

16. Report the accuracy of the regressions in terms of the average percent relative deviations, and also report the shear stress and shear rate ranges of the data. Major deviations from the Power Law model should be explained. You should also compare your results to results for a similar polymer (HDPE or an ethylene copolymer), using the vast literature on polymer rheology.

17. Also examine the flow/displacement behavior of the screw extruder. Use the Q vs. screw RPM data. Theoretically, the flow should scale linearly with RPM, because the polymer is virtually incompressible, and all flow in the extruder should be within the flights of the screw. Any deviation from the linear relationship provides a measure of change in the degree of slippage and back-mixing in the extruder.[5] Explain why the amount of slippage varies in the manner observed.

18. Compare the diameter of the extrudate with the die diameter at the lowest temperature you use to find the "die swell ratio" (extrudate diameter/die diameter).[7] Is there any correlation between operating conditions and the "die swell ratio"? Does the character of the extrudate change at high shear rates? What do you think are the reasons for these changes, if any?

Representative Results:

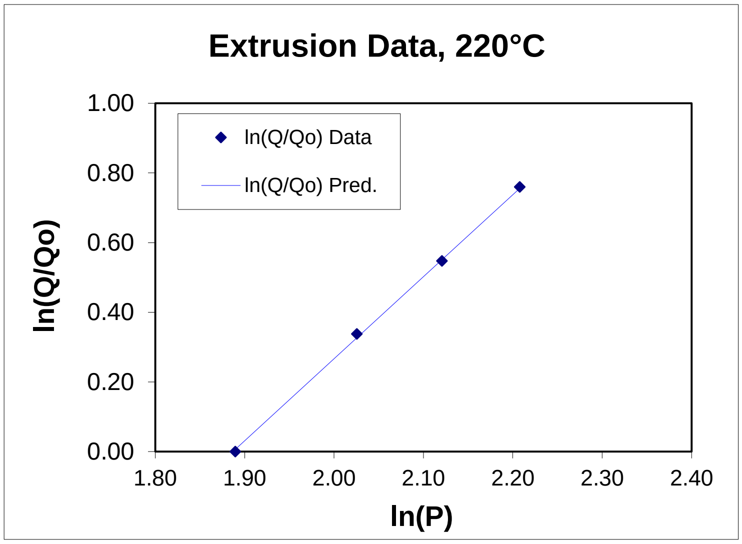


Figure 2: Results depicting the relationship between pressure (P) and flow rate (Q). The linear regression to the Power Law model appears to be a good fit.

Some data and a fit to **Equation 2** by linear regression are shown (**Figure 2**). These data spanned the ranges: mass flow = 11-28 g/min, shear rate (at wall) = 35-85 s^{-1}, viscosity (at wall) = 760-460 Pa·s.

The linear regression fit was good (R^2 = 0.9996). However, note that in order to apply linear regression to **Equation 2** you must regress the log ratio of Q to Q_0 (Q_0 can be any data point, but the lowest Q was used here), so you lose one degree of freedom. This is not the case for nonlinear regression, so in principle the nonlinear regression should give a better fit. The Power Law index and modulus of viscosity were calculated from the data shown. The power law index (n) is 0.42 and the modulus of viscosity (m) is 2.2 x 10^{-2} MPa*s^n.

Flow rate appeared to have some slight effect on the die swell ratio. However, increasing flow rate had no effect on polymer slippage, at least for these data (**Figure 3**).

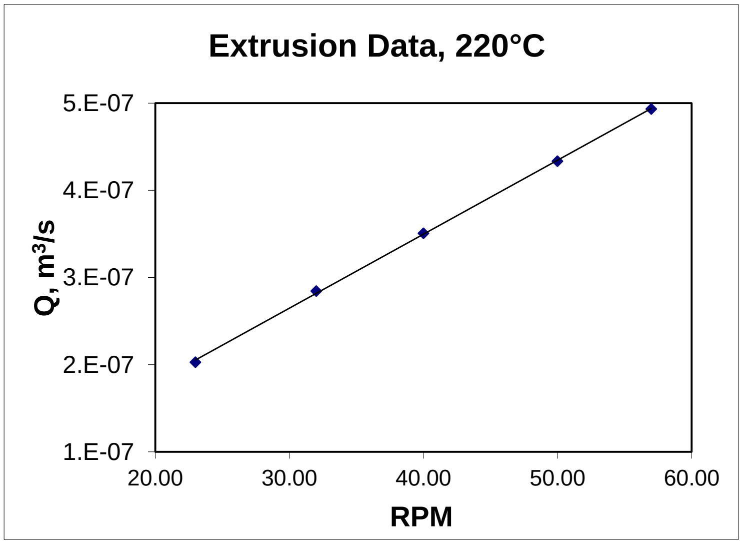


Figure 3: Results depicting the relationship between volumetric flow rate (Q) and speed in RPM. The line is the Excel-computed linear trend line. Since the relationship looks linear, there is no slippage observed for these data.

Summary:

Polymer extrusion begins by melting polymer resins that enter the extruder through the hopper. The flow of the molten polymer depends on the viscosity (ratio of shear stress to shear rate) behavior of the substance. The polymer leaves through the die, and is shaped to desired dimensions. The flow of polymer is expected to follow the Power Law model. The Q vs. ΔP relationship calculated by this model takes on a simple form for flow in a conduit of simple geometry (here, the die). From the flow, speed, and temperature measurements, the Power Law constants can be calculated, and other quantities such as shear rate, shear stress, and degree of slippage.

The take-home messages of this experiment are the mechanics of the Power Law model, how it is used in conjunction with the z-direction equation of motion to analyze the flow of a non-Newtonian fluid, and how greatly the flows and viscosities change in response to screw speed and T. Viscoelastic fluids have a Power Law index <1, compared to 1 for

Newtonian fluids. That means that as we increase the speed the viscosity decreases and we need less power/mass to flow the melt.

Applications:

Extrusion is a primary process for creating many types of pipes and tubing, films, wire insulation, coatings, and other plastic products.[2,8] Extrudable products include polyvinyl chloride (PVC), commonly used for piping, polyethylene and its copolymers, often used for packaging, polypropylene, ABS, acetals, and acrylics.[2]

Extrusion is an efficient process for converting polymers into simple shapes. However, many extruders also function to mix non-polymeric materials with polymers; the helical flow through the flights promotes efficient mixing. Such non-polymeric additives include plasticizers (organic compounds used to lower the viscosity and make the product more ductile), antioxidants, and flame retardants. Even inorganic fillers such as carbons, clays and talc can be added, within limits (because they don't melt). Fillers modify the mechanical properties of the final product, often imparting more toughness.

Other extrusion processes such as blown film extrusion and over-jacketing extrusion can create unique products, but they are more specialized, for a limited range of products. But a key use for extruders is to feed the products to either blow or injection molders, and injection molding makes a wide variety of complex products ranging from car body and under-hood parts to toys to gears.

A variety of extrusion techniques exist. Over-jacketing extrusion is used to coat electrical wires, while tubing extrusion (annular die) creates industrial and residential piping. Plastic sheets are created by flow through a die that looks similar to a coat hanger.[2,9]

Extruders are also frequently used in food processing.[9] Products such as pasta, bread, and cereals are extruded in mass quantities. Starches are most commonly processed in food extrusion due to their moisture content and viscosity profile. The process of melting in plastic extrusion becomes the process of cooking in food production. Other food products created through extrusion are confectionaries, cookie doughs, and pet foods.

Legend

Figure 1. Schematic of the extruder assembly. TIC = temperature-indicating controller, PI = pressure indicator. The die is cylindrical, 12.5 mm long by 2 mm inside diameter.

Figure 2. Results depicting the relationship between pressure (P) and flow rate (Q). The linear regression to the Power Law model appears to be a good fit.

Figure 3. Results depicting the relationship between volumetric flow rate (Q) and speed in RPM. The line is the Excel-computed linear trend line. Since the relationship looks linear, there is no slippage observed for these data.

Materials List:

Name	Company	Catalog Number	Comments
Equipment			
Single-Screw Extruder	SIESCOR		3/4" diameter screw, L/D ratio = 20
LLDPE	Dow	LLD2	Alternative polymer to BA50, melting temperature= 191 °C, s.g. = 0.930
HDPE Copolymer	ExxonMobil	Paxon BA50	Melting temperature= 204 °C, s.g. = 0.949
¼ HP DC Motor	MINARIK		Single reduction worm gear reducer, ratio 31:1

References:

1. Some basics of operation can be viewed at: https://www.youtube.com/watch?v=WaB-dsB1Kfk, https://www.youtube.com/watch?v=Tp2Rdx69SSo, https://www.youtube.com/watch?v=Zo6dqO4VOb4 https://www.youtube.com/watch?v=IsrKWeIulXo (all accessed 9/8/16).

2. A comprehensive reference on flow of polymers is: *Principles of Polymer Processing,* Z. Tadmor and C.G. Gogos, Wiley Intersicence, Hoboken, 2006. Ch. 3 covers the Power Law model, Ch. 4 the basics of extrusion, Ch. 6 details on pumping, and Chs. 9-10 detailed mechanical design. A more specialized text is: *Analyzing and Troubleshooting Single Screw Extruders*, G. Campbell and M.A. Spalding, Carl Hanser, Munich, 2013. Ch. 1 covers the basics, Ch. 3 and Appendix A3 the rheology.

3. Almost all basic polymer science books cover this topic. Many basic fluid mechanics books in Chemical Engineering do also. Examples include: *Transport Phenomena* by R.B. Bird, W.E. Stewart, and E.N. Lightfoot, John Wiley, New York, 1960 (Ch. 2-3) and *Process Fluid Mechanics* by M.M. Denn, Prentice-Hall, Englewood Cliffs, 1980 (Ch. 2, 8, 19)

4. Melt temperatures and densities for almost all commercial extrudable resins by trade name can be found at: http://www.matweb.com/index.asp?ckck=1, and by chemical name at http://www.polymerprocessing.com/polymers/alpha.html, accessed 9/8/16.

5. Physical explanation of the backmixing: https://www.youtube.com/watch?v=IsrKWeIulXo (accessed 9/8/16).

6. A good discussion of the concept of propagation of errors in experimental work is by V. Lindberg: http://www.rit.edu/~w-uphysi/uncertainties/Uncertaintiespart2.html, or at http://www.itl.nist.gov/div898/handbook/mpc/section5/mpc55.htm, both accessed 9/23/16.

7. http://web.mit.edu/nnf/research/phenomena/Demos.pdf and http://web.mit.edu/nnf/ (short discussions of die swell with pictures and video), both accessed 9/8/16.

8. https://www.youtube.com/watch?v=UIAk_dlkUPE (pipes)

9. http://www.clextral.com/technologies-and-lines/technologies-et-procedes/twin-screw-extrusion-technology/

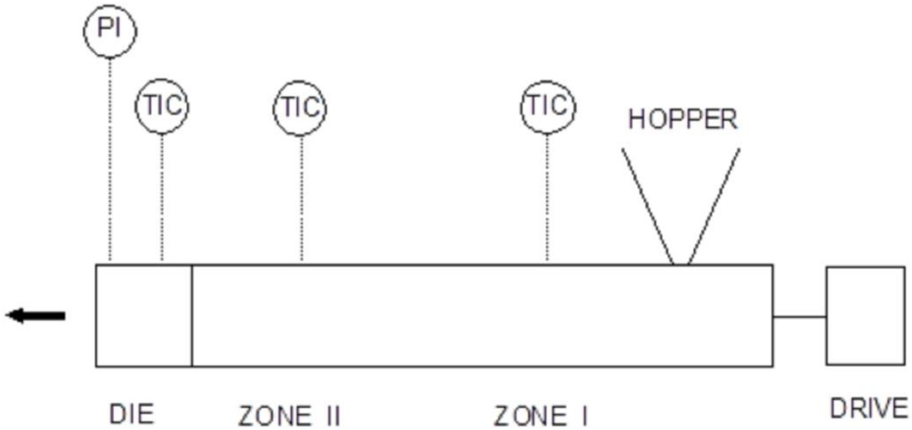


Fig. 1 Schematic of the extruder assembly

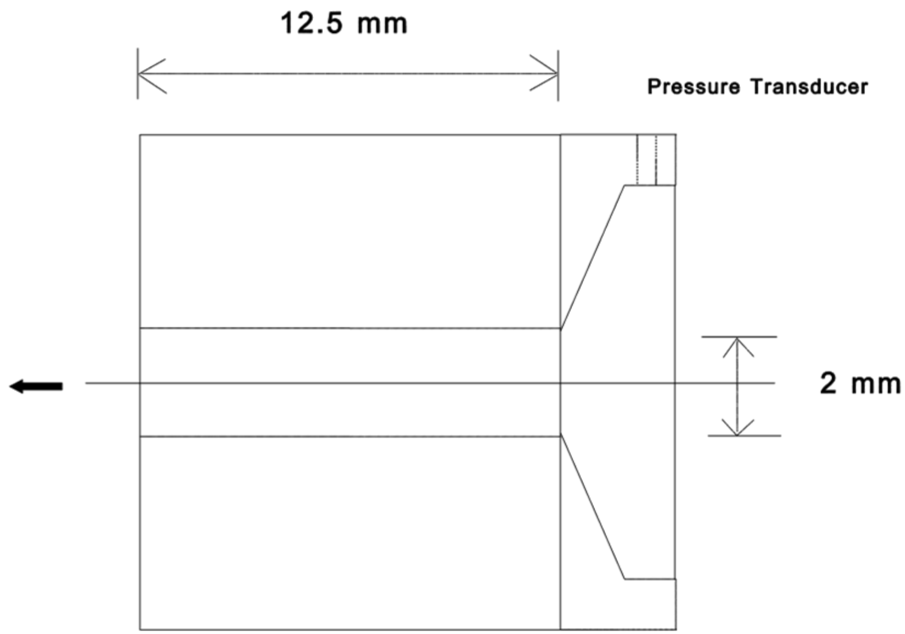
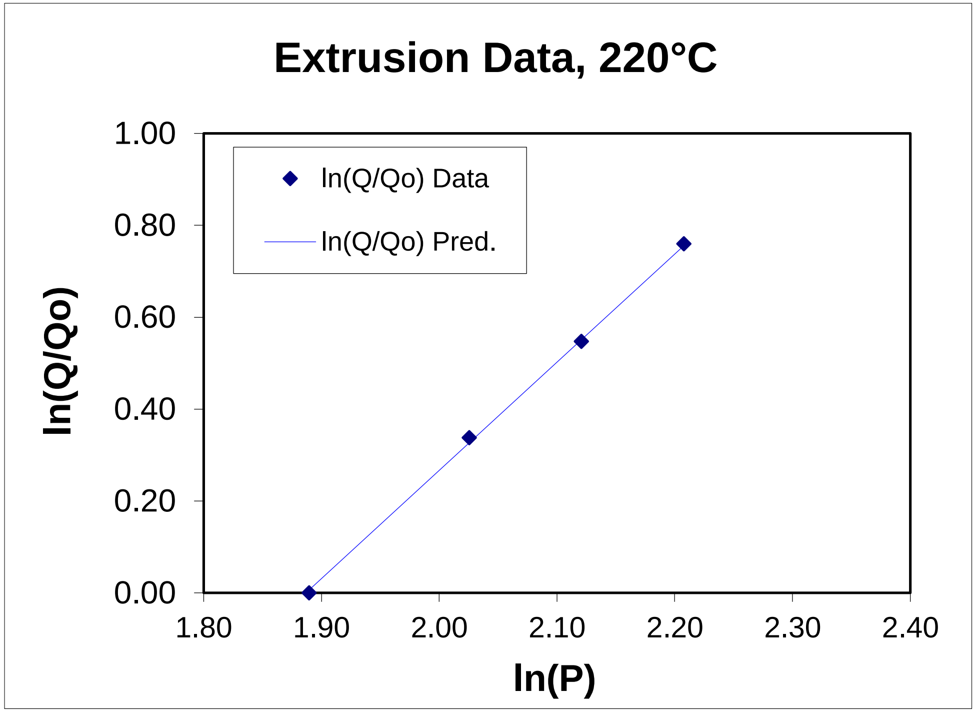
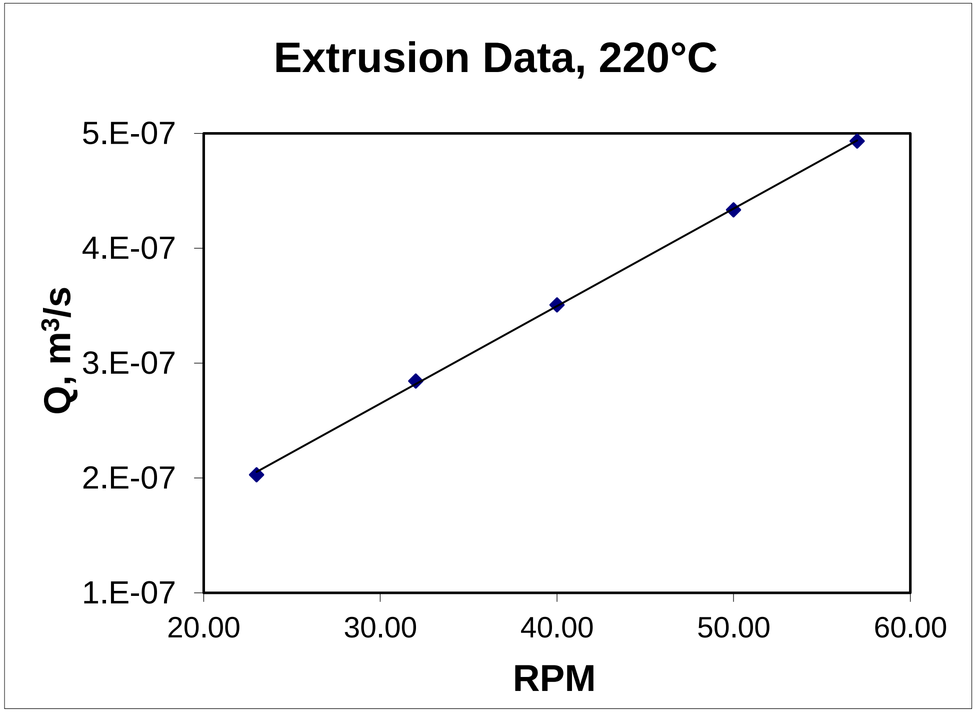


Fig 2. Schematic of the die


Extrusion Data, 220°C
ln(Q/Qo)
1.00
0.80
0.60
0.40
0.20
0.00
ln(Q/Qo) Data
ln(Q/Qo) Pred.
1.80
1.90
2.00
2.10
2.20
2.30
2.40
ln(P)


Extrusion Data, 220°C
5.E-07
4.E-07
3.E-07
2.E-07
1.E-07
Q, m3/s
20.00
30.00
40.00
50.00
60.00
RPM