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**Edited by D. R. Heldman, Michigan State University**

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# A REVIEW OF FUNDAMENTAL AND ENGINEERING ASPECTS OF EXTRUSION OF BIOPOLYMERS IN A SINGLE-SCREW EXTRUDER

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## ABSTRACT

*A review of fundamental and engineering aspects of extrusion in a single-screw is given. Attention is focussed on flow patterns, flow rates, residence time distributions and power consumption as a function of extruder design and screw rotation speed. The relationship for these engineering variables in extrusion of a purely Newtonian liquid and a power law liquid are reviewed. Where possible these relationships are compared with experimental results from the literature (corn-grits) and with our own measurements on biopolymers (corn-grits, defatted soy flour, modified di-amylopectin).*

## INTRODUCTION

Single screw extruders were first used for extrusion of thermoplastic materials in the early 1930's. Their use in food processing dates back to 1935 when a single-screw extruder was first applied for the continuous extrusion of pasta products (Rossen *et al.* 1973). At present extrusion is applied in a wide variety of processes: preparation of snacks, cereals, pasta foods, confectionery products, pet foods, animal feed, sausage products, protein supplements, meat analogs, beverage powders, breadings etc. (Rossen *et al.* 1973; Harper and Harmann 1973)

During extrusion of a food material several processes can, in principle, take place. The material is subjected to considerable shear forces and is mixed. Heat generated by viscous dissipation of mechanical energy and/or heat added leads to gelatinization of starches, denaturation of proteins, cooking or even sterilization. At the die, the material is formed while the sudden release in pressure at the exit of the die can lead to puffing or (flash) drying of the food material. The single-screw extruder, (Fig. 1a,b), is mainly applied for the production of snacks, cereals, pastas, confectionery, pet foods and animal feed, protein supplements and meat

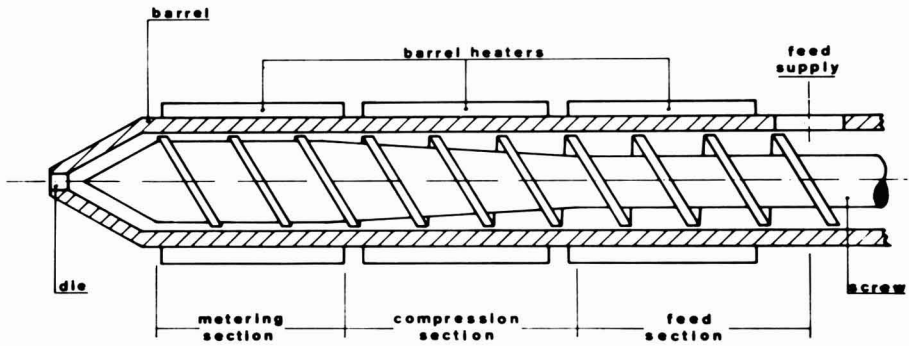


FIG., 1a. SIMPLIFIED EXTRUDER MODEL

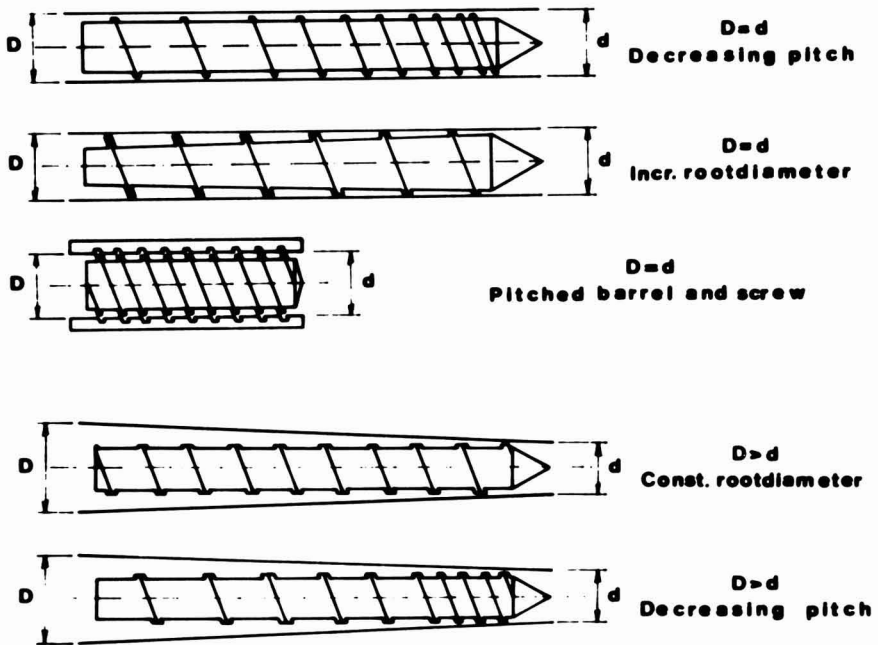


FIG. 1b. A SURVEY OF SINGLE SCREW EXTRUDER DESIGNS

analogs. The food material is fed to the apparatus from a hopper. The material is then picked up by the flights which convey it down the extruder channel and begin to work the material into a plasticized mass or dough. Via a compression section (transition section), where the root diameter of the screw usually increases somewhat, the material finally

enters the metering section where the diameter of the screw remains constant. The die is located at the end of the metering section. Temperatures in the compression-metering sections can be up to  $\approx 180$  °C and pressures up to  $2 \times 10^7$  Pa are no exception.

The purpose of this paper is to discuss some engineering aspects such as flow patterns, residence time distributions, power consumption and pressure drop- temperatures build-up in single-screw extruders. We will refer, at times, more specifically to a single-screw extruder originating from the Battenfeld design that is used in our laboratory research on extrusion of biopolymers like straight starch (corn-grits), fractionated potato starch derivatives (di-amylopectin phosphate, "Extrusamil" from AVEBE) and proteins (soy flour). Details of our work on extrusion of corn-grits and di-amylopectin phosphate have been given elsewhere (van Zuilichem *et al.* 1975; van Zuilichem 1976) while some preliminary results on the extrusion of soy-flours have been reported (van Zuilichem *et al.* 1977).

## EQUIPMENT

The extruder used is shown in Fig. 2. The root diameter of the screw increases from a diameter  $D_F$  ( $= 38.3$  mm) in the feed region to a constant diameter  $D_E$  in the metering section of the screw. The bore of the profiled barrel ( $D$ ) has a constant diameter of 48.4 mm. The length of the barrel is 610 mm. The compression ratio (C.R.) is defined as the ratio of the clearances  $h_1$  and  $h_2$  between barrel and screw root as shown in Fig. 2 and can be written as  $CR = h_1/h_2 = 4.85/h_2$  with  $h_2$  in mm (29). We have available screws with different pitches (24, 32, 45 mm) and compression ratios (C.R. 1.15, 1.4, 1.7, 2.0 3.0). The experiments were carried out with screws of constant pitch of 45 mm and compression ratios of C.R. = 1.15, 1.7, 2.0 and 3.0. Dies with diameters ( $d$ ) of 1.6, 2.4, 2.7 mm length ( $l$ ) of 1.9, 2.8, 3.2 mm were used in our work on corn grits. Extrusion of di-amylopectin phosphate was done through dies of 3, 4 and 6 mm diameter and length 5.0, 7.0 and 10 mm. Soy flour was extruded through dies with diameters 6, 8 and 10 mm and length 18, 24, 30 mm.

Rotation speed of the screw was varied from  $N = 0.8$  s<sup>-1</sup> to  $N = 2.33$  s<sup>-1</sup>. Mostly a rotational range between  $N = 1.0$  and  $N = 1.66$  was used. A meter based on the torsion bar principle with strain-gauge response is mounted to measure the torque needed for rotation (van Zuilichem *et al.* 1976).

Temperature profiles were measured at the barrel wall and inside the screw by thermocouples. Kistler 601 H piezo-quartz crystal pressure

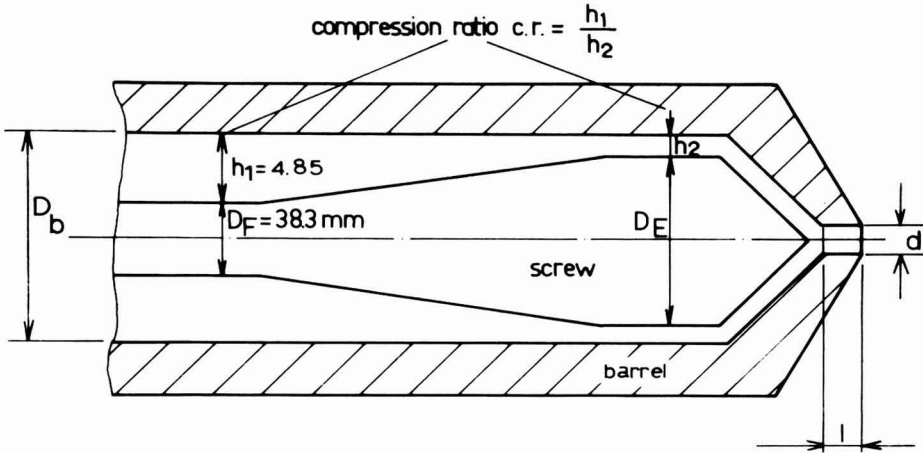


FIG. 2. SCHEMATIC CROSS SECTION OF SCREW AND BARREL. DEFINITION OF COMPRESSION RATIO.

probes were used to measure pressures at the same measuring points along the barrel. Pressure and torque responses are written with a Honeywell 1858 oscillographic recording system. Figure 3 gives details of the mounted pressure and temperature probes. Also in the extruder die-head temperatures and pressures were measured (Fig. 4); for details (van Zuilichem *et al.* 1975).

## FLOW PATTERNS AND TEMPERATURES PROFILES

### Theory

The main reasons why we are interested in the flow patterns inside an extruder are that it provides insight into mechanisms of mixing, residence time distribution, prediction of flow rates, pressure drop and power consumption. Figure 5 gives a definition sketch of the flow-channel in a single-screw extruder.

The basic problem in description of flow patterns inside the extruder is that the flow in the compression and metering section is non-Newtonian and not isothermal. Therefore the equation of motion as well as the equation of energy should be solved in order to obtain exact flow patterns. The general procedure to be followed is indicated in Table 1. Solutions to the set of equations can only be obtained if sweeping approximations are made. The most important are the assumption of steady state, neglect of inertia and gravity forces and the assumption of fully developed incompressible lubrication type of flow (independent of the  $x$ - and  $z$ - coordinates (Fig. 5). With these assumptions the flow in the extruder channel

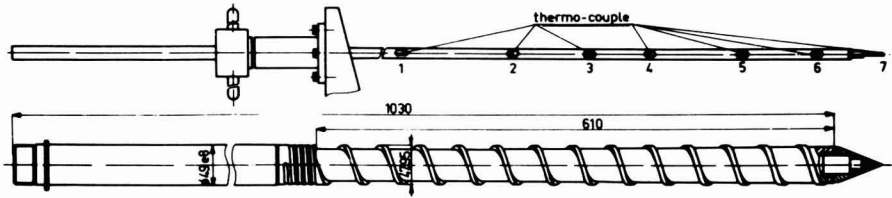


fig 3a Lance and screw

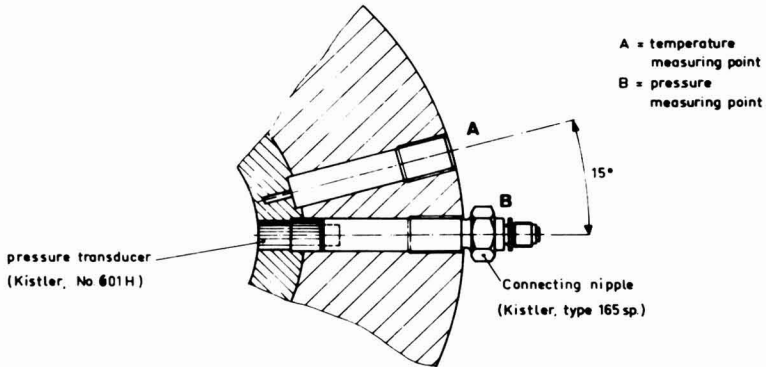


FIG. 3. DETAILS OF TEMPERATURE- AND PRESSURE MEASURING POINTS IN EXTRUDER HOUSING

is reduced to the flow in a slit of height  $h$  and width  $w$ , with one wall moving with velocity  $\pi N D \cos \theta$  and against a pressure gradient  $\partial p / \partial z$ . The flow is considered with respect to a rectangular coordinate system  $x, y, z$ , (Fig. 5) that rotates with the angular velocity of the axle. The barrel has a velocity,  $U$ , with respect to this coordinate system with components:  $U_x = \pi N D \sin \theta$ ,  $U_y = 0$ ,  $U_z = \pi N D \cos \theta$ . The flow in  $z$ -direction is the result of two driving forces: the drag caused by the  $z$ -component of the relative velocity of the barrel,  $U_z$ , and the pressure gradient in  $z$ -direction. With these assumptions and simplifications the solution of the equation of motion together with the equation of energy is feasible.

The most simple case arises when the material has a Newtonian flow behavior with temperature-independent viscosity and when we neglect the velocity components in  $y$ -direction near the flights. The latter assumption is a reasonable approximation when  $(h/w < 0.1)$ . Table 2 illustrates how the velocity profiles  $v_x$  and  $v_z$  can be calculated, and how temperature profiles could be derived when the heat flux at the barrel surface and the screw root would be given. Such approximate velocity profiles were derived about 25 years ago (Carley and Strub 1953; Carley *et al.* 1953) specifically for extruders and are also derived in the general literature on hydrodynamics (Schlichting 1968).

It is interesting to note that the equation of energy is fairly difficult to solve even for this extremely simplified rheological behavior because of

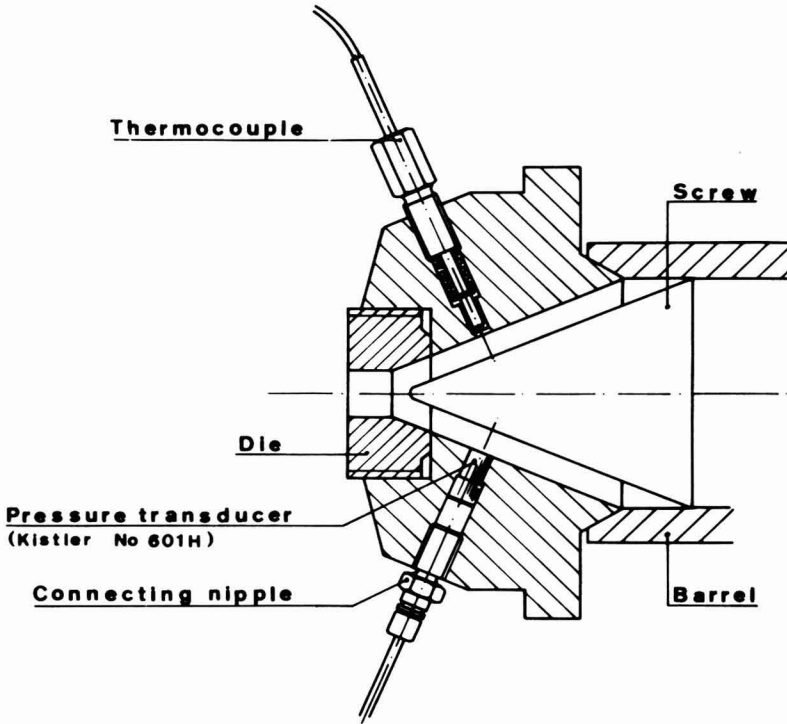


FIG. 4. TEMPERATURE- AND PRESSURE PROBES IN DIE-HARD ASSEMBLY

the convection term. Only when adiabatic extrusion is assumed and temperature gradients in  $y$ -direction are assumed to be negligible is a relatively simple result obtained.

The flow rate of the material can be derived from the velocity profile  $v_z$  by integration over the cross-section of the flow channel and multiplication of this flow with the number of flights,  $k$ , of the screw:

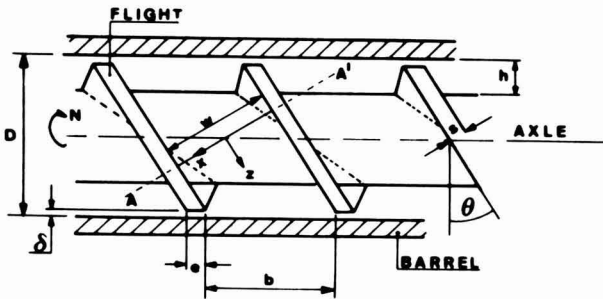
$$\Phi_v = k \int_0^w \int_0^h v_z \, dx \, dy \quad (1)$$

Using the velocity profile from Table 2 we obtain:

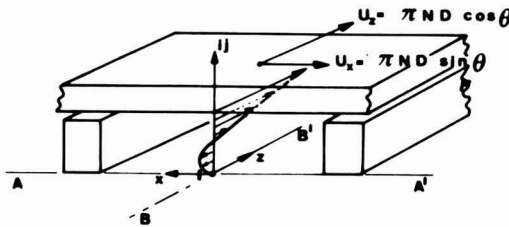
$$\begin{aligned} \Phi_v &= \frac{1}{2} h k w U_z \left(1 - \frac{1}{3} P\right) = \\ &= \frac{1}{2} h k w U_z - \frac{1}{12} \frac{h^3 k w}{\mu} \left(\frac{\partial p}{\partial z}\right) \end{aligned} \quad (2)$$

where P is defined as:

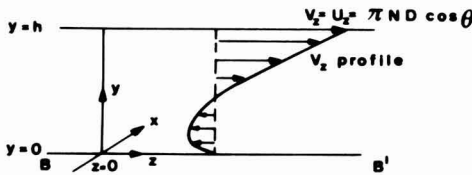
$$P = \frac{h^2}{2 \mu U_z} \left(\frac{\partial p}{\partial z}\right) \quad (3)$$



1. SECTION OF EXTRUDER WITH DEFINITION OF GEOMETRY



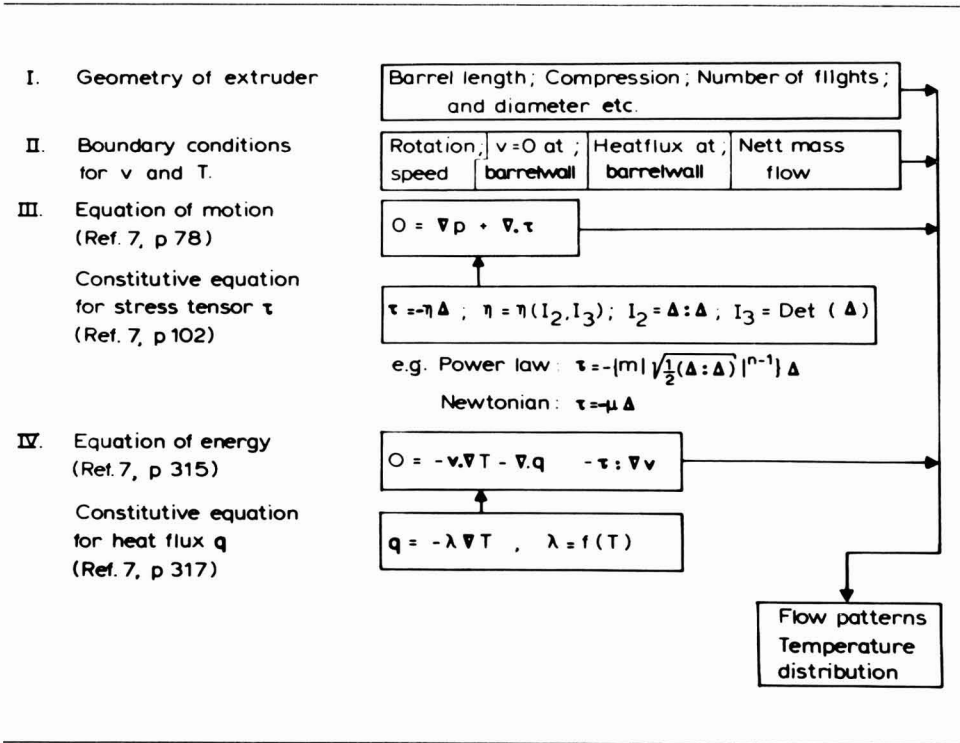
2. SIMPLIFIED FLOW GEOMETRY (SECTION AA')



3. FLOW PROFILE IN COORDINATE SYSTEM USED (SECT. BB')

FIG. 5. DEFINITION SKETCH FOR SINGLE-SCREW EXTRUDER

Table 1. Schematic approach for calculation of flow patterns in an extruder



In equation (2) the leakage flow through the clearance between the barrel and flight faces has been neglected. Griffith (1960) derived a relation between  $\Phi_v$  and P for a Newtonian liquid with temperature dependent viscosity:

$$\mu = \mu_{av} \exp [-b(2 T - T_r - T_b) / 2] \tag{4}$$

where  $T_r$  and  $T_b$  are root and barrel temperatures, b, a coefficient of the temperature dependence of  $\mu$  and  $\mu_{av}$ , the viscosity at temperature  $(T_b + T_r)/2$ . The equation of energy from Table 2 can be written in dimensionless form (we assume  $T_b = T_r$ ):

$$0 = \text{Pé} v_z^* \frac{\partial T^*}{\partial \xi} + \frac{\partial^2 T^*}{\partial \xi^2} + \text{Br} e^{-T^*} \left\{ \left( \frac{\partial v_z^*}{\partial \xi} \right)^2 + \left( \frac{\partial v_x^*}{\partial \xi} \right)^2 \right\} \tag{5}$$



where  $Pé'$  and  $Br$  are the dimensionless Péclet and Brinkman numbers:

$$Pé' = \frac{\rho C_p \pi N D h^2 \sin \theta}{\lambda L} \tag{6}$$

$$Br = \frac{\mu_{av} b \pi^2 N^2 D^2}{\lambda} \tag{7}$$

and  $v_z^*$ ,  $v_x^*$ ,  $T^*$ ,  $\xi$  and  $\xi$  are dimensionless variables defined by:

$$\left. \begin{aligned} v_z^* &= \frac{v_z}{\pi N D} ; v_x^* = \frac{v_x}{\pi N D} \\ T^* &= b (2 T - T_r - T_b) / 2 \\ \xi &= \frac{y}{h}, \xi = \frac{Z}{L} \sin \theta \end{aligned} \right\} \tag{8}$$

When representative values for corn-grits are substituted into equation (6) and (7) we find  $Pé' \simeq 6$  and  $Br \simeq 2$ . This is an indication that in solving equation (5) it is not quite reasonable to neglect the convective heat transport in z-direction when considering extrusion of biopolymers. Griffith (1960) neglected the convective term in equation (5) when solving it in combination with an equation of motion. Pearson (1976) also indicates that the circumstances under which the temperature profiles would be independent on x and z (fully developed) seldom arise.

When the leakage flow between flight faces and barrel wall is taken into account, a correction term on equation (2) is needed (Bernhardt 1959).

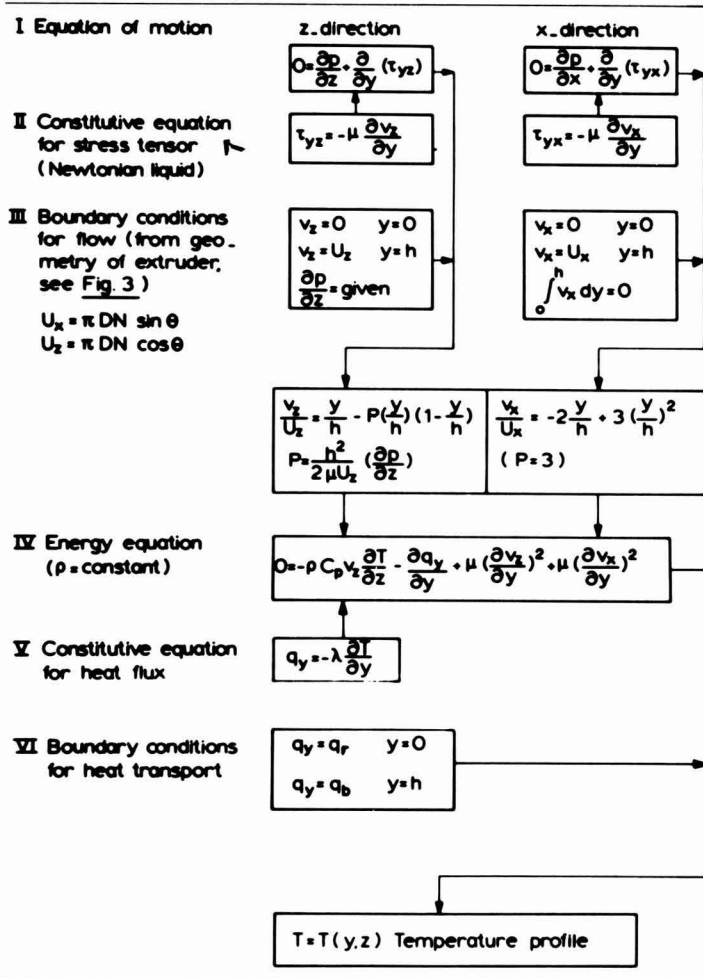
$$\Phi_v = \frac{1}{2} h k w U_z - \frac{1}{12} \frac{h^3 k w}{\mu} \left( \frac{\partial p}{\partial z} \right) - \frac{k w U_x \delta}{2 \tan \theta} \tag{9}$$

The flow  $\Phi_v$  has to pass through the die. If we assume the die to be a circular channel with diameter d and length  $l$  we have:

$$\Phi_v = \frac{\pi d^4 \Delta p}{128 \mu l} \tag{10}$$

$$\left( \frac{\Phi_v \sin \theta}{k w h N L} \right) = \frac{\pi}{2} \frac{\left(1 - \frac{\delta}{h}\right) \left(\frac{D}{L}\right) \sin \theta \cos \theta}{1 + \frac{32}{3\pi} \left(\frac{h}{d}\right)^3 \frac{l}{d} \frac{wk}{L} \sin \theta} \equiv \Lambda \quad (11)$$

Table 2. Calculation scheme for flow patterns in a single screw extruder for an incompressible Newtonian fluid with constant heat conductivity and viscosity.



The RHS is a constant for a given extruder geometry. We introduced a dimensionless flow rate by using the product of rotation speed  $N$ , ( $s^{-1}$ ) and the total channel volume of the extruder  $kwhL/\sin\theta(m^3)$  as a reference flow rate. Equation (11) implies that  $\Phi_v$  is proportional to

the rotation speed of the screw for a given extruder geometry. In practice most of the food materials to be extruded have non-Newtonian rheological behavior. Several authors have analyzed such situations. Pearson (1976) recently presented a summary on model studies for melt-fed extruders using a power law type expression for the stress tensor  $\tau$  (Bird *et al.* 1960):

$$\tau = - \eta \Delta \tag{12}$$

with the apparent viscosity,  $\eta$ , defined as:

$$\eta = m \left| \sqrt{1/2 (\Delta : \Delta)} \right|^{\eta-1} \tag{13}$$

For the x- and z-components of the equation of motion we need mainly the  $\tau_{yz}$  and  $\tau_{yx}$  elements of the stress tensor. From equations (12) and (13) we obtain, (Appendix 1)

$$\tau_{yz} = -m \left| \sqrt{\frac{\partial v_x^2}{\partial y} + \frac{\partial v_z^2}{\partial y}} \right|^{\eta-1} \frac{\partial v_z}{\partial y} \tag{14}$$

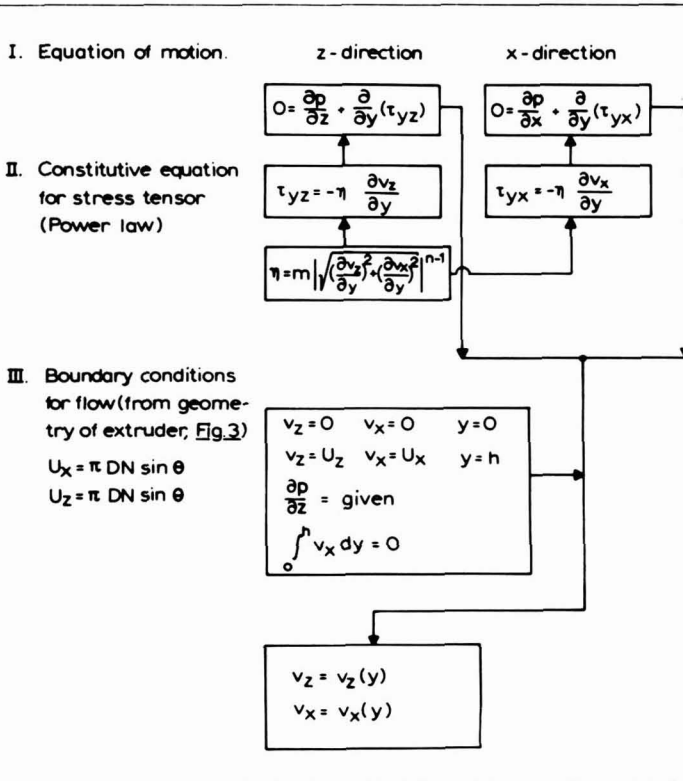
and

$$\tau_{yx} = -m \left| \sqrt{\frac{\partial v_x^2}{\partial y} + \frac{\partial v_z^2}{\partial y}} \right|^{\eta-1} \frac{\partial v_x}{\partial y} \tag{15}$$

We see from Table 3 that a considerable complication arises in solving the equation of motion and the equation of energy, because the z-component and the x-component of the equation of motion are coupled through equations (14) and (15). Jacobi (1960) derives equations for flow profiles in extruders for power law rheology in which  $(\partial v_x / \partial y)$  is set equal to zero in the equation for  $\tau_{yz}$ . This does not seem to be justified because  $v_x$  and  $v_z$  are of comparable order of magnitude for screws with angles  $\theta < 0.01$  radians which is usually the case. Bigg (1973) has developed a calculation scheme for the equation given in Table 3. Griffith (1962) gave a theory which relates  $\Phi_v$  to extruder geometry, screw speed and rheology for a power law fluid. He also included a temperature-dependent power law viscosity. On the same grounds as discussed before, we do not think the approximations made by Griffith are applicable for extrusion of biopolymers. His results for the isothermal case (i.e.  $Br = 0$  because  $b = 0$ ) are however, of great interest. Figure 6 gives his results, plotted as  $\Phi_v / (h k w U_v)$  against  $P_n$ .  $P_n$  is the logical extension of  $P$  to a power law fluid:

$$P_n = \frac{h^{n+1}}{2 m U_z^n} \left( \frac{\partial p}{\partial z} \right) \tag{16}$$

Table 3. Calculation of flow profiles for an incompressible non-Newtonian (power law) fluid in a single screw extruder.



Data on the rheological behavior of food materials under extrusion conditions (temperature, pressure) are extremely scarce in literature. Harper *et al.* (1967) and Harmann *et al.* (1974) report data on cooked cereal dough rheology measured with a straight tube viscosimeter and calculated from flow rate plus pressure drop across the die. These data, together with preliminary results obtained in our laboratory are presented in Table 4. In literature three methods to determine constants in the constitutive equation for the stress tensor are mentioned (Harper *et al.* 1967).

- (1) Measurement of pressure buildup in the extruder with closed die.
- (2) Measurement of flow plus pressure drop with various dies.
- (3) Measurement of flow plus pressure drop with capillary or slit viscosimeters mounted on the extruder tip.

In view of the difficulties in description of the exact flow patterns and maintaining a constant temperature in the extruder the first method is

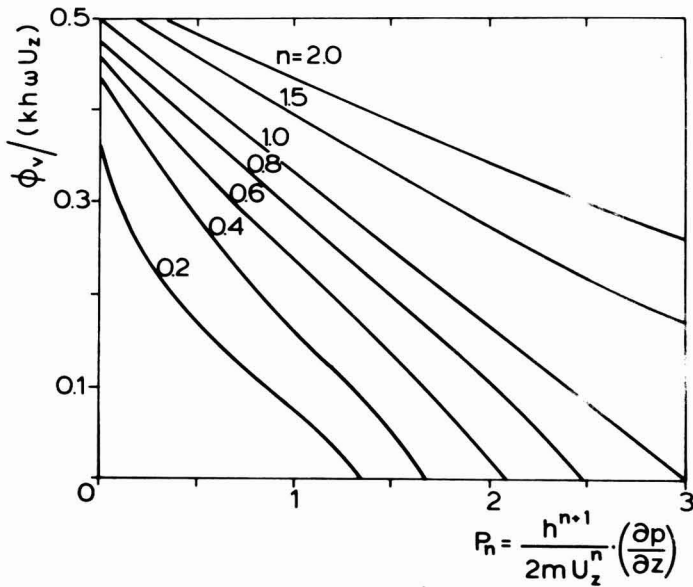


FIG. 6. FLOWRATE  $\phi_v$  FOR EXTRUSION OF POWER LAW FLUIDS AT CONSTANT TEMPERATURE  
(Griffith, Bigg)

virtually useless. The second method suffers from the requirement that the die usually has a short channel length so corrections for entrance and end effects are necessary. Only the use of capillary or slit viscosimeters can give accurate results at an accurately known temperature.

**Practice**

Conclusions from the previous section are that it is still impossible to describe flow patterns in single-screw extruders processing biopolymers. The main reason for this is the lack of data on rheological properties of the biopolymers under extrusion conditions. This rheology can be very complex. The constitutive equation for the stress tensor may need the introduction of normal stress coefficients and elasticity effects (relaxation times, retardation times). Also it is very important that chemical reactions occur during the extrusion process (e.g. gelatinization of starch or starch-derived materials, denaturation of proteins, Maillard reactions) which can strongly influence rheological properties of the biopolymer during flow through the extruder. Obviously, when this happens it is questionable whether realistic measurement of the rheological properties can be done.

An important problem also is that the theory given in section 2.1 only

Table 4. Data on rheological properties of food materials under extrusion conditions

Material	Properties			
	T (K)	p (N/m <sup>2</sup> )	ω <sub>w</sub> (kg/kg)	Rheological model η in (Ns/m <sup>2</sup> )
Cooked cereal dough (80% corn grits, 20% oat flour) Ref. 14	340- 373	-	0.25-0.30	<u>Power law (capillary tube)</u> $n = m \left  \frac{dv_z}{dr} \right ^{n-1} =$ $= m' \left  \frac{dv_z}{dr} \right ^{n-1} \frac{e^{-\frac{\alpha}{T}}}{e^{-\beta \omega_w}}$ $m' = 48.1 \quad (\text{Ns}^n/\text{m}^2)$ $n = 0.51 \quad (-)$ $\alpha = 2664. \quad (\text{K})$ $\beta = 7.62 \quad (\text{kg/kg})$
Pregelatinized corn flour ("Lora- dex" - Krause Milling) Ref. 15	360		0.32	<u>Power law (die-extruded)</u> $m = 1.72 \times 10^4 \quad (\text{Ns}^n/\text{m}^2)$ $n = 0.338 \quad (-)$
Corn grits Ref. 22				<u>Power law (slit viscosimeter)</u> $n = 0.45 - 0.55 \quad (-)$
				$m \quad (\text{Ns}^n/\text{m}^2)$
	450	$2.4 \times 10^7$	0.13	$2.8 \times 10^4$
	466	$1.5 \times 10^7$	0.13	$1.73 \times 10^4$
	480	$7 \times 10^6$	0.13	$0.76 \times 10^4$

pertains to the metering section and (part of) the compression section of the extruder. The food material is often fed to the extruder as a powder or a granulate which is transformed in the feed-section and (part of) the compression section into a plastic- or dough-like material. Study of this part of the extrusion process might be compared with plasticating extruders in polymer processing technology, where model studies are available (Pearson 1976; Lindt 1976; Tadmor 1966; Tadmor *et al.* 1967) for Newtonian melts and power law melts with temperature-dependent power law constant  $m$ .

Experimental results for flow rates of extruded biopolymers are available in literature, but only on a limited scale. Harmann and Harper

(1973) report flow rates as a function of screw speed and compression ratio in a  $\frac{3}{4}$ " Brabender extruder, extruding dry milled defatted corn grits with moisture contents varying from 11.6 to 14.6% by weight. The main conclusion from these experiments was that a linear increase of flow rate with increasing screw rotation speed was found. Measurements on corn grits were also performed in our laboratory with the extruder as described above. The maximum temperatures along the barrel were in the range of 175–210°, pressures varied from 14–36  $\times 10^6$  Pa.

In Fig. 7 such measurements (Lamers 1976) are compared with flow rates predicted by equation (11). The general trend is that theory predicts too high a flow rate and that deviations become larger at lower compression ratios of the extruder screw.

Figure 8 gives experimental flow rates of a modified di-amylopectin phosphate ("Extrusamil," AVEBE) on extrusion in our laboratory extruder. Temperatures ranged from 115–180°C, pressures from 7–27  $\times 10^6$  Pa, in the die-head and screw nose. Only data for one screw rotation speed:  $N = 1.33 \text{ s}^{-1}$  are given. Various dies were used ( $d = 3, 4, 6 \text{ mm}$ , with die lengths  $l = 5, 7, 10 \text{ mm}$ ), the moisture content was varied ( $\omega_w = 0.14, 0.15, 0.16 \text{ kg/kg}$ ) and screws with compression ratios of 1.15, 1.7 and 2.0 were applied. At a compression ratio of 1.15 the capacity is roughly independent of the die diameter. At a compression ratio of 2.0 a peculiar effect is found that the flow rate,  $\Phi_v$ , decreases with increasing diameter of the die. We have no satisfactory explanation for these measurements.

Figure 9 gives some results of extrusion of defatted soy flour, again in our laboratory extruder, as a function of screw rotation speed and for several dies ( $d = 6, 8$  and  $10 \text{ mm}$ ; with  $l/d = 3.0$ ). The soy flour used had a moisture content  $\omega_w = 0.08$ . The protein content ( $N \times 6.25$ ) is 52% weight, carbohydrate content 30% weight and a fat content  $< 1.5\%$  weight. The protein dispersibility index (PDI) is 70 using the standardized A.O.C.S. method. This flour thus is only slightly toasted and is extremely suitable for texturizing purposes. Experiments were done at moisture contents  $\omega_w = 0.19, 0.25$  and  $0.27 \text{ kg/kg}$ . The pressures measured are in the region of  $25 \times 10^5$  to  $70 \times 10^5 \text{ Pa}$ . The temperatures in the feed section had to be kept below 100°C by cooling and rises up to about 200°C in the metering section. We also measured the same dies with  $l/d = 7.0, 10.0$  and  $25.0$  and at moisture contents of  $\omega_w = 0.19$  and  $\omega_w = 0.27$ , but these data will not be reported here for brevity sake. The data indicate that despite the complex rheological behavior of soy flour under extrusion conditions the flow rate,  $\Phi_v$ , is proportional to the screw rotation speed as predicted by equation (11).

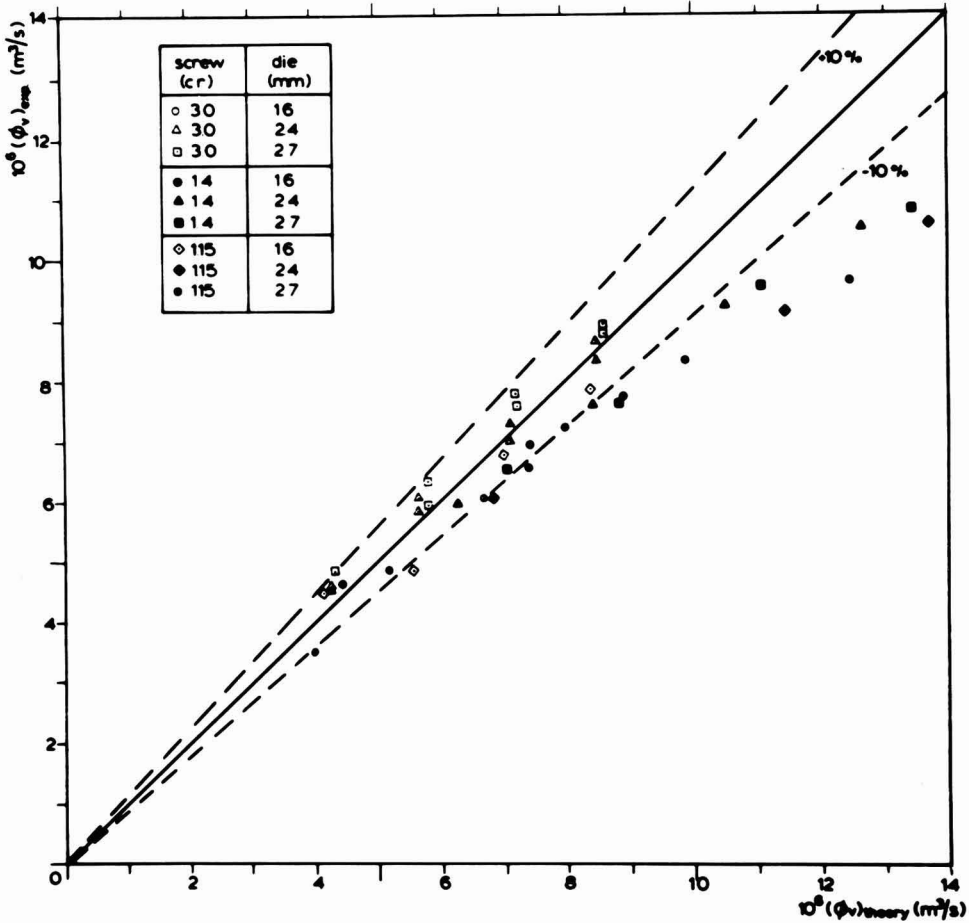


FIG. 7. COMPARISON OF CALCULATED AND EXPERIMENTAL FLOWRATES FOR CORN GRITS  
RESIDENCE TIME DISTRIBUTIONS

### Theory

One of the most important properties in the performance of an extruder is the residence time distribution (RTD) of the food material during its passage through the apparatus. The residence time distributions provide an "overall" effect of the velocity distributions inside the extruder channel. On the one hand we can calculate the RTD from known velocity profiles but, more important perhaps, we can measure the RTD by suitable experiments. From the RTD we can learn about the degree of mixing, life expectancy of the fluid elements and the degree of uniform-



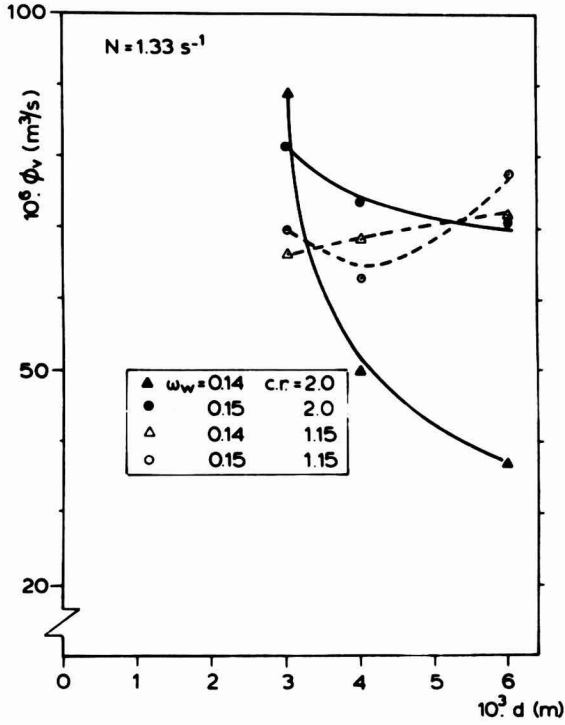


FIG. 8. FLOWRATE AS A FUNCTION OF DIE-DIAMETER FOR EXTRUSION OF DI-AMYLOPECTIN PHOSPHATE

ness of the strain exerted on the fluid elements during their life time in the extruder. The theory of residence time distribution was developed by chemical engineers and uses the F-diagram and the E-diagram to summarize the RTD. The F-diagram is the response of the system when a stepwise change in inlet concentration (e.g. a tracer) is made, the E-diagram is the response of the system to a pulse-like injection of a tracer at the inlet. For further details we refer to Danckwerts (1953) and Levenspiel (1972). Danckwerts (1953) defined the E(t)-function so that  $E(t) dt$  is the fraction, at the exit, of flow that has spent a time between  $t$  and  $(t + dt)$  in the system. The F(t)-function is obtained by integration to give a cumulative RTD function:

$$F(t) = \int_0^t E(t) dt \tag{17}$$

The most simple case for which the E(t)- and F(t)-function can be

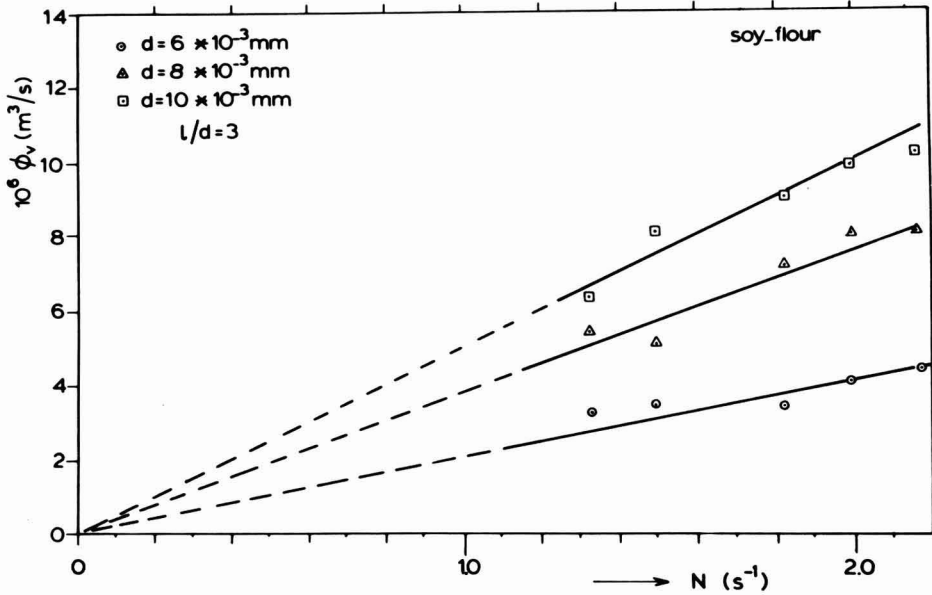


FIG. 9. EXPERIMENTAL FLOWRATES VERSUS SCREW-ROTATION SPEEDS FOR EXTRUSION OF DEFATTED SOY FLOUR

calculated is the flow of a Newtonian fluid with constant viscosity  $\mu$  through the extruder. For such a liquid we derived the velocity-profiles in section 2.1 (Table 2):

$$v_z = U_z \xi (1 - P + P \xi) \quad (18)$$

$$v_x = U_x \xi (2 - 3 \xi) \quad (19)$$

where we have introduced a dimensionless  $y$ -coordinate  $\xi \equiv y/h$ .

In Fig. 10 we give a sketch of these velocity profiles for  $P = 2$ . We see that due to the nature of the velocity profiles a particle will circulate in the  $(x,y)$  plane moving to the right when  $\frac{2}{3} < \xi < 1$  and to the left when  $0 < \xi < \frac{2}{3}$ . A particle located at  $\xi = \xi_1$  when moving to the left will always come back at  $\xi = \xi_r$  to move to the right. The relation between  $\xi_1$  and  $\xi_r$  follows from the fact that we assume no net flow to occur in  $x$ -direction:

$$\int_0^{\xi_1} v_x d\xi + \int_{\xi_r}^1 v_x d\xi = 0 \quad (20)$$

which leads to:

$$\xi_1 = \frac{1}{2} \{ 1 - \xi_r + \sqrt{1 + 2 \xi_r - 3 \xi_r^2} \} \tag{21}$$

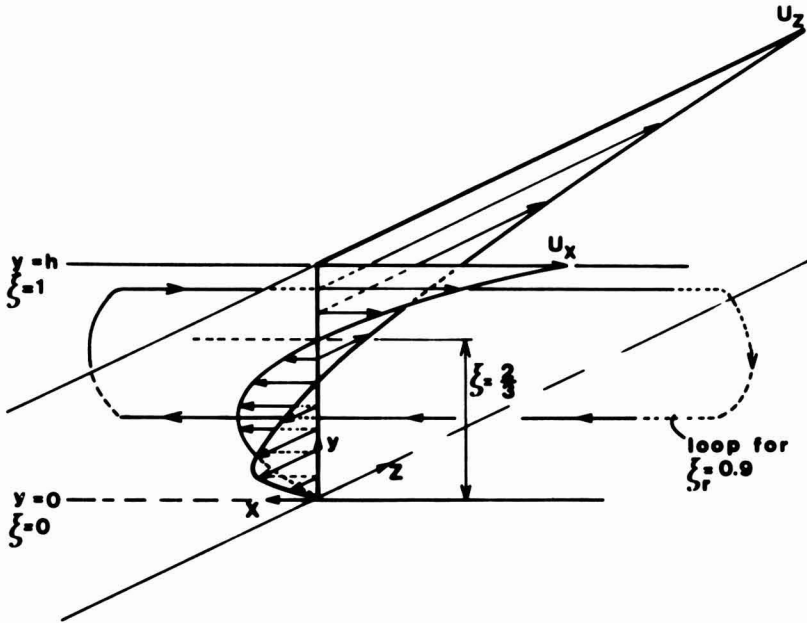


FIG. 10. TWO-DIMENSIONAL FLOW PROFILE FOR A CONSTANT VISCOSITY NEWTONIAN LIQUID (P-2)

In Fig. 10 we have indicated one such a closed path qualitatively for a particle at  $\xi_r = 0.9$ . The result is that the particles all flow down the channel in a helical path with a net positive component in z-direction. Pinto and Tadmor (1970) have analyzed the RTD's resulting from these velocity profiles. The E(t)-function then becomes:

$$E(t) = \frac{9}{2} \frac{\pi N D \sin \theta \cos \theta (1 - \frac{1}{3}P)}{L} \frac{\xi^3 \{ \xi - 1 - \sqrt{1 + 2 \xi - 3 \xi^2} \}^3}{(6 \xi^2 - 4 \xi - 1) \sqrt{1 + 2 \xi - 3 \xi^2 + 3 \xi - 1}} \tag{22}$$

where  $P = 3 \{ 1 - 2\Phi_v / (kwhU_z) \}$  and where  $\xi$  is related to  $t$  by:

$$t = \frac{L}{3 \pi N D \sin \theta \cos \theta \cdot (1 - \frac{1}{3}P)} \cdot \frac{3 \xi - 1 + 3 \quad 1 + 2 \xi - 3 \xi^2}{\xi \{ 1 - \xi + \quad 1 + 2 \xi - 3 \xi^2 \}} \quad (23)$$

The  $F(t)$  function follows by integration of the  $E(t)$ -function:

$$F(t) = \frac{1}{2} \{ 3 \xi^2 - 1 + (\xi - 1) \cdot (1 + 2 \xi - 3 \xi^2) \} \quad (24)$$

where  $\xi$  again is related to  $t$  by equation (23). From these equations the mean-residence time,  $\bar{t}$ , and the  $E$ -diagram and  $F$ -diagram can be calculated. For  $\bar{t}$  the result is:

$$\bar{t} = \int_0^{\infty} t E(t) dt = k h w l / (\Phi_v \sin \theta) \quad (25)$$

Comparing with equation (11) gives the useful result:

$$\bar{t} = (\Lambda N)^{-1} \quad (26)$$

so the average residence time is proportional to the reciprocal screw rotation speed  $N$ . In Fig. 11 and Fig. 12 we give the  $E$ - and  $F$ -diagrams plotted in the usual way. We see that an extruder behaves somewhere in between laminar flow through a pipe and plug flow.

An important element of extrusion is that the material is brought under the influence of shear forces. When flow profiles and RTD are known it is possible to calculate a measure of the amount of deformation the extruder is able to exert. Pinto and Tadmor (1970) defined a weighted average total strain (WATS)  $\bar{\gamma}$  as follows:

$$\bar{\gamma} = \int_0^{\infty} \dot{\gamma} E(t) dt \quad (27)$$

where  $\gamma$  is a shear strain calculated by combining the shearing the material experiences during its helical path down the extruder in the following way:

$$\gamma = (1 - f) \dot{\gamma}_t t + f \dot{\gamma}_r t \quad (28)$$

where  $f$  is the fraction of the total residence time,  $t$ , spent in that part of the channel where  $v_x$  is directed to the right (see Fig. 8.). The shear rates  $\dot{\gamma}_x$ ,  $\dot{\gamma}_z$  are calculated from:

$$\dot{\gamma}(\xi) = \sqrt{\left(\frac{\partial v_x}{\partial y}\right)^2 + \left(\frac{\partial v_z}{\partial y}\right)^2} \quad (29)$$

with  $\xi = \xi_1$  and  $\xi = \xi_r$  respectively, with  $\xi_1 = f(\xi_r)$  according to equation (21). From the velocity profiles (18) and (19) we find by differentiation:

$$\dot{\gamma}(\xi) = \frac{\pi N D}{h} \sqrt{\{ 4 (1 - 3 \xi)^2 \sin^2 \theta + (1 - P + 2 \xi) \cos^2 \theta \}} \quad (30)$$

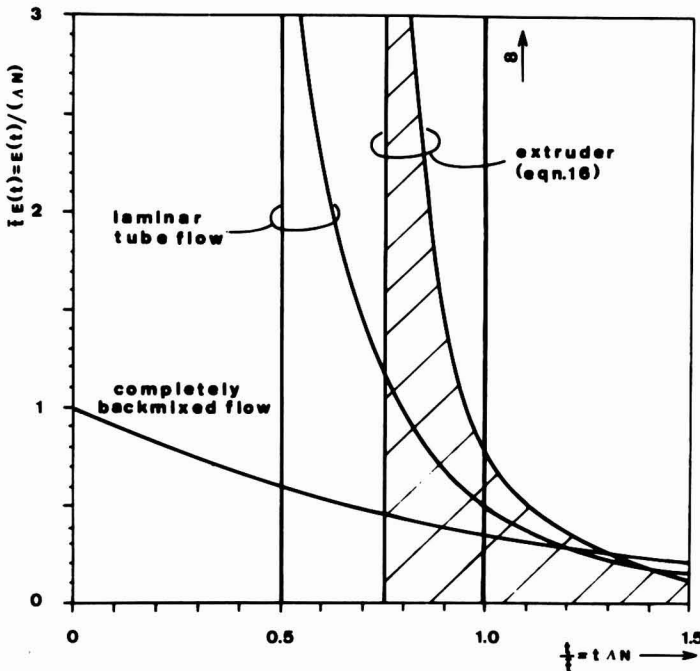


FIG. 11. E(t) DIAGRAM FOR FLOW OF A NEWTONIAN LIQUID THROUGH AN EXTRUDER

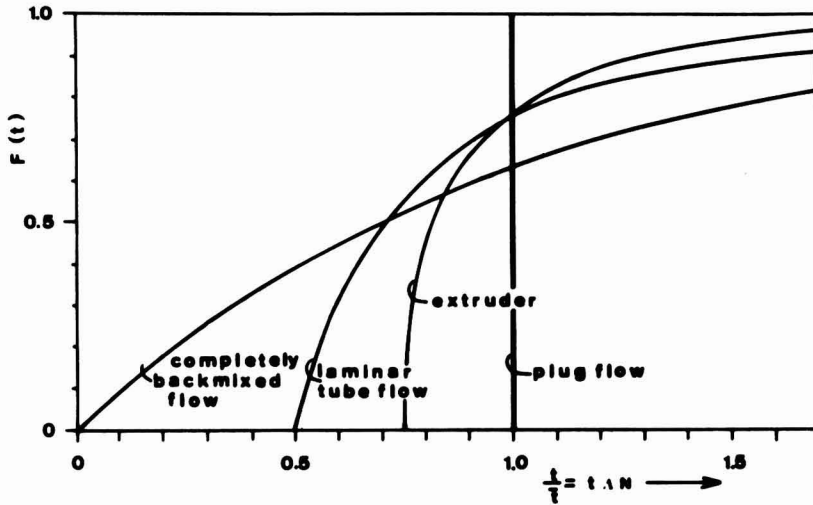


FIG. 12. F-DIAGRAM FOR FLOW OF A NEWTONIAN LIQUID THROUGH AN EXTRUDER

The WATS is directly proportional to  $(L/h)$ . Therefore a normalized WATS  $\bar{\gamma}^* \equiv \bar{\gamma} \cdot h/L$  is plotted in Fig. 13, against the two variables on which  $\bar{\gamma}$  only depends:  $P$  and  $\theta$ . From Fig. 13 one can conclude that the screw angle  $\theta$  has only a limited influence in the practical range ( $15^\circ < \theta < 35^\circ$ ).

The order of magnitude of  $\bar{\gamma}$  can be derived from a simple model in which one assumes that the shear rate during flow through the extruder is  $(U_z/h)$ . This shear rate is roughly exerted over a period of time equal to the average residence time  $\bar{t}$ . Thus we can make  $\bar{\gamma}$  dimensionless with a reference  $\bar{\gamma}_R$ :

$$\bar{\gamma}_R = \frac{\bar{t} U_z}{h} = \frac{\pi N D w L \cos \theta}{\Phi_v \sin \theta} \quad (31)$$

Defining  $\bar{\gamma}^{**}$  as  $\bar{\gamma}/\bar{\gamma}_R$  we can derive that:

$$\bar{\gamma}^{**} = \bar{\gamma}^* \frac{1}{2} \sin \theta \left(1 - \frac{1}{3} P\right) \quad (32)$$

In Fig. 14 this dimensionless WATS is plotted against  $P$ . Clearly  $\bar{\gamma}^{**}$  is of the order of magnitude of unity and is independent of  $\theta$ . The theory of residence time distributions and weighted average total strain have been extended to plasticating extruders by Lidor and Tadmor (1976).

The main engineering variables left thus are the  $(L/h)$  ratio and the back pressure parameter  $P$  which is influenced by both extruder- and the

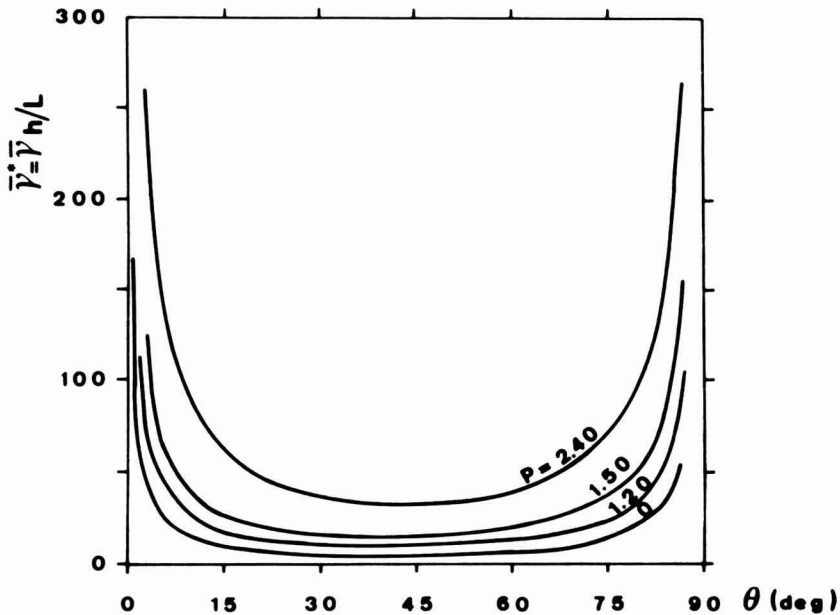


FIG. 13. WEIGHTED AVERAGE STRAIN IN AN EXTRUDER FOR A NEWTONIAN LIQUID (Pinto & Tadmor, 24)

die design. When we substitute eqn. (10) and (11) into the definition of P (equation 3) we obtain:

$$P = A (1 - \delta/h) / (1 + \frac{1}{3} A) \tag{33}$$

with

$$A = \frac{32}{\pi} \left( \frac{h}{d} \right) \left( \frac{l}{d} \right) \left( \frac{w}{L} \right) k \sin \theta \tag{34}$$

We see that for the case of a purely Newtonian fluid the parameter P is completely determined by extruder and die design and not by screw rotation speed.

Non-Newtonian liquids in a single screw extruder have been analyzed by Bigg and Middleman (1974). The basis of the calculated RTD and WATS were the solutions to the velocity profiles for a power law liquid as indicated in Table 3. In Fig. 15 a typical F(t)-diagram is given, while Fig. 16 gives a plot of  $\gamma^{**}$  against the parameter  $P_n$  defined by equation (16). An interesting observation is that the shape of the F(t)-curve can be

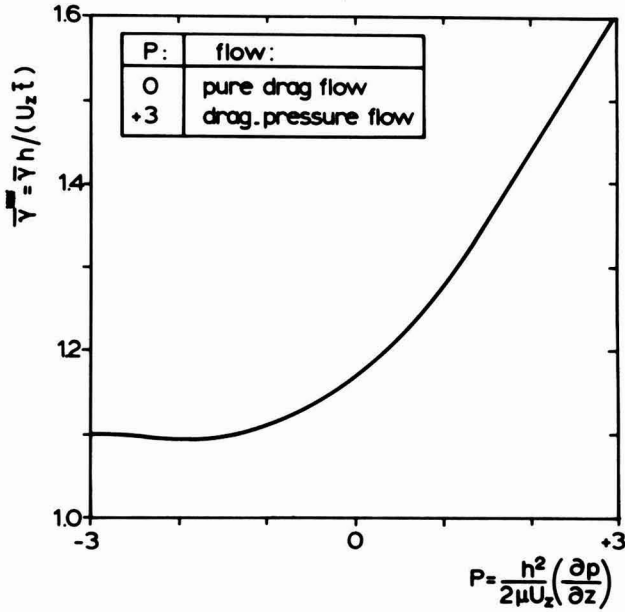


FIG. 14. WEIGHTED AVERAGE SHEAR STRAIN  $\bar{\gamma}^{**}$  IN AN EXTRUDER FOR A NEWTONIAN LIQUID  
(Curve calculated for  $\theta = 10^\circ$ ,  $\theta = 20^\circ$  and  $\theta = 30^\circ$ )

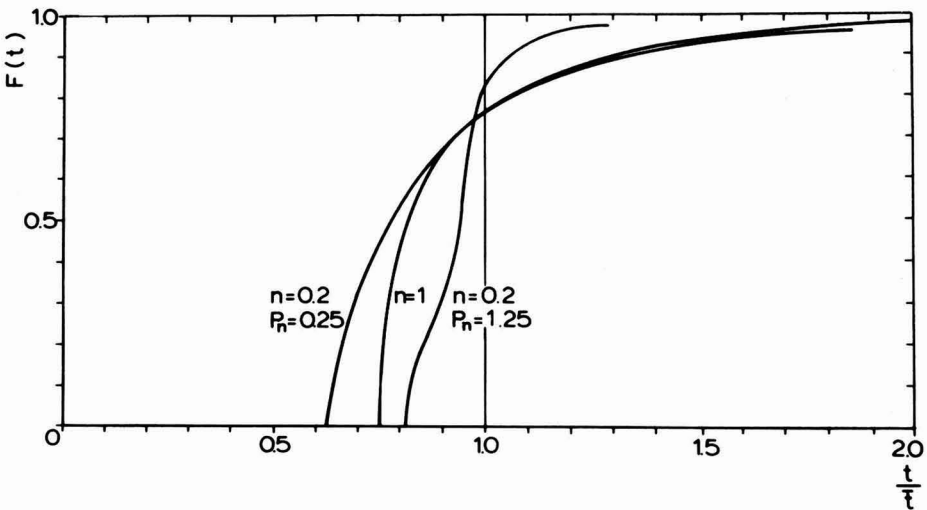


FIG. 15. TYPICAL  $F(t)$  DIAGRAMS FOR A POWER LAW FLUID WITH  $n=0.2$  AT DIFFERENT VALUES OF THE PARAMETER  $P_n$ .

significantly influenced by  $P_n$ . Increasing the back-pressure gives a residence time distribution that is more towards pure plug flow.



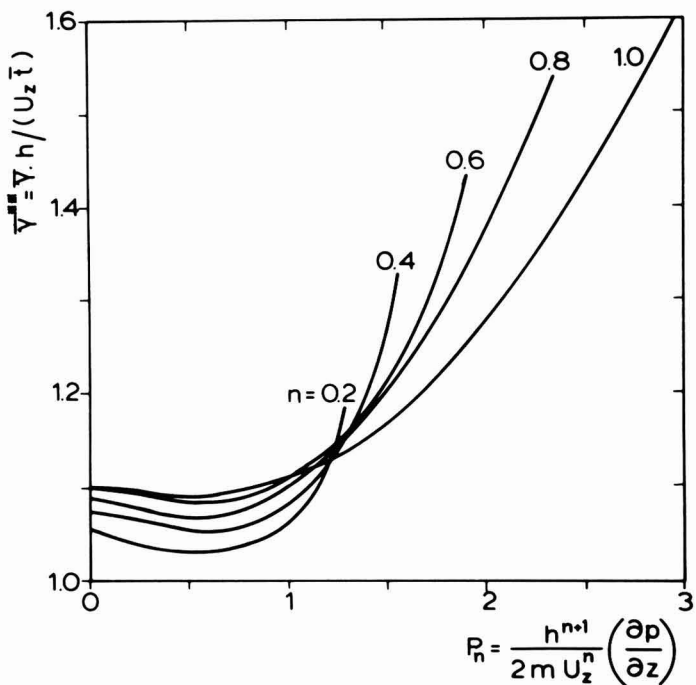


FIG. 16. WEIGHTED AVERAGE SHEAR STRAIN  $\bar{\gamma}^{**}$  IN AN EXTRUDER FOR A POWER LAW FLUID, WITH VARYING  $n$

**Practice**

Several studies have been directed towards experimental verification of the RTD's of single-screw extruders when processing Newtonian and non-Newtonian materials. Bigg and Middleman (1974) checked the F(t)-diagram for Newtonian liquids in a Brabender  $\frac{3}{4}$  " extruder with a screw of constant channel depth with corn syrups and Vorite 125, a polymerized castor oil (viscosities 2.92 and 177 Ns/m<sup>2</sup> respectively). Also a 1% polyacrylamide solution in water was used to check non-Newtonian behavior ( $n=0.34$ ) in the range of shear rates from 10 to 100 s<sup>-1</sup>. In all cases the agreement between measurements and theory was excellent. Wolf and White (1976) used a Prodex extruder (screw diameter  $44.2 \times 10^{-3}$  m) with a feed section, compression section and metering section and studied liquid conveying (a polyester resin), solids conveying (polyethylene) and plasticating extrusion (low density polyethylene). The F(t)-diagram as derived by Pinto and Tadmor (1970) provided a reasonable description of the liquid conveying and plasticating extrusion. In Fig. 17 some results are given.

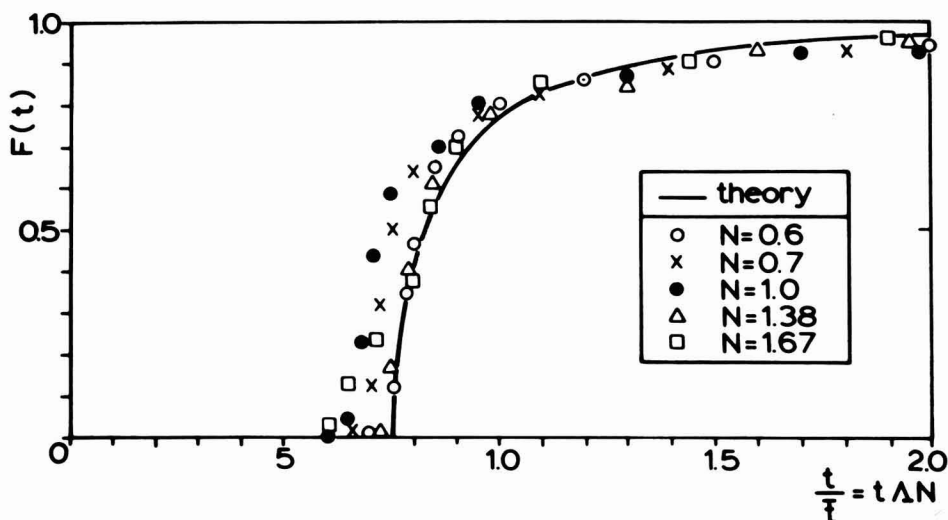


FIG. 17. COMPARISON OF EXPERIMENTAL AND THEORETICAL RTD FOR PLASTICATING EXTRUSION

(Woft & White, 27)

The only measurements of RTD's for extrusion of biopolymers that we are aware of are those by van Zuilichem *et al.* (1973) and Buisman (1972) in the extruder of Fig. 2. By a radio-tracer technique using  $^{64}\text{Cu}$  as the tracer directly the  $E(t)$ -diagram was measured. Figure 18 gives the experimental set-up; for details we refer to Buisman (1972) and van Zuilichem *et al.* (1973). The material extruded was corn-grits with moisture content between 14 and 20% weight. Experiments were carried out at three screw rotation rates ( $N = 1.33, 1.67$  and  $2\text{S}^{-1}$ ) and two die diameters. Figure 19 gives some typical  $F(t)$ -diagrams, for several screw rotation speeds and two moisture contents. There is some tendency for early break-through at higher rotation speeds when compared with the theory of Pinto and Tadmor. Also a higher moisture content tends to give a response that is closer to plug flow. It is not possible at this time to give an unambiguous explanation of these effects. It is likely that differences in temperature (and thus viscosity)-distribution and non-Newtonian flow effects may be the cause of these deviations.

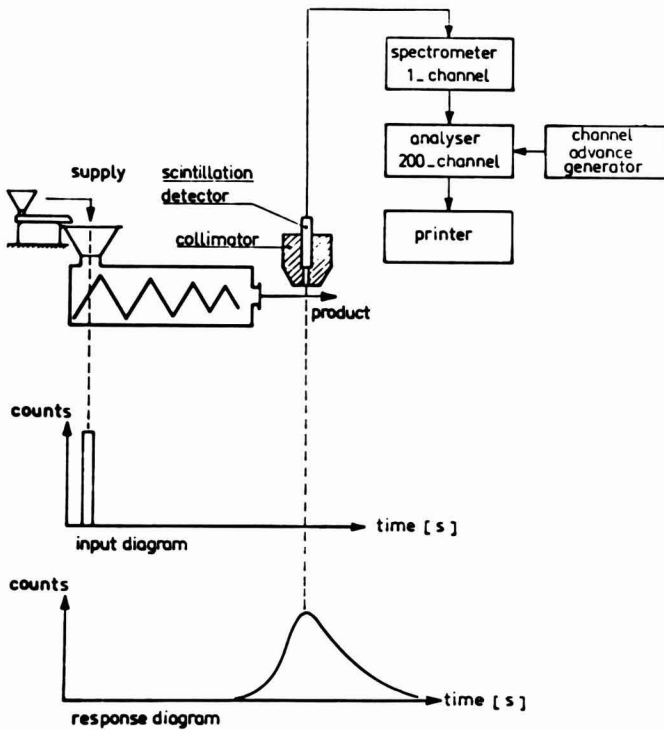


FIG. 18. BLOCK-DIAGRAM OF THE EXPERIMENTAL SET-UP

### POWER CONSUMPTION

#### Theory

The schematic approach to calculating power requirements for an extruder are given in Table 5. The macroscopic mechanical energy balance (Bird *et al.* 1960) is written for the total volume of the extruder. The rate at which the system performs mechanical work on the surroundings,  $W$ , is numerically equal to the shaft power that has to be delivered to the screw. We see that  $W$  can be calculated when the temperature-, pressure- and velocity distributions in the extruder are known by solving the local equation of motion and equation of energy.

The power requirements for extrusion of a Newtonian incompressible

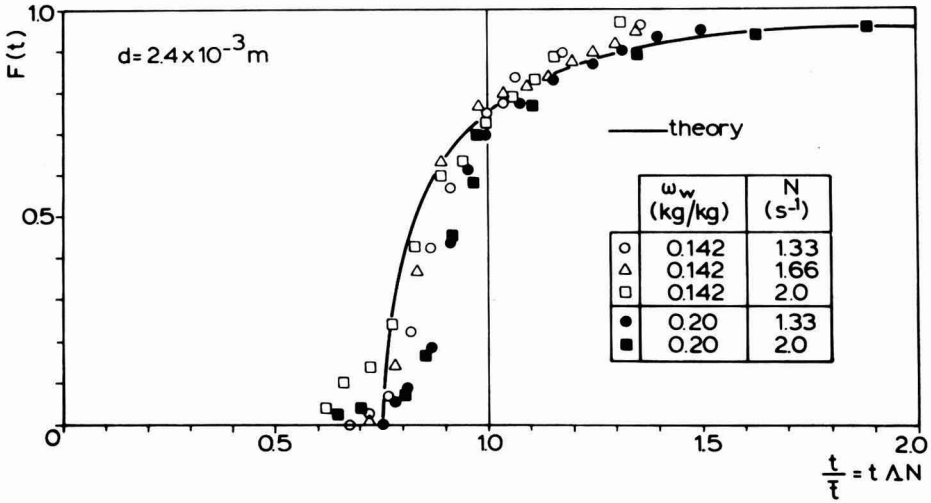
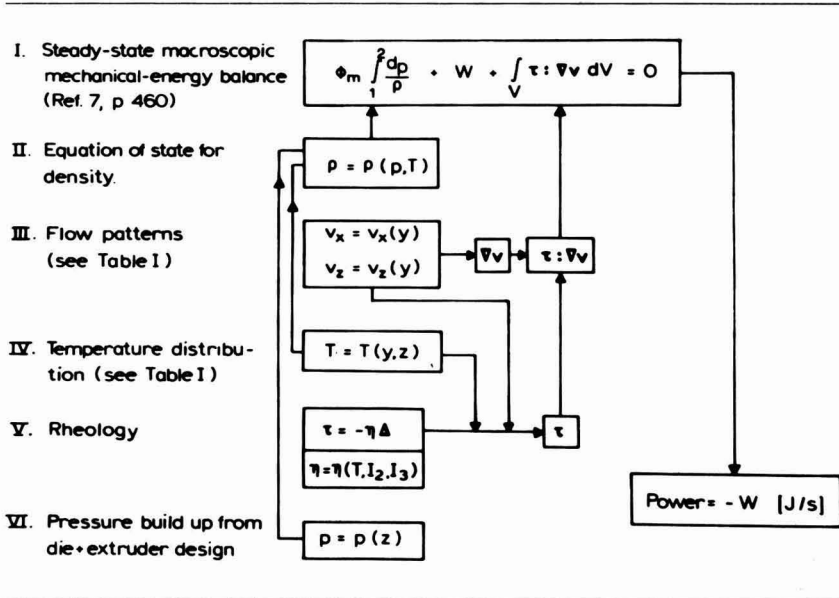


FIG. 19.  $F(t)$  CURVES FOR EXTRUSION OF CORN GRITS IN A SINGLE SCREW EXTRUDER

Table 5. Schematic approach for calculation of power requirements in a single screw extruder



liquid with temperature independent viscosity is fairly easy to calculate with the aid of the velocity profiles derived in section 2. The result is (leakage flow is neglected):

$$\frac{Po}{\mu k N^2 D^3} = \pi^2 \left(\frac{L}{D}\right) \left(\frac{w}{h}\right) \left(4 \tan^2 \theta + 1 + \frac{1}{3} P^2 + \frac{A}{3 \sin \theta}\right) \tag{35}$$

where A is defined by equation (34). The left hand side of equation (35) is the usual "power number" for mixing in the viscous flow region. The R.H.S. of equation (35) is a constant for a given extruder geometry and die design. Equation (35) must be refined for situations where channel curvature and channel wall-effects are significant (Carley 1962; Bernhardt 1959; Harmann and Harper 1973). When the clearance between flight faces and barrel wall is narrow a very considerable part of the power consumption takes place in this region. The power consumption is readily corrected for this dissipation (Bernhardt 1959; Mallouk and McKelvey 1953). The increase,  $\Delta Po$ , in power consumption is:

$$\Delta Po = \mu k N^2 D^3 \left\{ \pi^2 \frac{e}{\delta} \left(\frac{L}{D}\right) \cotan \theta \right\} \tag{36}$$

When the viscosity  $\mu$  is not constant but a function of temperature an empirical approach is to substitute the viscosities  $\mu_d$ ,  $\mu_c$  and  $\mu_\delta$  at the appropriate places in the power consumption equation giving:

$$\begin{aligned} \frac{Po}{\mu_c k N^2 D^3} &= \pi^2 \left(\frac{L}{D}\right) \left(\frac{w}{h}\right) \left\{ 4 \tan \theta + 1 + \frac{1}{3} P^2 + \frac{\mu_d}{\mu_c} \frac{A}{3 \sin \theta} \right. \\ &+ \left. \frac{\mu_\delta}{\mu_c} \left(\frac{e}{w}\right) \cdot \left(\frac{h}{\delta}\right) \cotan \theta \right\} \end{aligned} \tag{37}$$

For non-Newtonian materials the calculation scheme of Table 5 is still applicable. As far as we know the numerical solution of these equations has not been reported in the literature.

**Practice**

Power consumption (or torque) measurements have been reported in the literature on extrusion of corn-grits (van Zuilichem *et al.* 1975; Harmann and Harper 1973; Lamers 1976). General trends are that power requirements decrease with increasing temperature and increasing moisture content. Of course these effects are qualitatively in accordance with the fact that the power consumption is in the viscous flow region (i.e. we expect  $Po/(\mu_{app} N^2 D^3)$  to be a constant) and that  $\mu_{app}$  decreases with temperature and moisture content. Figure 20 gives results from our own

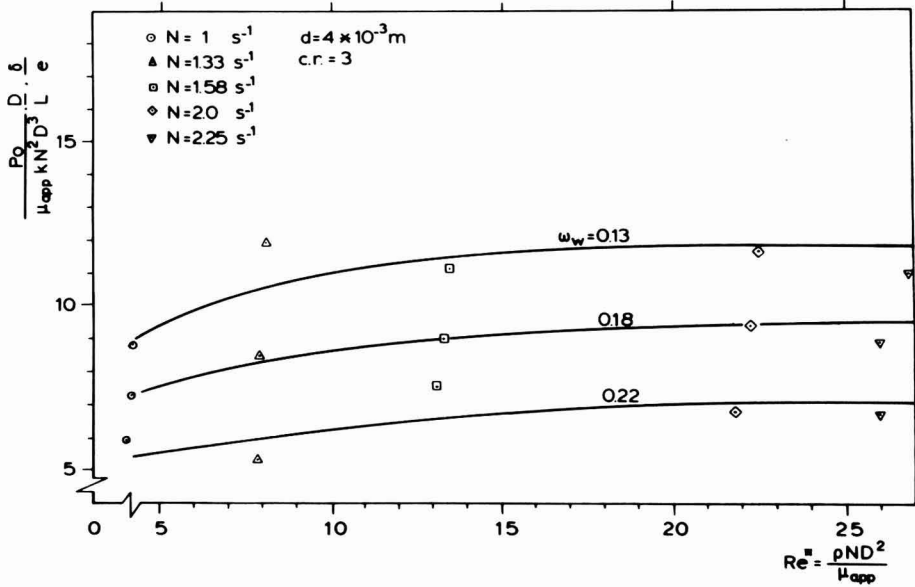


FIG. 20. POWER NUMBER AGAINST  $Re^*$  FOR EXTRUSION OF CORN-GRITS

measurements on corn-grits. We plot here the power number against a Reynolds number  $Re^*$  ( $\equiv \rho N D^2 / \mu_{app}$ ). The apparent viscosity  $\mu_{app}$  is taken as the viscosity of corn-grits at a moisture content of  $\omega_w = 0.13 \text{ kg/kg}$  and the shear rate and temperature at the screw tip pertaining to that situation. When more accurate data on the moisture content dependence and shear-rate dependence of  $\mu_{app}$  were available one would expect the curves to coincide and give on horizontal line.

Figure 21 gives power consumption data measured when extruding the modified di-amylopectin phosphate ("Extrusamil", AVEBE) mentioned before. Here we plot a specific power consumption (in  $\text{kJ/kg}$  extruded product) defined by  $Po / (\rho \Phi_v)$  against the moisture content of the extruded material for several compression ratios and dies and one screw rotation speed of  $1.33 \text{ s}^{-1}$ . We see that initially the specific power consumption for extrusion decreases with increasing moisture content but, especially for the 4 mm die, it increases again at higher moisture content. A tentative explanation for this may be that the gelatinization rate is strongly dependent on moisture content. Further research is needed to explain such behavior.

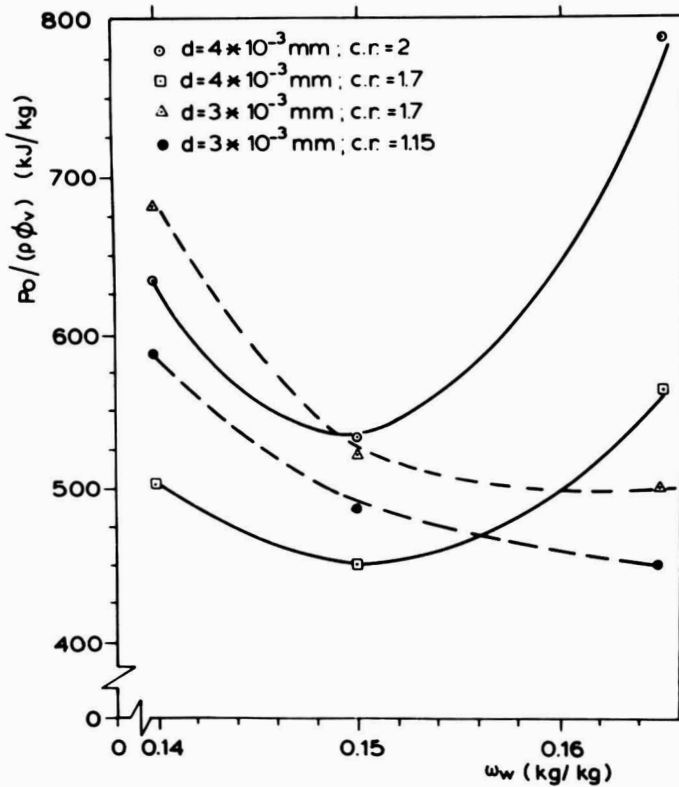


FIG. 21. POWER CONSUMPTION FOR EXTRUSION OF A MODIFIED DI-AMYLOPECTIN PHOSPHATE

Recent measurements on power consumption during extrusion of defatted soy-flour are given in Fig. 22. Various die diameters have been used ( $d = 6, 8$  and  $10$  mm) with channel lengths such that  $l/d = 3$ . The main conclusion is that the specific power consumption ( $= Po / (\rho \phi v)$ ) does not vary much with rotor speed, perhaps because of a strongly non-Newtonian rheology and temperature effects. For a Newtonian liquid at constant temperature, one would expect  $Po / (\rho \phi v)$  to vary proportionally with  $N$ , for a power law liquid with exponent  $n$ ,  $Po / (\rho \phi v)$  would vary at constant temperature roughly proportional with  $N^n$ . The experimentally observed insensitivity of the specific power input may thus indicate that at higher screw rotation speeds higher temperature buildup during the extrusion might decrease the apparent viscosity. Also excessive slip along the barrel wall might explain the results (i.e.  $n$  approaches zero).

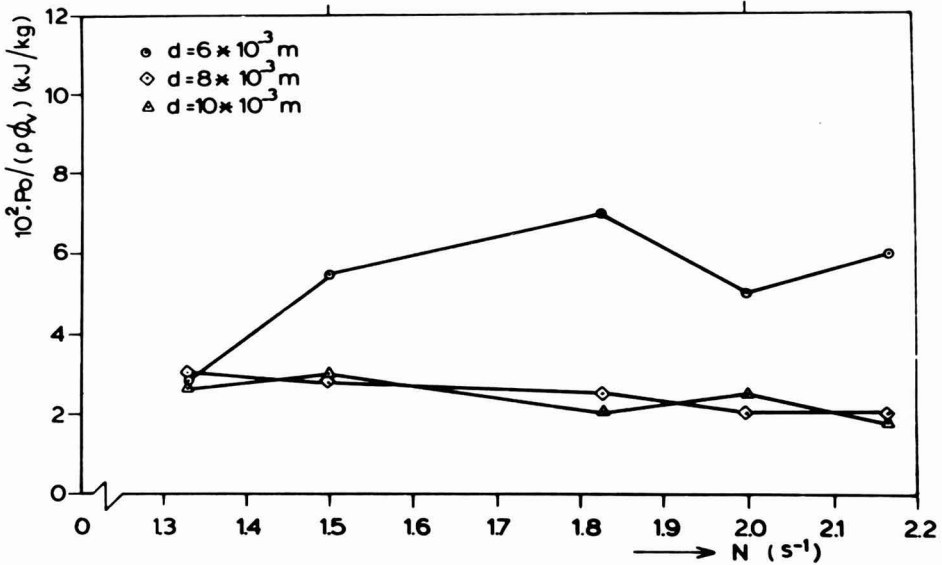


FIG. 22. SPECIFIC POWER CONSUMPTION DURING EXTRUSION OF DEFATTED SOY FLOUR

### CONCLUSIONS

The prediction of flow rates during extrusion of biopolymers in single screw extruders is at present not well possible. An important reason is the lack of rheological data for materials under extrusion conditions. A second important reason is that most biopolymers undergo chemical reactions during extrusion-cooking. These chemical reactions significantly change the rheological behavior and the conversion is influenced by the time-temperature history of the biopolymer during the extrusion process. The time-temperature history itself is, however, again influenced by the rheological behavior of the polymer. The use of rheological parameters measured on a specific extruder in predicting flow rates on an extruder with different geometry is therefore hardly possible, especially if a scale-up is involved. Much further work is needed to improve this situation.

The prediction of residence times and residence time distributions of corn grits is reasonably well possible with a model valid for Newtonian fluid. Work is underway at our laboratory for other types of biopolymers.

The power consumption during extrusion is mainly caused by viscous effects, hence the group  $Po/(\mu_{app} N^2 D^3)$  is the best way to correlate experimental results. We have indications that experiments, again with



corn grits, corroborate this. However, also in this case the lack of representative rheological data hampers adequate data correlation. Further work in this area is strongly recommended.

APPENDIX 1

The components of the stress tensor of a power law liquid for the flow geometry of a single-screw extruder.

The stress tensor  $\tau$  depends on the symmetrical rate of deformation tensor  $\Delta$  according to:

$$\tau = - \{m | \sqrt{1/2 (\Delta:\Delta)^{n-1}} \} \Delta \tag{I:1}$$

The components of  $\Delta$  in a cartesian coordinate system (i, j, k) are:

$$\Delta_{ij} = \left( \frac{\partial v_i}{\partial x_j} + \frac{\partial v_j}{\partial x_i} \right) \tag{I:2}$$

For the simplified flow geometry we say, a priori, that  $v_x = v_x(y)$ ,  $v_y = 0$ ,  $v_z = v_z(y)$ . This means that the rate of deformation tensor has the components:

$$\Delta = \begin{pmatrix} 0 & \frac{\partial v_x}{\partial y} & 0 \\ \frac{\partial v_x}{\partial y} & 0 & \frac{\partial v_z}{\partial y} \\ 0 & \frac{\partial v_z}{\partial y} & 0 \end{pmatrix} \tag{I:3}$$

The double dot product  $\Delta:\Delta$  then becomes:

$$\Delta : \Delta \equiv \sum \sum \Delta_{ij} \Delta_{ji} = 2 \left( \frac{\partial v_x}{\partial y} \right)^2 + 2 \left( \frac{\partial v_z}{\partial y} \right)^2 \tag{I:4}$$

The stress tensor  $\tau$  can now be calculated by substitution of (I:4) and (I:3) into equation (I:1):

$$\left. \begin{aligned} \tau_{xx} = \tau_{yy} = \tau_{zz} = \tau_{xz} = \tau_{zx} = 0 \\ \tau_{yz} = \tau_{zy} = -m \left| \sqrt{\left(\frac{\partial v_x}{\partial y}\right)^2 + \left(\frac{\partial v_z}{\partial y}\right)^2} \right|^{\eta-1} \left(\frac{\partial v_z}{\partial y}\right) \\ \tau_{yx} = \tau_{xy} = -m \left| \sqrt{\left(\frac{\partial v_x}{\partial y}\right)^2 + \left(\frac{\partial v_z}{\partial y}\right)^2} \right|^{\eta-1} \left(\frac{\partial v_x}{\partial y}\right) \end{aligned} \right\} \text{(I:5)}$$

## NOMENCLATURE

A	dimensionless group defined in eqn. (34)	(-)
b	coefficient for temperature dependence of viscosity	(K <sup>-1</sup> )
Br	$\mu_{av} b \pi^2 D^2 / \lambda =$ Brinkman number	(-)
C <sub>p</sub>	heat capacity	(kJ/kgK)
d	die diameter	(m)
D	barrel diameter	(m)
e	flight width in axial direction	(m)
E(t)	exit age distribution function	(s <sup>-1</sup> )
f	fraction of total residence time spent in region where v <sub>x</sub> is negative	(-)
F(t)	cumulative residence time distribution function	(-)
h	channel depth	(m)
I <sub>2</sub> , I <sub>3</sub>	second and third invariants of the rate of deformation tensor	(s <sup>-2</sup> ; s <sup>-3</sup> )
k	number of flights on extruder axle	(-)
l	length of die channel	(m)
L	length of extruder screw	(m)
m, m'	parameter in power law model	(Ns <sup>n</sup> /m <sup>2</sup> )
n	exponent in power law model	(-)
N	screw rotation speed in revolutions per second	(s <sup>-1</sup> )
p	pressure	(Pa)
P	ratio between pressure flow and drag flow	(-)
P <sub>n</sub>	same as P but for power law liquid	(-)
Pe	$= \pi N D h^2 \rho C_p \sin \theta / (\lambda L) =$ Péclet number	(-)
Po	power consumption	(W)
q	heat flux vector, component q <sub>x</sub> , q <sub>y</sub> , q <sub>z</sub>	(J/m <sup>2</sup> s)
Re*	$= pND^2 / \mu_{app}$ Reynolds number for non Newtonian liquid	(-)

s	width of flight	(m)
t	time	(s)
T	temperature	(K)
U	= $\pi N D$ relative velocity of barrel wall with respect to centre line of screw	(m/s)
v	velocity vector, components $v_x, v_y, v_z$	(m/s)
dV	volume element	(m <sup>3</sup> )
w	width of extruder channel	(m)
W	rate of work done by system on surroundings	(Nm/s)
x,y,z,	coordinates defined in Fig. 3	(m)

GREEK SYMBOLS

$\alpha$	parameter for temperature dependence of m	(K)
$\beta$	parameter for moisture content dependence of m	(kg/kg)
$\dot{\gamma}$	magnitude of shear rate	(s <sup>-1</sup> )
$\gamma$	shear strain (= $\dot{\gamma} \cdot t$ )	(-)
$\bar{\gamma}$	weighted average total strain (WATS) defined in equation (27)	(-)
$\bar{\gamma}^*$	= $\bar{\gamma} h/L$ normalized WATS	(-)
$\bar{\gamma}^{**}$	= $\bar{\gamma} h/(U_z \bar{t})$ reduced WATS	(-)
$\delta$	flight clearance	(m)
$\Delta$	rate of deformation tensor	(s <sup>-1</sup> )
$\xi$	dimensionless z-coordinate	(-)
$\eta$	non-Newtonian viscosity	(Ns/m <sup>2</sup> )
$\theta$	angle of flight	(-)
$\Lambda$	dimensionless group defined in eqn. (11)	(-)
$\lambda$	heat conductivity	(W/mK)
$\mu$	Newtonian viscosity	(Ns/m <sup>2</sup> )
$\xi$	dimensionless y-coordinate	(-)
$\rho$	density	(kg/m <sup>3</sup> )
$\tau$	shear stress tensor	(kg/ms <sup>2</sup> )
$\Phi_v$	volumetric flow rate	(m <sup>3</sup> /s)
$\Phi_m$	mass flow rate	(kg/s)
$\omega_w$	mass fraction of water	(kg/kg)

SPECIFIC SYMBOLS AND OPERATORS

$x^*$	made dimensionless
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$\bar{x}$	averaged quantity: $\bar{x} = \int_0^{\infty} x \times E(t) dt$	
$\nabla$	“del” or “nabla” operator	(m <sup>-1</sup> )
$\cdot$	vector dot product	
Det ( $\Delta$ )	determinant of rate of deformation tensor	
$\cdot\cdot$	tensor double dot product	
C.R.	compression ratio of screw	

### INDICES

app	apparent
av	at average temperature $(T_b + T_r)/2$
b	at barrel surface
c	in extruder channel
d	in die
$\delta$	in flight clearance
	in left hand direction
r	at screw root surface, in right hand direction
x,y,z	x-component, y-component, z-component

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# A VISCOSITY MODEL FOR A COOKING DOUGH<sup>1</sup>

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## ABSTRACT

*A five parameter semi-empirical model is developed describing the apparent viscosity of a cooking dough as a function of applied shear rate, temperature, and time-temperature history. Such a model will be useful in the mathematical simulation of the extrusion cooking process.*

*Other factors which can affect the apparent viscosity of a cooking dough are also discussed. The viscosity model is verified and the constants evaluated for a typical dough by combining the results of Amylograph and capillary viscometer experiments.*

## INTRODUCTION

Extrusion cooking is an increasingly popular food processing technique by which cereal, oilseed, or other protein/carbohydrate/water mixtures are converted to products such as meat analogs and extenders, pet foods, or snacks. The process combines mixing, high-temperature short-time cooking, texturizing and shaping of food, all in one relatively inexpensive step using equipment which closely resembles the screw extruders used in the processing of thermoplastics (DeMuelenare and Buzard 1969; Smith 1971; Rossen and Miller 1973).

This similarity between the food and plastic processing operations has been identified as the key to upgrading the art of extrusion cooking to a more scientific level. That is, the design and optimization of extrusion cooking processes, which are now at the "cut-and-try" stage, could be more rationally approached by applying the theory, mathematical models, and accompanying computational routines for simulating thermoplastic extrusion already well developed as a result of polymer industry research (Harper and Harmann 1973; Fricke *et al.* 1977). The lone obstacle in the critical path to achieving this technology transplant is the

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problem of describing the food, as if it were a thermoplastic resin, in terms of the physical properties required as input to an existing extruder modeling scheme, for example, that of Tadmor and Klein (1970).

For a specific extrudable food formula, physical properties such as density and specific heat can be estimated by analogy to other food materials or determined by a one-point measurement and assumed constant throughout the extrusion process without incurring substantial error (Fricke *et al.* 1977). One physical property which cannot be readily estimated nor assumed constant is the apparent viscosity (hereafter referred to as viscosity) which may vary more than an order of magnitude during the extrusion cooking process as a result of changes in shear rate, temperature, and intermolecular association.

Accordingly, the purpose of this study is to describe these viscosity changes in the form of a mathematical model suitable for incorporation into an extrusion cooker model, and to show how the constants in such a viscosity model can be evaluated for a typical food formulation.

## BACKGROUND AND THEORY

### Description of a Dough System

Unlike the case of homogeneous or simple mixtures of polymers, the food mixtures which are to be extruded (hereafter referred to as "doughs") are of such complexity that the exact chemical composition and structure cannot be readily determined. Therefore, for the purposes of this study a dough will be described by the ingredients from which it was prepared, a proximate analysis stating the relative amounts of the major chemical component types, and the reactions the substances will undergo while cooking.

**Ingredients.** A variety of ingredients are used to formulate doughs suitable for extrusion cooking. Flours and grits milled from wheat, corn, oats, rice and other cereal grains as well as oilseed products such as soybean flours, concentrates and isolates comprise the bulk of most dough recipes. Examples of other typical ingredients and specific recipes are mentioned in the following references: Anderson *et al.* 1969, DeMuelenare and Buzzard 1969, Mottern *et al.* 1969, Mustakas *et al.* 1970, Bookwalter *et al.* 1971, Conway 1971, Payne *et al.* 1973, and Lorenz *et al.* 1974.

**Proximate Analysis.** A proximate analysis of any ingredient before the addition of water would indicate the proportions of the following components: protein, carbohydrate, water, fat, fiber and ash. Table 1 shows the proximate analysis for defatted soy flour, a typical ingredient, and for



Table 1. Proximate analyses of a defatted soy flour and a defatted soy flour dough

Component	Defatted Soy Flour <sup>a</sup> Weight %	Defatted Soy Flour Dough <sup>b</sup> Weight %
Moisture	6.0	32.
Protein (N × 6.25)	53.0	37.
Carbohydrate	33.9	24.
Fat	0.9	7.
Fiber	2.5	7.
Ash	6.5	7.

<sup>a</sup>Staley I-200 Extracted Soy Flour, A. E. Staley Manufacturing Company, Technical Data Sheet No. 95.

<sup>b</sup>Hypothetical dough prepared by combining 100 parts of Staley I-200 Extracted Soy Flour with 40 parts water by weight.

a hypothetical dough prepared by mixing 100 parts of this flour with 40 parts water by weight.

**Reactions.** Two important reactions of dough which take place during extrusion cooking are protein denaturation and polysaccharide gel formation.

Proteins are linear polymers of  $\alpha$ -amino acids, that is, a long backbone chain of amide linkages each separated by a single carbon atom. The different properties of proteins arise from the order and type of pendant groups attached to these backbone carbon atoms. Each protein molecule type occurs naturally in a unique folded shape according to the hydrophilic and hydrophobic nature of the pendant groups. Both hydrogen bonds and disulfide bonds between pendant groups also contribute to their characteristic three-dimensional shape (Lehninger 1970).

Denaturation or, more specifically, irreversible thermal denaturation is the unfolding of these protein molecules in response to a rise in temperature. In some cases a return from a small temperature excursion will allow renaturation or refolding to the original shape. However denaturation can be irreversible if during the period of elevated temperature, individual protein molecules become oriented (for example, when subjected to a velocity gradient) in such a manner that upon cooling, they preferentially associate with neighboring molecules forming a network (Fig. 1). Disulfide-disulfide interchanges may also occur resulting in the conversion from intramolecular crosslinks to intermolecular crosslinks (Tanford 1968).

The carbohydrate fraction may be made up of polysaccharides, oligosaccharides and/or monosaccharides. Of particular interest is the polysaccharide starch. Starch occurs in granules about  $10^{-5}$ m in diameter which in turn consist of a mixture of two glucose polymers: amylose

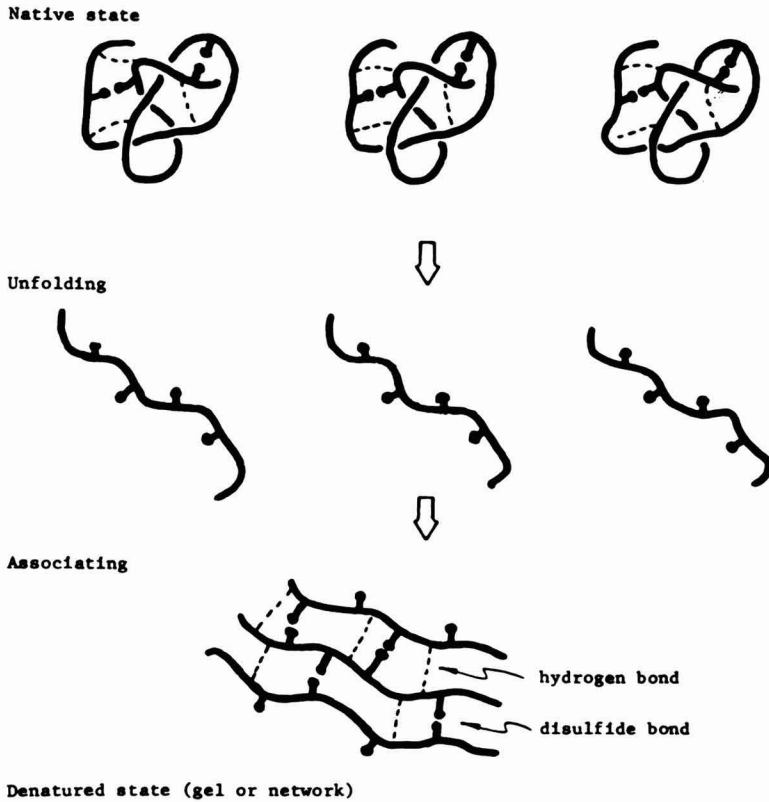


FIG. 1. MECHANISM OF PROTEIN DENATURATION

(helical and crystalline) and amylopectin (branched and amorphous). Upon the application of moist heat, starch undergoes gelatinization, a reaction discussed at length by Leach (1965). A reaction mechanism for gelatinization assumed for the purpose of this study is outlined in Fig. 2.

Note that unlike the reversible melting and solidification of a thermoplastic, the overall cooking reaction (a combination of denaturation and gelatinization) is essentially an irreversible network formation, joining or associating previously distinct and isolated particles or molecules by covalent bonds, hydrogen bonds or physical entanglements. This is where the extrusion cooking/thermoplastic extrusion analogy stops. Here a more appropriate comparison is made between doughs and thermosetting plastics.

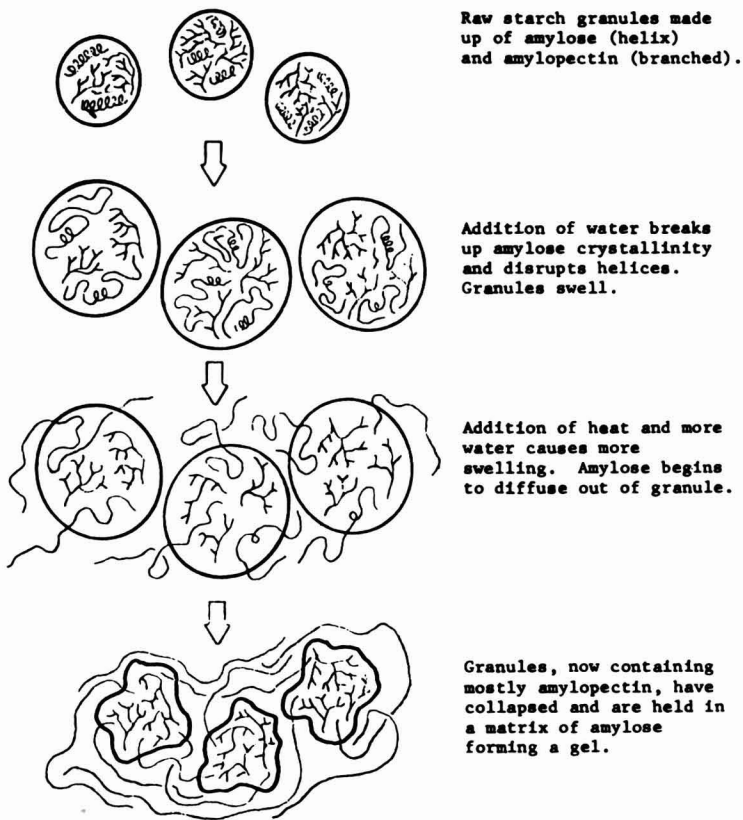


FIG. 2. MECHANISM OF STARCH GELATINIZATION

### Development of the Model

In this section, three variables which can affect the apparent viscosity of a dough during extrusion are discussed individually and then assembled into a proposed viscosity model. Other factors which may affect the dough viscosity but which were not included in the model are also mentioned.

**The Effect of Shear Rate.** Most polymer melts and solutions of polymers are pseudoplastic. This type of time-independent non-Newtonian flow behavior, characterized by decreasing apparent viscosity with increasing shear rate, is frequently represented by the power-law equation:

$$\eta = \frac{\tau}{\dot{\gamma}} = m (\dot{\gamma})^{n-1} \quad (1)$$

The constants  $m$  and  $n$  are called the consistency coefficient and the flow behavior index, respectively (Rodriguez 1970).

Since proteins and polysaccharides are polymers, mixtures of polysaccharides and/or proteins with water would be expected to exhibit pseudoplastic flow behavior. Table 2 lists some food systems of this type for which power law constants have been evaluated.

Table 2. Power-law constants measured on some pseudoplastic food systems

Food System	Temperature °C	Power-law Constants	
		$m$ dyne · sec <sup>n</sup> · cm <sup>-2</sup>	$n$ no units
Cooked cereal dough (Harper <i>et al.</i> 1971) 56% corn grits 14% oat flour 30% water	85	$2.2 \times 10^4$	0.51
Cooked cereal dough (Harper and Harmann 1974) 68% pregelatinized corn flour 32% water	88	$1.7 \times 10^5$	0.34
Raw wheat flour dough (Launay and Bure 1973) 57% wheat flour 43% water	33	$4.5 \times 10^4$	0.35
Raw sausage batter (Toledo <i>et al.</i> 1977) 12% protein 23% fat 63% water	15	$4.3 \times 10^3$	0.21
Milk protein suspension (Hermansson 1975) 20% casein 80% water	25	$2.9 \times 10^3$	0.90

**The Effect of Temperature.** The effect of temperature on the viscosity of a Newtonian fluid can be represented by an Arrhenius expression (Glasstone *et al.* 1951):

$$\mu = \mu_{\infty} \exp (\Delta E_{\eta} / RT) \quad (2)$$

For pseudoplastics, Metzner (1959) has argued that the same expression can be successfully used to describe the temperature dependence of the consistency coefficient, while the flow behavior index may be considered to be relatively temperature independent. Thus the power-law expression including temperature effects would be:

$$\eta = m_{\infty} \exp (\Delta E_{\eta} / RT) \dot{\gamma}^{n-1} \quad (3)$$

$\Delta E_{\eta}$  is an activation energy for a molecule or segment of a molecule to move from one equilibrium position to another relative to its neighbors.

**The Effect of Extent of Reaction or Time.** As stated earlier, denaturation and gelatinization may be considered to be aggregation or network forming reactions where individual molecules become interconnected at several points per molecule as a result of covalent bonds, hydrogen bonds, or physical entanglements. Any of these interactions will inhibit the movement of one molecule past another; therefore, an increase in apparent viscosity is expected. Note that, although a covalent network may exist, the flow of sulfur crosslinked protein chains past each other is still possible and is explained by Blokzma (1975).

Network formation or aggregation may also be thought of as an increase in molecular weight or effective molecular weight. The viscosity of polymer solutions is experimentally observed to increase proportional to a power of the molecular weight or chain length:

$$\eta = A_1 (MW)^{A_2} \quad (4)$$

or

$$\ln (\eta) = \ln \left[ A_1 + A_2 \ln (MW) \right] \quad (5)$$

$A_2$  usually has a value of 3.4 and only  $A_1$  is a function of temperature (Fox *et al.* 1956)

If one assumes that the mechanism of the denaturation and gelatinization reactions involves first the formation of small aggregates from individual molecules followed by the combination of the smaller aggregates to form larger aggregates, the effective molecular weight would then be a geometric function of the extent of reaction or some reaction coordinate,  $\epsilon$ , which can be expressed:

$$(MW) = A_3 (\epsilon)^{A_4} \quad (6)$$

or after substitution into Equation (5):

$$\ln(\eta) = A_5 + A_6 \ln(\epsilon) \quad (7)$$

The reaction coordinate is related to time by kinetics. In general, first order or pseudo first order kinetics have been shown to hold for protein denaturation (Johnson *et al.* 1954) as well as starch gelatinization (Suzuki *et al.* 1976). That is:

$$\ln(\epsilon) = A_7 + A_8 t \quad (8)$$

or, after combination with Equation (7) is:

$$\ln(\eta) = A_9 + kt \quad (9)$$

where  $k$  is an "apparent kinetic factor" as defined by Roller (1975).

Roller (1975), in studying the curing of epoxy resins noted that the viscosity during this thermosetting reaction followed the relationship of Equation (9), and then made the following modification to account for non-isothermal reaction conditions:

$$\ln(\eta) = \ln(\eta^0) + \Delta E_\eta/RT + \int_{t_0}^t k_\infty \exp(\Delta E_k/RT) dt \quad (10)$$

**The Overall Model.** The overall viscosity model for a cooking dough is obtained by combining Equations (1) and (10) to yield a function of shear rate, temperature and time-temperature history:

$$\begin{aligned} \ln \eta(T, \dot{\gamma}, t) = & \ln(\eta^*) + \Delta E_\eta/RT \\ & + (n - 1) \ln \dot{\gamma} + \int_{t_0}^t k_\infty \exp(\Delta E_k/RT) dt \end{aligned} \quad (11)$$

The three independent variables are shear rate,  $\dot{\gamma}$ ; temperature,  $T$ ; and time since the start of reaction,  $t$ . The five constants which must be evaluated are a reference viscosity,  $\eta^*$ ; the flow behavior index,  $n$ ; the activation energy for the flow process,  $\Delta E_\eta$ ; the apparent kinetic factor at infinite temperature,  $k_\infty$ ; and the activation energy for the cooking reaction,  $\Delta E_k$ , be it denaturation or gelatinization, or both.  $R$  is the universal gas constant.

### Effects Not Included in the Model But Still Worthy of Note

The effects discussed here have not been included in the viscosity model for a cooking dough for one or more of the following reasons: the effect is or can be made negligible, the effect is too complex to quantify, or the effect only occurs in certain flow conditions. It will be necessary to avoid or account for some of these effects when measuring the viscosity of a particular dough.

**Thixotropy.** Fluids with some degree of order or structure such as a protein or starch gel may tend to break down when subject to a constant shear rate, resulting in a decrease in viscosity with time (Launay and Bure 1973). This effect may partially or totally counteract the increase in viscosity caused by the forward or gelling reaction. To account for this thixotropic effect an additional term must be included in Equation (11). This term, inherently negative, would be added to the right side of Equation (11) and would be of the form:

$$(\Delta \ln \eta)_{\text{thixo}} = \int_{t_0}^t f(\dot{\gamma}) dt \quad (12)$$

Because this term has been neglected in the overall model, any thixotropic effect would show up as a shear dependent reduction of the apparent kinetic factor,  $k_{\infty}$ , as if the thixotropic effect were a reaction inhibitor or a reverse reaction. Since one of the purposes of extrusion cooking is to build up gel structure, hence texture, it would be advantageous to keep the residence time in the extruder short enough to avoid or minimize thixotropic breakdown. The data do not yet exist to establish how significant this effect may be in practice.

**Viscous Heating.** Viscous heating occurs if the internal frictional heat, caused by molecules flowing past one another, is not transferred to the surroundings faster than it is generated. The resulting accumulation of energy produces a temperature rise, and therefore a drop in viscosity. Since the magnitude of this effect is proportional to the product of the shear stress and shear rate (or  $\eta\dot{\gamma}$ ) it is most significant in high viscosity fluids at high shear rates. Viscous heating effects are often mistaken for thixotropic effects (Van Wazer *et al.* 1963).

Because it can be treated as a temperature effect, there is no explicit viscous heating term in the proposed viscosity model.

**Dependence of Flow Behavior Index on Extent of Reaction.** An interesting observation has been made on the effect of the extent of reaction on the power-law constant,  $n$ , or flow

behavior index. In two studies on reacting power-law fluids, one on the coagulation of milk by rennet (Tuszynski and Scott Blair 1967) and one on the retrogradation of a potato starch gel by  $\alpha$ -amylase (Nedonchelle and Schutz 1967), conditions were such that temperature was constant and the only variable was extent of reaction or the effective gel molecular weight. Flow curves were measured at various extents of reaction and fit to a power-law ( $\tau = m\dot{\gamma}^n$ ) equation, thereby generating a set of  $m, n$  ordered pairs, one pair for each extent of reaction investigated. The power-law constants were shown to be interrelated by an equation of the form:

$$\ln(m) = -A_{10}(n) + A_{11} \quad (13)$$

Therefore,  $n$  must not really be a constant, but rather a function of time and temperature:

$$n(t, T) = n^* - \int_{t_0}^t A_{12} \exp(\Delta E_k/RT) dt \quad (14)$$

If this expression were substituted into the overall model Equation (11) and the time integrals combined, the pre-exponential factor  $k_\infty$  would not be a constant but rather a linear function of the logarithm of the shear rate.

$$\begin{aligned} \ln(\eta) = & \ln(\eta^*) + (\Delta E_\eta/RT) + (n^* - 1) \ln(\dot{\gamma}) \\ & + \int_{t_0}^t (k_\infty - A_{12} \ln(\dot{\gamma})) \exp(\Delta E_k/RT) dt \end{aligned} \quad (15)$$

The evidence does not yet exist to establish that soy flour behaves this way.

**Composition.** Harper *et al.* (1971) have shown that over a very narrow range of water concentration (25–35%) the logarithm of viscosity is proportional to water content. The concentration effect on the viscosity behavior of suspensions of rigid spheres (Kao *et al.* 1975) has been applied to suspension of protein but was not successfully extrapolated to high (>50%) solids concentrations (Remsen 1977). In extrapolation to high concentrations, one may unknowingly pass through a possible discontinuity where water ceases to be the continuous phase. Furthermore, as a dough is cooked, the mixture becomes less like a suspension of rigid spheres and more like a polymer solution.

All the constants in Equation (11) are likely to be dependent on the



selection of the dry ingredients. When enough values of these constants are known, "mixing rules" for each constant can be developed, perhaps in the format suggested by Harper *et al.* (1971), namely, composition perturbations about a set base formula.

**Viscoelasticity.** Flour doughs are generally considered to be complex viscoelastic materials (Greup and Hintzer 1953; Muller 1969; Funt Bar-David and Lerchenthal 1975; Greenwood and Ewart 1975). For steady flow in a conduit, the viscoelastic effect presents itself as a stored energy which must be built up during the first application of shear and is recovered upon the termination of shearing conditions. A normal stress (i.e., in a direction perpendicular to the flow direction), defined as the product of the shear stress and the recoverable viscoelastic strain or deformation, is a measure of this stored energy (Philippoff and Gaskins, 1958).

The viscoelastic effects were assumed to be limited to entrance and exit phenomena, and therefore negligible when modelling the flow in the screw channel of an extruder. Hence, they were omitted from the proposed model given by Equation (11). Viscoelasticity however will be important when measuring the viscosity of doughs and in the design of dies for food extruders.

**Yield Stress.** There is some question as to whether to include a yield stress in viscosity models for food mixtures of this type. Most experimental data are fit to the power-law model (Equation (1)) assuming no yield stress. Some investigators (Hermansson 1975; Toledo *et al.* 1977; Kaufman and Hatch 1977), for the sake of a better fit, have summarized selected flow curve data using a "mixed type" or Herschel and Bulkley model which allows for a non-zero intercept on the shear stress axis:

$$(\tau - \tau_0) = m (\dot{\gamma})^n \quad (16)$$

Even though the mixed type model may provide a better fit in the range of the experimental data, the pure power law extrapolates to zero shear rate with higher fidelity to the pseudoplastic concept. (For examples of pure pseudoplastic flow curves see: Ostwald and Auerbach 1926; Beuche 1954; Merrill 1959). The better fit provided by Equation (16) over Equation (1) has been at the expense of switching from a 2-constant equation to a 3-constant equation.

## EXPERIMENTAL

### Special Considerations When Measuring the Viscosity of a Cooking Dough

In studying cooking (denaturation and gelatinization) kinetics, one is

faced with several questions: Is change in viscosity the best indicator of extent of reaction or can the kinetics be more accurately measured by another method? Which reaction mechanism step corresponds to the chosen extent of reaction indicator? And, finally, how does one account for the effect of temperature on the reaction kinetics?

Nitrogen solubility is one method frequently used to measure the extent of the protein denaturation reaction (Belter and Smith 1952; Wolf 1971; Shen 1976). Other indicators of degree of denaturation or gelatinization have been examined, for example, a change in specific heat (Hoyer 1968), the degradation of thiamine and riboflavin in corn grits (Beetner *et al.* 1974), the inactivation of an enzyme or an enzyme inhibitor (Mustakas *et al.* 1970) or the softening of rice grains due to moisture take-up as they cook (Suzuki *et al.* 1976). Effective utilization of any of these methods for our purposes would have to include proof that the kinetics of the reaction which produces the indicator parallels that of the reaction which produces the increase in viscosity.

The mechanisms for the protein denaturation and starch gelatinization reactions presented in Fig. 1 and 2 are greatly simplified and generalized. For example, there is a possibility that protein molecules can join an aggregation without first unfolding, or that the protein can unfold and become insoluble but not aggregate, producing no increase in viscosity. Mechanisms which incorporate these effects would contain branches and shunts (Johnson *et al.* 1954; Warner and Levy 1958; Catsimpoolas and Meyer 1970). Thus, any of the above mentioned extent of reaction indicators could correspond to an unimportant step in the reaction mechanism or one which is unrelated or not representative of the aggregation step that results in the viscosity increase.

The temperature dependence of the rate of reaction also presents a problem. It is difficult to get meaningful data from isothermal reaction studies since the reaction will occur during the warm-up of the sample (Wolf and Tamura 1969). Non-isothermal data must be obtained from a system so defined that the temperature is known at any time and at any point in the reactor. This condition specifies a reactor with a simple geometry and a programmed temperature scan.

Another interesting feature of both protein denaturation and starch gelatinization is the existence of a critical temperature below which no reaction occurs. In the case of proteins, this can be attributed to the unfolding step (Fig. 1). The protein has been held in its characteristic shape by the collective strength of many weak bonds (a phenomenon termed "cooperativity"). The breaking of one of these bonds therefore increases the likelihood that the next bond will break and so forth, so that the protein molecule literally "unzips" itself. Below the critical temperature the molecule is completely folded in the native state, and above it is com-

pletely unfolded in the denatured state. There is apparently no intermediate state (Lehninger 1975). This critical temperature has been observed at 50 °C for wheat gluten (Bale and Muller 1970), 60 °C for single cell protein concentrate (Huang and Rha 1971), and 65 °C for soy protein isolate (Circle *et al.* 1964). For starch the critical temperature is between 55 and 70 °C depending on the plant source of the starch (Leach 1965).

### **Purpose, Method Selection and Strategy**

The purpose of the experimental work is to verify the proposed form of the viscosity model (Equation (11)) and to evaluate the constants for a typical dough.

The typical dough chosen is described in Table 1. Defatted soy flour was selected because it is a common ingredient in many extruded products and also because it is relatively starch free. As a result of the latter, only one reaction, protein denaturation, need be considered. The 100:40 flour (wet basis) to water recipe yields a 32% moisture dough which is in the 25–35% moisture range of commercial extruder feed formulas.

A detailed discussion of the selection of the instruments for measuring the viscosity of the material as it cooked may be found elsewhere (Remsen 1977). Since the dough was expected to have a high viscosity and viscoelastic properties a capillary technique was chosen over coaxial cylinder or cone-and-plate methods. But because of the flow-through nature of any capillary method, the time dependent effects on dough viscosity could not be measured. The Amylograph is equipped to handle time dependent effects, but it is a constant (and unknown) shear rate device and its use is further limited to flour/water mixtures of low concentrations (i.e. suspensions rather than doughs).

The strategy therefore involved the use of both instruments since no one viscometer available was capable of monitoring all of the variables at once. A capillary viscometer was used to study the viscosity of dough in a temperature range below that temperature at which the protein denatures and in a range of shear rates encountered in an extruder cooker. The Amylograph was then used to study the relative viscosity change during the cooking reaction as a function of time and temperature.

### **Capillary Viscometer Experiment**

The Instron capillary rheometer was chosen for this experiment because the instrument is capable of handling high-viscosity material and can maintain close temperature control. A detailed description of this instrument appears in Van Wazer *et al.* (1963).

**Procedure.** The dough sample was prepared according to the recipe in

Table 1 using a Hobart mixer with a paddle blade at the lowest speed. The action of the mixer blade cut the dough into crumbs about one-sixteenth of an inch in diameter. The sample was sealed into polyethylene bags until used.

For each run, the dough sample was loaded 2 or 3 crumbs at a time into the top of the barrel and tamped down. When the barrel was nearly full, the plunger was run down on top of the sample and then stopped at the first appearance of extrudate at the bottom of the capillary. The sample was allowed to equilibrate to the barrel temperature for 10 min.

Temperature was varied from 35°C to 60°C, crosshead speed was varied from 0.02 in./min to 1.0 in./min, and capillary length was varied from 1 in. to 4 in. The capillary diameter was 0.05 in. and constant. At each set of conditions, plunger force measurements were taken in triplicate and averaged.

**Analysis of Data.** The following equations were used to reduce the data:

$$\tau_w = \frac{F/A_z}{4 [(L/D) + N]} \times \left( 6.944 \times 10^4 \frac{\text{dyne/cm}^2}{\text{lb}_f/\text{in}^2} \right) \quad (17)$$

or

$$\dot{\gamma} = \frac{32 V_{xh} A_z}{\pi D^3} \left( \frac{3n + 1}{4n} \right) \times \left( \frac{1}{60} \frac{\text{min}}{\text{sec}} \right) \quad (18)$$

No slip at the wall is assumed.

To obtain the end correction,  $N$ , data was taken at 50°C using each of the 4 capillaries. For each crosshead speed a plot of plunger force versus capillary length to diameter ratio was made and the corresponding regression lines were extrapolated to zero plunger force (Fig. 3). The intercept on the "L/D" axis is  $N$ , the combined end effect correction of Bagley (1957) and the viscoelastic normal force correction of Philippoff and Gaskins (1958). In this case, the end corrections exhibited a dependence on crosshead speed as follows:

$$\ln(N) = 2.22 - 0.279 \ln(V_{xh}) \quad (19)$$

The Rabinowitsch correction (Duvdevani and Klein 1968) for shear rate at the wall was obtained by plotting  $\ln(\tau_w)$  versus  $\ln(\dot{\gamma}_w)$  assuming  $n = 1$  for each temperature studied. The slopes of the regression lines fit to these data sets yielded an average flow behavior index of  $0.34 \pm 0.01$ , independent of temperature.

Consistency coefficients,  $m$ , were calculated for each run by plotting  $\ln(\tau_w)$  versus  $\ln(\dot{\gamma})$  this time using  $n = 0.34$ . The intercepts of the regres-

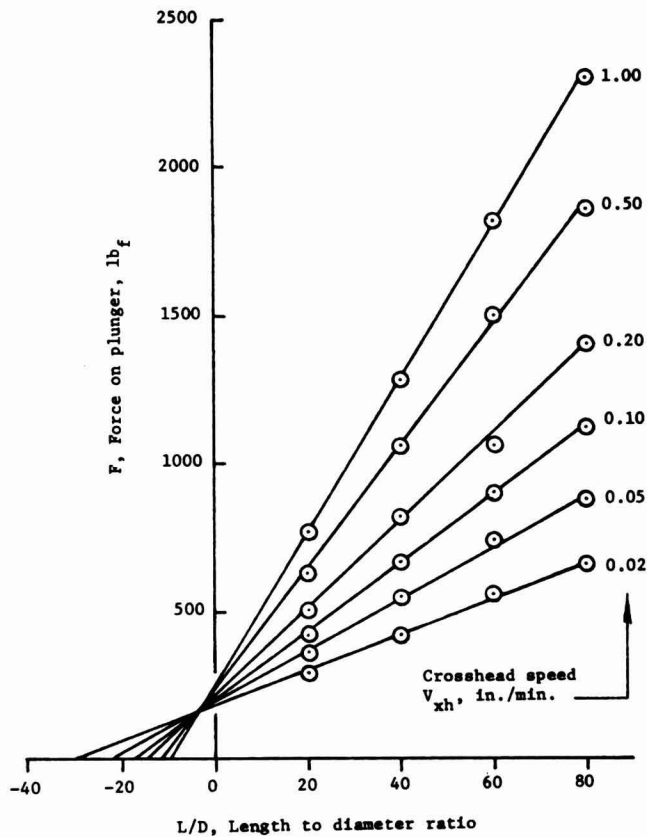


FIG. 3. BAGLEY PLOT FOR 32% MOISTURE EXTRACTED SOY FLOUR DOUGH AT 50°C IN A 0.05 INCH DIAMETER CAPILLARY WITH A 90° ENTRANCE ANGLE

sion lines on the  $\ln(\tau_w)$  axis is the natural logarithm of the consistency coefficient.

Finally, the log of the consistency coefficient was plotted versus the reciprocal of the absolute temperature (Fig. 4). The slope and intercept of the regression line are the constants for the Arrhenius temperature dependence of the consistency coefficient.

**Results.** The overall results may be summarized in a recast form of Equation (3):

$$\ln(\eta) = \ln(m_\infty) + \frac{\Delta E_\eta}{RT} + (n-1) \ln(\dot{\gamma}) \quad (20)$$

For which the constants have been evaluated in this experiment to be:

$$\ln(m_\infty) = 2.07$$

$$\Delta E_\eta = 7300 \text{ cal/mol}$$

$$n = 0.34$$

A hint as to the effect on viscosity of moisture loss from the sample is given in Fig. 4. As the sample dried out with time it became more viscous but the Arrhenius activation energy is apparently unaffected.

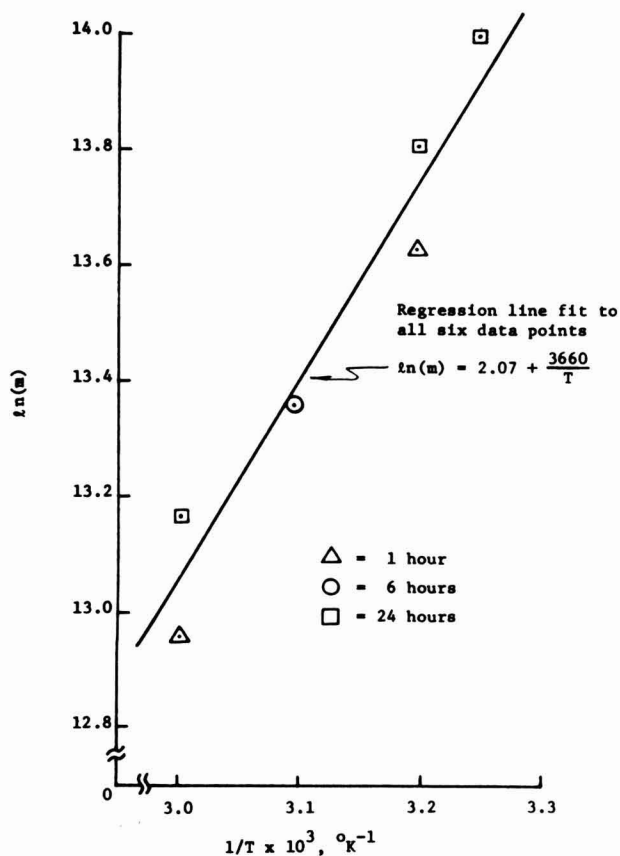


FIG. 4. TEMPERATURE DEPENDENCE OF THE CONSISTENCY COEFFICIENT

Symbols indicate time elapsed between sample preparation and measurement.

### Amylograph Experiment

The Amylograph is a special purpose device used in the food industry to measure relative changes in the viscosity of aqueous starch or protein suspensions (>75% water) as they gel when subject to an increase in temperature. The instrument provides a continuous viscosity measurement during a programmed temperature rise. A detailed description of the instrument is found in Van Wazer *et al.* (1963). The flow in the Amylograph is too complex to describe mathematically. For one particular set of conditions, the effective shear rate was estimated to be 40 or 50 reciprocal seconds (Wood and Goff 1973).

**Procedure.** For all runs, the bowl speed was 50rpm and the heating rate was 1.5 °C/min. Either the "350" or "700" sensitivity torsion spring was used. A 200-g balance weight was placed in the zero suppression pan when needed to provide 940 Brabender Units of zero suppression with the "700" sensitivity spring in place.

Staley I-200 extracted soy flour (Table 1) was mixed with distilled water in a Hobart mixer at the lowest speed until homogeneous. Twenty and 25 weight % flour suspensions were prepared. Thinner suspensions were not viscous enough to cause a significant pen deflection on the strip-chart and thicker suspensions were too viscoelastic and tended to climb out of the Amylograph bowl when subjected to shear. The bowl was filled to the top of the bowl pins and the viscosity measurement was started within 15 min after sample preparation.

One run was made with the 25% suspension using the "700" sensitivity spring and 2 runs were made on the 20% suspension, one with the "350" and one with the "700" sensitivity springs. The curves were transferred from the stripchart to rectangular coordinates (Fig. 5) after adjustments were made for scale changes and zero suppression. The 2 runs at 20% flour concentration were superimposable.

**Analysis of Data.** It is desired to fit these curves to the constant shear rate form of the proposed model:

$$\ln \eta = \ln \eta^0 + \Delta E_{\eta} / RT + \int_{t_0}^t k_{\infty} \exp(\Delta E_k / RT) dt \quad (21)$$

Each curve in Fig. 5 was reduced to a set of 67 ordered pairs spaced at one-degree centigrade intervals. From the shape of the curves, 70 °C was taken to be the temperature below which the denaturation reaction did not occur. This assumption allowed setting the last term in Equation (21)

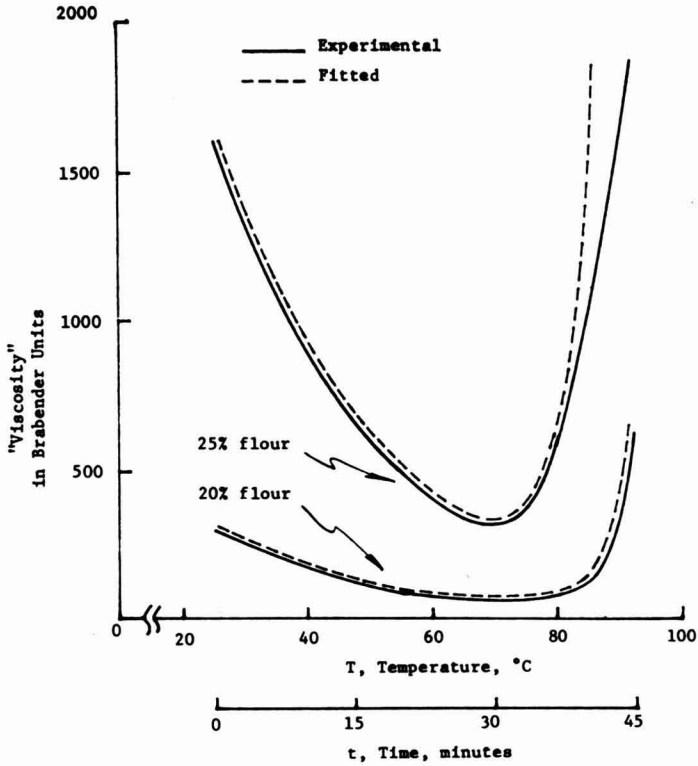


FIG. 5. AMYLOGRAPH CURVES FOR EXTRACTED SOY FLOUR SUSPENSIONS IN WATER. 1.5°C/MIN. HEATING RATE. "700" SENSITIVITY SPRING

to zero while  $\ln \eta$  versus  $1/T$  data for all the data below 70°C was fit by linear regression to:

$$\ln \eta = \ln \eta^0 + \Delta E_{\eta}/RT \quad (22)$$

Then Equation (21) was rearranged to yield:

$$\ln \eta - \ln \eta^0 - \Delta E_{\eta}/RT = \int_{t_0}^t k_{\infty} \exp(\Delta E_k/RT) dt \quad (23)$$

and differentiated with respect to temperature:

$$\frac{d}{dT} (\ln \eta - \ln \eta^0 - \Delta E_{\eta}/RT) = k_{\infty} \exp(\Delta E_k/RT)/(dT/dt) \quad (24)$$



Note that  $dT/dt$  is the heating rate,  $1.5^\circ\text{C}$  per min, a constant.

Next the data for the second part of the curve was fit to a logarithmic form of the above equation by linear regression:

$$\ln \left[ \frac{d}{dT} (\ln \eta - \ln \eta^0 - \Delta E_\eta / RT) \right] = \ln (k_\infty / 1.5) + \Delta E_k / RT \quad (25)$$

The temperature derivative on the left-hand side of Equation (24) was evaluated numerically at each of the 67 points.

The values of the constants  $k_\infty$  and  $\Delta E_k$  obtained via this last linear regression were then used as starting guesses in a half-interval search routine to find the values of these constants which would minimize:

$$\sum_{T=25^\circ\text{C}}^{92^\circ\text{C}} (\eta_{\text{actual}} - \eta_{\text{calculated}})_T^2 \quad (26)$$

**Results.** The evaluated constants are listed in Table 3 and the fitted curves are plotted in Fig. 5. For the 20% flour suspension the model matches the actual curve to within  $\pm 20$  Brabender Units at any point. The model matches the 25% suspension curve well for temperatures below  $83^\circ\text{C}$ , but overestimates the viscosity at temperatures above this point. This error can be explained by a foaming condition which forced a fraction of the sample above the top of the bowl pins or a thixotropic breakdown of newly formed gel. In our considered judgment, there is no significant difference in the values reported in Table 3 for the constants  $k_\infty$ ,  $\Delta E_k$ , and  $\Delta E_\eta$  for the two different concentration suspensions.

Table 3. Constants evaluated for equation (21) from Amylograph curves<sup>a</sup>

Constant	Suspension Flour Concentration	
	20%	25%
$\ln \eta^0$	- 7.48	- 5.91
$\Delta E_\eta$ , kcal/mol	7.8	7.9
$k_\infty$ , $\text{min}^{-1}$	$6.6 \times 10^{16}$	$5.8 \times 10^{16}$
$\Delta E_k$ , kcal/mol	-28.5	-28.0

<sup>a</sup>Equation (21) is:

$$\ln \eta = \ln \eta^0 + \int_{t_0}^t k_\infty \exp (\Delta E_k / RT) dt$$

### Combining the Experimental Results

The overall model (Equation (11)) for the viscosity of cooking soy flour dough complete with evaluated constants is obtained by adding the ex-

tent of reaction effects quantified in the Amylograph experiment to the capillary experiment results:

$$\ln \eta = 2.07 + \frac{7300}{RT} + (0.34 - 1) \ln \dot{\gamma} \quad (27)$$

$$+ \int_{t_{T=70}}^{t_{T>70}} 6 \times 10^{16} \exp(-28,000/RT) dt$$

Note that by just simply "adding" this reaction effect term to the capillary viscometer experiment results, the assumption has been made that the relative increase in apparent viscosity due to cooking is the same for concentrated flour suspensions as it is for doughs.

### DISCUSSION OF RESULTS

Unfortunately, there are no viscosity data on cooking doughs presently available to compare to the viscosity behavior predicted by this overall model. Therefore, other than the plots comparing the experimental data and the functions fit to the model fragments as described above, the only remaining criterion for evaluating the validity of the postulated model is to judge how reasonable or physically meaningful are the values obtained for the model parameters for the typical dough.

A value of 0.34 for the flow behavior index,  $n$ , was not unexpected and compares well with the values listed in Table 2 for other typical doughs.

For the activation energy for the Arrhenius temperature dependence of viscosity,  $\Delta E_{\eta}$ , the 7.3 kcal/mol. value obtained for the soy flour dough in the capillary experiment is close to the 7.8 and 7.9 kcal/mol values derived from the Amylograph experiment for suspensions, indicating that this constant is probably not a strong function of water concentration. For a corn and oat dough, Harper *et al.* (1971) measured a value of 5.0 kcal/mol for this constant which, when compared to the results of this study suggests a dependence on dry ingredient composition.

The activation energy for the protein denaturation reaction  $\Delta E_k$  is 28 kcal/mol which falls in the 20 to 30 kcal/mol range of activation energies listed by Eyring and Stearn (1939) for the thermal denaturation reactions of 23 different proteins.

The parameters  $k_{\infty}$  and  $\eta^*$  are somewhat more difficult to judge. The value for  $k_{\infty}$  should be very large since at moderate temperatures it will be multiplied by the exponential of a small negative number. The value of

$10^{16}$  reciprocal minutes obtained for the cooking soy flour can be loosely compared to the range of values  $10^8 - 10^{12}$  reciprocal minutes measured by Roller (1975) for curing epoxies. On the other hand,  $\eta^*$  would be expected to be, and turns out to be, fairly small since it represents the apparent viscosity of the dough at an infinitely high temperature and an infinitesimal shear rate in the absence of any reaction.

### CONCLUSIONS

(1) Soy flour dough is pseudoplastic, can be represented by a two-parameter power-law viscosity equation, and its apparent viscosity decreases with increasing temperature according to an Arrhenius relationship. The flow behavior index is essentially independent of temperature.

(2) Above  $70^\circ\text{C}$ , the protein in soy flour dough denatures and the logarithm of the viscosity of a soy flour/water mixture will increase proportional to reaction time. This proportionality constant or apparent rate constant is a function of temperature.

(3) The effects mentioned in the previous two conclusions can be summarized in a single model expressing apparent viscosity versus time, temperature and shear rate (i.e., Equation (11)) in a form suitable for incorporation into an extruder cooker modelling scheme.

(4) The constants in this model can be evaluated using the combined results from Amylograph and capillary viscometer experiments if the assumption is made that the relative increase in viscosity due to cooking is the same for concentrated suspensions (20% flour) as it is for doughs (70% flour).

(5) Other rheological phenomena such as viscoelasticity, thixotropy, and viscous heating, although not included in the model, are nonetheless present in dough systems and must be avoided or accounted for when measuring the apparent viscosity of doughs.

(6) Since the gelatinization of starch is similar to the denaturing of protein in that they both involve the aggregation of previously discrete and isolated particles or molecules, the viscosity behavior of a starchy dough or a starch/protein dough would be expected to follow the model developed in this study (Equation 11).

### RECOMMENDATIONS FOR FURTHER STUDY

The five constants in the new viscosity model have been evaluated for only one dough formulation. By repeating the capillary and Amylograph

experiments on other dough formulations (different moisture contents and different dry ingredient blends), "mixing rules" for each constant could be developed such that the constants for a new formulation could be predicted without having to perform any additional experiments. The mixing rules could be on the basis of the proportions of flour types in the recipe or overall percent protein, percent starch, etc.

Clearly the weakest point in this study is the assumption that the relative increase in apparent viscosity due to cooking is the same for concentrated flour suspensions as it is for doughs. This assumption had to be made in order to combine the results from the Amylograph (an instrument which cannot handle stiff doughs and has a constant and undefined shear pattern) with the results from the capillary experiment (an instrument incapable of measuring time dependent effects).

This problem could be avoided by using a single viscometer. Such a viscometer might ideally be a rotational type, preferably cone-and-plate, so that the same sample material would be subject to the same mathematically definable shear rate for the entire test run. A closed cavity to contain the sample would be required to counteract the normal forces which tend to squirt the sample out of the gap. Some commercial closed cavity viscometers currently available are the Rheometrics Mechanical Spectrometer biconical attachment (Madonia and Gogos 1971; Broyer and Mackosko 1975), and the Monsanto Rheometer or Mooney Viscometer used for test curing rubber (Wise 1973).

The sample cavity should be designed to provide evenly distributed heat transfer to the sample to avoid temperature gradients and hot spots. The temperature control should allow for programmed scans. Isothermal reaction runs are of limited value because the reaction will occur during the sample warm-up period.

A viscometer with all these characteristics would also permit an investigation of thixotropic effects, any effect of extent of reaction on the flow behavior index, and the shear rate dependence of the apparent kinetic factor,  $k_{\infty}$ .

Finally, the viscosity model for cooking dough developed in this study should be used in conjunction with a Tadmor and Klein (1970) type extruder model to simulate an extruder cooker.

## NOMENCLATURE

$A_1, A_2, \dots$	general constants
$A_z$	cross section area of barrel or plunger of Instron Capillary Rheometer, in <sup>2</sup>
D	diameter of capillary in a capillary viscometer, in

e	2.718281....
$\Delta E_\eta$	activation energy for the viscous flow process, cal/mol
$\Delta E_k$	activation energy for the cooking reaction, cal/mol
f	an unspecified function used in Equation (12)
F	force on the plunger of the Instron Capillary Rheometer, lb <sub>f</sub>
k	apparent kinetic factor as defined in Equation (9), min <sup>-1</sup>
$k_\infty$	apparent kinetic factor at infinite temperature, min <sup>-1</sup>
L	length of capillary in a capillary viscometer, inches
m	consistency coefficient, a power-law parameter, dyne sec <sup>n</sup> /cm <sup>2</sup>
$m_\infty$	consistency coefficient at infinite temperature, dyne sec <sup>n</sup> /cm <sup>2</sup>
MW	effective molecular weight of aggregate or gel, g/mol
n	flow behavior index, a power-law parameter, dimensionless
n*	flow behavior index of an uncooked flour water mixture, dimensionless
N	end correction for a capillary viscometer, dimensionless
R	gas constant, 1.98717 cal/mol °K
t	time, min
$t_0$	time at start of cooking reaction, min
T	temperature, °C or °K
$V_{xh}$	crosshead speed of Instron Capillary Rheometer, in/min
$\dot{\gamma}$	shear rate, sec <sup>-1</sup>
$\dot{\gamma}_x$	shear rate at the wall corrected for a non-Newtonian flow profile in a capillary viscometer, sec <sup>-1</sup>
ε	reaction coordinate, dimensionless
η	apparent viscosity defined as the shear stress divided by the shear rate, poise
η <sup>0</sup>	reference apparent viscosity, poise
η*	reference apparent viscosity, poise
μ	Newtonian viscosity coefficient, poise
μ <sub>∞</sub>	Newtonian viscosity coefficient at infinite temperature, poise
π	3.1415926.....
τ	shear stress, dyne/cm <sup>2</sup>
τ <sub>w</sub>	shear stress at the wall in a capillary viscometer, dyne/cm <sup>2</sup>

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# DESIGN AND MODELING OF A CAPILLARY FOOD EXTRUDER

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## ABSTRACT

*A simple capillary ram food extruder was designed, constructed and mathematically modeled. The mathematical model was developed from a group of meaningful dimensionless parameters derived from a dimensional analysis of the extruder. This approach to modeling greatly reduced the number of experiments required to determine the model equation constants.*

*Commercial defatted soy flour was used to conduct tests for validating the model. Flour moisture content, die length and die diameter were varied with all other variables remaining constant. Pressure loss through the extruder was the measured variable.*

*The mathematical model fit the observed data accurately. It is proposed that once all experimental constants are determined, the general model may be used to determine rheological and thermodynamic data for doughs during extrusion processes. These data may then be used to develop certain rheological material functions.*

## INTRODUCTION

New texturized plant protein products are being developed each year. Extruders first became important to the food industry as instruments to mix flours with steam or water and deliver these mixtures at elevated pressures to extrusion dies (Farrell 1971). Extruders have since been used as forming and/or cooking instruments in the food industry.

Harper and Harmann (1973) have effectively documented some of the research needs in extrusion cooking and forming. They concluded that to facilitate further applications of the food extruder, there are specific research needs in the following areas where special attention should be given: (a) extruder modeling, (b) die design, (c) physical properties, (d) product quality, and (e) process control.

Mathematical models of simple screw extruders have been developed and tested using polymer extrusion theory for plastics (Harmann and Harper 1973, 1974). Numerous screw extruder flow equations have been developed by Carley *et al.* (1953), Chung (1971) and Squire (1958) that may be applied to plant-protein extrusion theory.

However, the thermodynamic and rheological properties of plant-protein doughs during extrusion must be known before theoretical screw extruder equations can effectively be used for modeling. Because their properties are functions of operating variables such as temperature, shear rate, temperature-time history, moisture content, etc., adequate thermodynamic and rheological models are needed for application to screw extrusion theory. The variability of temperature, shear rate, pressure and temperature-time history throughout a screw extruder makes it nearly impossible to use screw extruders to determine rheological or thermodynamic properties of doughs during extrusion. Only in the case of major simplifying assumptions (i.e. Newtonian fluid flow, isothermal or adiabatic flow conditions) can this be done accurately. If Newtonian fluid flow is assumed, considerable error may result for it is a well known fact that plasticated doughs do not behave as Newtonian fluids.

The capillary rheometer has been used as a tool for measuring viscosities of molten polymers over a wide range of shear rates (Pezzin 1962; Einhorn and Turetsky 1963; and Zahler and Murfitt 1963). Pezzin (1962) has used a capillary rheometer to obtain a series of rheological data on polystyrene and other molten polymers. He concluded that such data can be correlated with behavior of polymers in the manufacturing process.

Therefore, a capillary rheometer should provide an adequate means of measuring certain rheological properties of plant-protein materials (mainly flour doughs) operating under conditions similar to those encountered in cooking extruders. The amount of shear energy converted to heat via viscous dissipation will be considerably less and therefore must be replaced with direct heating. Preliminary experimental investigation by the authors has revealed that by substituting conductive heat transfer for viscous shear heating, a texturized protein extrudate can be produced (using a capillary rheometer) that is comparable in texture and density to that of a simple screw extruder product. Hence, a capillary rheometer should serve as a useful laboratory extruder for studying rheological and thermodynamic properties of plant-protein materials subjected to typical extrusion operating conditions.

However, conventional testing procedures for using capillary rheometers to obtain non-Newtonian rheological data require a large number of series of tests when several variables such as temperature,

shear rate, moisture content, and temperature-time history are being studied. Therefore, a simple method is needed for minimizing the number of tests required for obtaining rheological data of plant-protein doughs when using a capillary rheometer.

The objectives of this research were:

(1) To design and construct a simple capillary ram food extruder for studying rheological and thermodynamic properties of doughs during extrusion; (2) to develop a mathematical model of such an extruder which would reduce the required number of tests for obtaining a complete set of rheological data, and (3) to experimentally validate the model using a commercial defatted soy-flour.

### DESIGN AND CONSTRUCTION OF CAPILLARY EXTRUDER

Initially, a standard 0.9525-cm diameter capillary rheometer similar to the Instron capillary rheometer was tested as a possible capillary ram extruder. However, experimental tests revealed that filling problems existed with the barrel when high moisture flours were being used. It appeared that bridging was responsible for plugging the barrel resulting in incomplete filling and uneven lengthy filling times. As a result, a redesign of the capillary rheometer was necessary to obtain a larger diameter barrel.

An inside diameter of 1.5 cm was found to be satisfactory with a minimum and consistent loading time of 10 sec. A 1.5 cm inside diameter stainless-steel capillary extruder barrel of length 40.64 cm was constructed with an outside diameter of 3.33 cm. Three thermocouple wells were drilled and installed at the location indicated in Fig. 1. Tips of the shielded thermocouples were held firmly in contact with the bottom of the wells. The bottom of each well was milled to a distance of not more than 0.30 cm from the inside barrel wall. It was assumed that barrel temperature at this point was very near that of the inside wall. Ten 100 watt band heaters were placed on the barrel as indicated in Fig. 2. Capillary dies were held in place by use of a machined lock nut. A stainless-steel 1.27 cm diameter plunger rod was constructed and equipped with a 2.54 cm long teflon plunger head. This teflon plug was used to improve sealing characteristics and minimize barrel wall erosion and drag. The leading surface of the plug was concave at a 45° angle to improve sealing when pressure was applied.

To ensure perfect alignment of the plunger with the barrel, the plunger rod was attached to the load cell via a swivel bearing while the barrel assembly was also secured in a support stand with a swivel bearing. The support stand was bolted to the base of a Tinius-Olsen Load Testing

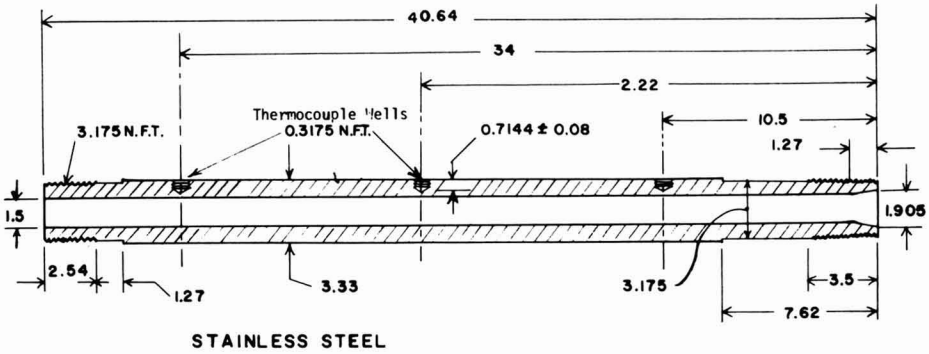


FIG. 1. SCHEMATIC OF CAPILLARY EXTRUDER BARREL

All dimensions in cm.

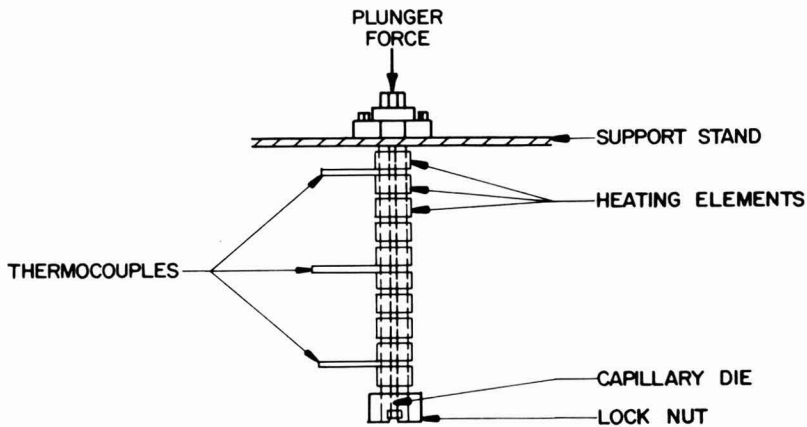


FIG. 2. CAPILLARY EXTRUDER BARREL SHOWING LOCATION OF BAND HEATERS AND THERMOCOUPLES

machine. After installation of thermocouple wells and band heaters, the barrel assembly was wrapped with 8 cm of high temperature-resistant insulation.

### THEORETICAL DEVELOPMENT OF MATHEMATICAL MODEL

Dimensional analysis was used to develop various meaningful dimensionless parameters which were used in developing a mathematical model of the previously described capillary ram extruder. Listed in Table 1 are

Table 1. List of pertinent quantities

Symbol	Description	Dimensions
<b>Initial Flour</b>		
<b>Properties</b>		
(1) MC	Flour moisture content, % wet basis	---
(2) NP	Percentage by weight of Nitrogen, % in dry flour	---
(3) PSP	Percent by weight of soluble protein	---
(4) $\rho_i$	Initial dry flour density (bulk)	ML <sup>-3</sup>
(5) $C_p$	Specific heat of dry flour	L <sup>2</sup> T <sup>-1</sup> $\theta$ <sup>-2</sup>
(6) k	Thermal conductivity of dry flour	MLT <sup>-1</sup> $\theta$ <sup>-3</sup>
(7) $T_i$	Initial temperature of flour (absolute)	T
(8) $M_f$	Mass of flour used in the extrusion process	M
<b>Extrusion</b>		
<b>Properties</b>		
(9) $\theta_{pe}$	Pre-Extrusion cooking time at T= $T_p$	$\theta$
(10) $\theta$	Time after beginning extrusion	$\theta$
(11) $\rho_p$	Pre-Extrusion cooking density (bulk)	ML <sup>-3</sup>
(12) $T_p$	Temperature of inside barrel wall and die during pre-extrusion cooking and extrusion (absolute)	T
(13) $V_p$	Velocity of plunger head during extrusion	L $\theta$ <sup>-1</sup>
(14) $\Delta P$	Pressure applied (measured) to the plunger head during the extrusion process	ML <sup>-1</sup> $\theta$ <sup>-2</sup>
<b>Extruder</b>		
<b>Variables</b>		
(15) $D_b$	Inside diameter of extruder barrel	L
(16) $D_d$	Inside diameter of extruder die	L
(17) $L_d$	Length of extruder die	L
(18) $\alpha$	Die entrance angle, radians	---

the pertinent quantities (variables) of the extruder and plant-protein substance considered in this study. It was understood that not all the variables which affect extrusion performance of a high protein dough are well established and hence the defatted flour properties listed are only those thought to be the most important to this research. However, the same modeling procedure will apply regardless of the number of pertinent quantities used.

Murphy (1950) states that the number of pertinent variables required for a complete dimensional analysis is equal to the number of significant primary variables plus one secondary variable (the dependent variable). For efficient modeling, Murphy (1950) indicates that each primary

variable should be independent of all other variables included. Therefore, if a physical quantity can be completely described in terms of the included primary quantities, then it need not be necessary to include that dependent quantity.

Inspection of Table 1 reveals that no rheological properties of the material, either in the initial or processing state, have been included. It is assumed that all rheological properties of the material undergoing extrusion can be completely described as a function of the material and extruder independent primary variables already listed. Therefore, as indicated in the previous discussion, it is not necessary to include rheological properties of the material if it is assumed that they may be described as a function of the listed variables. For example, shear viscosity may be a function of temperature, shear rate, time-temperature history, etc. which can all be described in terms of the listed variables in Table 1 for a specific material.

It is extremely important to note that the dimensional analysis modeling procedure presented in this study is valid only for a specific material. In other words, model equation constants as presented later will have to be experimentally determined for each new or different material.

The pressure drop,  $\Delta P$ , through the extruder is considered the measured (secondary) dependent quantity of the remaining variables in Table 1. It would be desirable to develop a relationship of the form

$$\Delta P = F(\text{MC}, \text{NP}, \text{PSP}, \rho_i, C_p, k, T_i, M_r, \theta_{pe}, \theta, \rho_p, T_p, V_p, D_b, D_d, L_d, \alpha) \quad (1)$$

It can be shown that if Equation (1) is differentiable over the range of variables considered then  $\Delta P$  may be expressed as the following:

$$\Delta P = C_1 (\text{MC})^{a_1} (\text{NP})^{a_2} (\text{PSP})^{a_3} (\rho_i)^{a_4} \dots (\alpha)^{a_n} \quad (2)$$

where  $a_i$ 's are experimentally determined constants, and

$C_1$  is a dimensionless coefficient that may be a function of the variables MC, NP, . . . .

Equation (1) can now be reduced according to Buckingham's Pi Theorem which states that the number of dimensionless and independent quantities required to express a relationship among the variables of any phenomenon is equal to the number of quantities involved, minus the number of dimensions in which those quantities may be measured (Murphy 1950). For this study there are 4 dimensions (mass (M), length (L), temperature (T) and time ( $\theta$ )), and 18 variables, therefore there are  $18-4=14$  independent dimensionless quantities required to adequately

describe the capillary extrusion phenomenon for a particular substance.

The independent dimensionless quantities, Pi ( $\pi$ ) terms, developed for this model are listed in Table 2. There are other possible sets of dimensionless and independent  $\pi$  terms. With 14  $\pi$  terms in Table 2, Equation (1) may be reduced as

$$\pi_{12} = F(\pi_1, \pi_2, \pi_3, \pi_4, \pi_5, \pi_6, \pi_7, \pi_8, \pi_9, \pi_{10}, \pi_{11}, \pi_{13}, \pi_{14}) \quad (3)$$

which can also be written in the form

$$\pi_{12} = C_2 \pi_1^{c_1} \pi_2^{c_2} \pi_3^{c_3} \pi_4^{c_4} \dots \pi_{11}^{c_{11}} \pi_{13}^{c_{13}} \pi_{14}^{c_{14}} \quad (4)$$

where  $c_i$ 's are constants and  $C_2$  is a dimensionless quantity that may be a function of  $\pi_1, \pi_2, \pi_3, \dots, \pi_{14}$ . Equation (2) has 18 unknowns ( $a_1, a_2, \dots, a_{17}$  and  $C_1$ ) while Equation (4) has 14 unknowns. This reduction of 4 variables can result in considerable savings of experimentation time especially when replications are required. However, probably the more important aspect of Equation (4) is the ability to group physical variables into meaningful dimensionless quantities that may aid in understanding variable effects as grouped.

The  $\pi_1, \pi_2, \pi_3$ , and  $\pi_4$  terms of Table 2 are simply equal to MC, NP, PSP and  $\alpha$  respectively. The  $\pi_5, \pi_6, \pi_7, \pi_8$  and  $\pi_{14}$  terms are ratios of specific variables as indicated in Table 2.  $\pi_9$  is analogous to the Fourier number ( $F_o$ ) which is defined as the ratio of heat transfer conducted through the flour to the rate of heat stored in the flour column.  $\pi_{10}$  can be shown to be equal to an index of the ratio of total extrusion time to the pre-extrusion cook time.  $\pi_{11}$  is analogous to the Péclet (Pé) number or product of Reynolds (Re) times the Prandtl number (Pr). In this case, The Pé(RePr) number represents the ratio of the rate of heat being convected from the barrel wall to the flow of heat by conduction through the flour.  $\pi_{12}$  is the measured  $\pi$  term and is analogous to the Euler number (Eu) which is defined as the ratio of pressure forces to inertial forces.  $\pi_{13}$  is a term which represents the flour volume in terms of equivalent barrel diameters in height to which the barrel is filled with flour.

Equation (4) is the general model of the capillary extruder (for a particular substance) when the pertinent quantities in Table 1 are considered. The general model may also be written as

$$\begin{aligned} \Delta P = C_2 \rho_p V_p^2 (MC)^{c_1} (NP)^{c_2} (PSP)^{c_3} (\alpha)^{c_4} \left(\frac{T_p}{T_i}\right)^{c_5} \left(\frac{\rho_p}{\rho_i}\right)^{c_6} \left(\frac{L_d}{D_d}\right)^{c_7} \\ \cdot \left(\frac{D_b}{D_d}\right)^{c_8} (F_o)^{c_9} \left(\frac{M_f}{\rho_p D_b^2 V_p \theta_{pe}}\right)^{c_{10}} (RePr)^{c_{11}} \\ \left(\frac{M_f}{\rho_p D_b^3}\right)^{c_{13}} \left(\frac{\theta}{\theta_{pe}}\right)^{c_{14}} \end{aligned} \quad (5)$$

Table 2. List of independent dimensionless  $\pi$ -terms

$\pi$ -Term	Description
$\pi_1 = MC$	Flour moisture content, % wet basis
$\pi_2 = NP$	Percent Nitrogen
$\pi_3 = PSP$	Percent soluble protein
$\pi_4 = \alpha$	Die entrance angle
$\pi_5 = (T_p/T_i)$	Ratio of extrusion temperature to initial flour temperature
$\pi_6 = (\rho_p/\rho_i)$	Ratio of pre-extrusion cooking bulk density to the initial dry flour bulk density
$\pi_7 = \left(\frac{L_d}{D_d}\right)$	Length-to-diameter ratio of extruder die
$\pi_8 = \left(\frac{D_b}{D_d}\right)$	Ratio of barrel-to-die diameter
$\pi_9 = \left(\frac{\theta_{pe}k}{C_p\rho_p D_b^2}\right) = (F_o)$	Fourier number: Ratio of rate of conduction of heat to rate of heat storage
$\pi_{10} = \left(\frac{M_f}{\rho_p D_b^2 V_p \theta_{pe}}\right)$	Index of ratio of <i>total</i> extrusion time to the pre-extrusion cook time
$\pi_{11} = \left(\frac{\rho_p D_b^2 V_p C_p}{D_d k}\right) = (RePr)$	Reynolds times Prandtl number: Ratio of heat transfer by convection to that by conduction
$\pi_{12} = \left(\frac{\Delta P}{\rho_p V_p^2}\right) = (Eu)$	Euler number: Ratio of pressure force to inertia force
$\pi_{13} = \left(\frac{M_f}{\rho_p D_b^3}\right)$	Index of Ratio of Total flour volume to a barrel volume of length $D_b$
$\pi_{14} = (\theta/\theta_{pe})$	Ratio of extrusion time to pre-extrusion cook time

Murphy (1950) gives two possible methods of determining the coefficients in Equation (4). One method involves multiplication of component equations to obtain a general equation. Component equations are defined as relationships involving the dependent  $\pi$  term ( $\pi_{12}$ ) and one other independent  $\pi$  term ( $\pi_i$ ) while holding all other  $\pi$  terms constant ( $\bar{\pi}$ 's). Equation (6) is one component equation for the model presented in Equation (4).

$$\pi_{12} = F_1(\pi_1, \bar{\pi}_2, \bar{\pi}_3, \bar{\pi}_4, \dots, \bar{\pi}_{14}) \quad (6)$$



where the bar denotes constant values. The series  $\pi_1, \pi_2, \pi_3, \dots, \pi_{14}$  denotes all  $\pi$  terms except  $\pi_{12}$  which is the dependent  $\pi$ -term.

For practical purposes of reducing the complexity of Equation (4) and, hence, the number of experimental tests, some of the  $\pi$  terms were held constant throughout this study. When using Equation (4) for obtaining a  $\Delta P$  function to be used in developing a rheological material function, a particular material will have constant  $\pi_2$  and  $\pi_3$  terms. Also, it would not be necessary to vary  $\pi_4$  or  $\pi_{13}$  if one were interested in obtaining rheological data. This yields the following extrusion model for a specified material.

$$\pi_{12} = C_3 \pi_1^{c_1} \pi_5^{c_5} \pi_6^{c_6} \pi_7^{c_7} \pi_8^{c_8} \pi_9^{c_9} \pi_{10}^{c_{10}} \pi_{11}^{c_{11}} \pi_{14}^{c_{14}} \quad (7)$$

$$\begin{aligned} \text{or} \\ \Delta P = C_3 \rho_p V_p^2 (MC)^{c_1} \left( \frac{T_p}{T_i} \right)^{c_5} \left( \frac{\rho_p}{\rho_i} \right)^{c_6} \left( \frac{L_d}{D_d} \right)^{c_7} \left( \frac{D_b}{D_d} \right)^{c_8} \\ (F_o)^{c_9} \left( \frac{M_f}{\rho_p D_b^2 V_p \theta_{pe}} \right)^{c_{10}} (\text{RePr})^{c_{11}} \left( \frac{\theta}{\theta_{pe}} \right)^{c_{14}} \end{aligned} \quad (8)$$

In this study, the terms  $\pi_5, \pi_6, \pi_9, \pi_{10}, \pi_{11}$  and  $\pi_{14}$  were also assumed constant in order to minimize the number of tests required. The effect of these assumptions on the model is unknown, but will reduce the number of experimental tests required to illustrate the model and derivation procedure presented earlier. Thus, the final model for the data collected in this study was of the form

$$\pi_{12} = C_4 \pi_1^{c_1} \pi_7^{c_7} \pi_8^{c_8}$$

or

$$\Delta P = C_4 \rho_p V_p^2 (MC)^{c_1} \left( \frac{L_d}{D_d} \right)^{c_7} \left( \frac{D_b}{D_d} \right)^{c_8} \quad (9)$$

where  $C_4$  may be a dimensionless variable depending upon the values of the constant  $\pi$  terms.

It is important to note that Equation (9) is not a rheological material

function. It is simply a model equation describing the pressure drop as a function of the listed variables for a particular material during capillary extrusion. The model, once validated, may be used for generating  $\Delta P$  data that can be used in calculating rheological data for a substance.

### EXPERIMENTAL PROCEDURE AND MATERIALS

Table 3 is the experimental design used in this study. Three replications of each test combination were conducted. Design values of the  $\pi_7$  and  $\pi_8$  terms in Table 3 were obtained by using the following set of dies; diameter = 1.5875 mm with a length of 6.35 mm, diameter of 3.175 mm with lengths of 3.175 mm, 12.7 mm, and 25.4 mm, and diameter of 6.35 mm with a length of 25.4 mm. Table 4 contains values of each variable held constant in this study.

Table 3. Experimental design

Test Number	$\pi_1$ (MC)	$\pi_7$ ( $L_d/D_d$ )	$\pi_8$ ( $D_b/D_d$ )	$\pi_{12}$ ( $\Delta P/V_p^2 \rho_p$ )
1	27	4	4.72	Measure
<sup>a</sup> 2	32	4	4.72	Measure
3	36	4	4.72	Measure
4	32	1	4.72	Measure
<sup>a</sup> 5	32	4	4.72	Measure
6	32	8	4.72	Measure
7	32	4	2.36	Measure
<sup>a</sup> 8	32	4	4.72	Measure
9	32	4	9.45	Measure

<sup>a</sup>Identical tests

$\pi_1 = 32$ ,  $\pi_7 = 4$ ,  $\pi_8 = 4.72$

Table 4. List of constant variables and their values

Variable	Value	Variable	Value
$\rho_i$	0.52 g/cm <sup>3</sup>	$T_i$	298 °K
$C_p$	1.8 J/g— °K (estimated)	$T_p$	450 °K
$k$	0.45 w/m— °K (estimated)	$\theta_{pe}$	90 sec
$\rho_p$	1.15 g/cm <sup>3</sup>	$\theta$	9 sec
$M_f$	25 g	$D_b$	15 mm
		$\alpha$	3.14 rad.

Inspection of the  $\pi$  terms in Table 2 reveals that all  $\pi$  terms not includ-

ed in Equation (9) will remain constant with the exception of  $\pi_{11}$ . The  $\pi_{11}$  term may be held constant by varying the plunger velocity ( $V_p$ ) by the same proportion as,  $D_d$ , the die diameter. However, if  $V_p$  is varied, then  $\pi_{10}$  will change. A new term,  $\pi'_{10}$ , that would remain constant with varying  $V_p$  and  $D_d$ , was therefore created to replace  $\pi_{10}$ .

$$\text{Let } \pi'_{10} = \frac{M_f C_p}{D_b k \theta_{pe}} \tag{10}$$

Below is a list of constant  $\pi$  terms calculated for this study using values from Table 4.

$\bar{\pi}_2 = 8.8$	$\bar{\pi}_3 = 38.5$	$\bar{\pi}_4 = 3.14$
$\bar{\pi}_5 = 1.41$	$\bar{\pi}_6 = 2.21$	$\bar{\pi}_9 = F_o = 0.087$
$\bar{\pi}'_{10} = 74$	$\bar{\pi}_{11} = \text{RePr} = 2282$	$\bar{\pi}_{13} = 6.44$
$\bar{\pi}_{14} = 0.10$		

Since  $V_p$  was set proportional to  $D_d$  to maintain a constant RePr term, the three values of  $V_p$  used were 1.75, 3.5 and 7 mm/sec.

A commercial defatted soy flour (200-W, Central Soya) was used for conducting the experimental tests. The soy flour's percent nitrogen (NP) and percent soluble protein (PSP) were found to be 8.8 and 38.5 respectively. The initial moisture content (MC) of this flour was found to be 9% wet basis. Moisture samples were obtained by oven drying at 90°C for 48 hr. A mixing blender was used to hydrate 150-g samples of the 9% flour to the design moisture content. The hydrated samples were stored (all samples of each MC were combined) at 5°C for 48 hr to allow proper moisture absorption and equilibrium to be obtained. No attempt was made to determine the biological effect of such storage conditions, but only to maintain constant conditions between test samples. Samples were removed from the 5°C environment 8 hr prior to testing allowing them to reach an ambient room temperature of 25°C.

The capillary extruder barrel was heated to a constant barrel wall temperature of 450°K and maintained at that temperature throughout the testing period. The warm up period was extended to 2 hr to ensure that all heat loss components had reached steady-state conditions. Tests were conducted by loading the pre-heated barrel with 25 g of hydrated defatted soy flour and immediately preloading the flour sample to a pressure of 2.6 MPa which resulted in a pre-extrusion cooking density ( $\rho_p$ ) of 1.15 g/cm<sup>3</sup>. Preliminary tests had revealed that this was the pressure required to cause the dough to just begin to enter the capillary die. Pre-loading was performed with a plunger velocity of 8.68 mm/sec which was the maximum speed of the testing machine. This resulted in a constant

pre-loading time of 32 sec. The pre-extrusion cooking time,  $\theta_{pe}$ , started when the sample was placed in the heated barrel and therefore included the pre-loading time. After pre-loading the sample to a density of 1.15 g/cm<sup>3</sup>, the dough was allowed to cook until a pre-extrusion cooking time of 90 sec had expired. The sample was then extruded at the design plunger velocity and the load cell force-time curve was recorded and used for calculating  $\Delta P$  at time  $\theta$ . After each extrusion process, the die was removed and cleaned and any residue existing in the barrel was removed. Drag of the teflon plug with an empty barrel was found to be negligible when compared to force readings being obtained during testing (less than 1% of reading).

### RESULTS AND DISCUSSION

The effect of moisture content on  $\pi_{12}$  is shown in Fig. 3. A least-squares linear-regression analysis was used to obtain a best fit equation of the data on log-log coordinates. The data appear to fit an equation of the required form,  $\pi_{12} = A_1 \pi_1^{b_1}$  (Murphy 1950). The component equation obtained yielded an  $R^2$  of 0.978.

$$\pi_{12} = 8.126 \times 10^{12} (\pi_1)^{-2.81}; \bar{\pi}_7 = 4, \bar{\pi}_8 = 4.72 \quad (11)$$

Capillary rheometry theory reveals that total extrusion pressure should be linearly related to die  $L/D$  for constant shear rates. The Philippoff-Gaskin equation (Einhorn and Turetsky 1963) expresses  $\Delta P$  as linearly related to  $(L/D)_{die}$  with the slope being equal to four times the true shear stress at the capillary wall. The data points plotted in Fig. 4 are for a constant  $V_p$  and  $D_d$  (constant shear rate) and indicate that  $\pi_{12}$  is linearly related to  $L/D$ . Since  $\pi_{12} = \Delta P / \rho_p V_p^2$  and  $\rho_p$  and  $V_p$  are constant, this implies  $\Delta P$  is also linearly related to die  $L/D$  as theory implies. The linear fit was obtained with an  $R^2$  of 0.967. The equation in Fig. 4 does not conform with the required  $\pi_{12} = A_7 \pi_7^{b_7}$  equation form and therefore the following transformation was used to create a new  $\pi$  term ( $\pi'_7$ ).

$$\pi'_7 = (287.4 + 59.43 \pi_7) \times 10^6 \quad (12)$$

therefore

$$\pi_{12} = (1) (\pi'_7)^{(1)}; \bar{\pi}_7 = 32, \bar{\pi}_8 = 4.72 \quad (13)$$

Equation (13) does meet the required equation form and was used as the corresponding component equation.

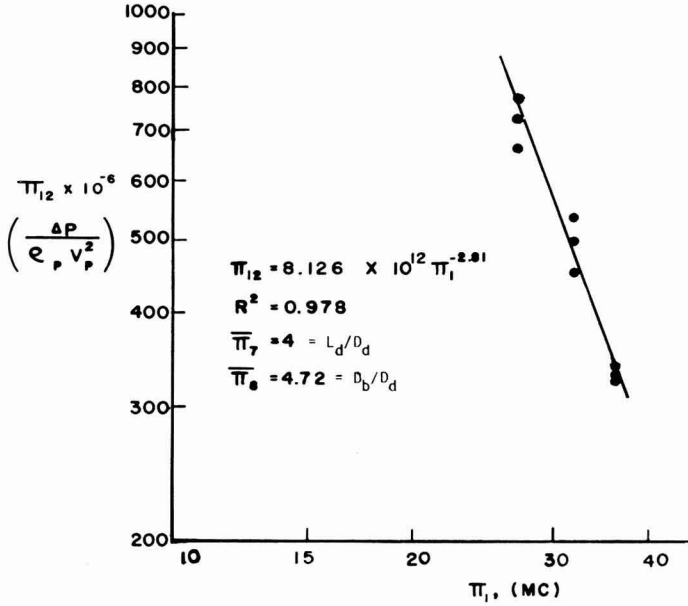


FIG. 3. EFFECTS OF DOUGH MOISTURE CONTENT ON EXTRUDER PRESSURE LOSS

Figure 5 indicates the relationship of  $D_b/D_d$  to  $\pi_{12}$ . The best fit least-squares regression equation obtained for log-log coordinates is

$$\pi_{12} = 6.51 \times 10^6 (\pi_8)^{2.73}; \pi_1 = 32, \pi_7 = 4 \tag{14}$$

with an  $R^2$  of 0.996.

The general model, Equation (15), was obtained by the method of multiplication of component equations (Murphy 1950).

$$\pi_{12} = 224.9 \times 10^6 (\pi_1)^{-2.81} (\bar{\pi}_7) (\pi_8)^{2.73} \tag{15}$$

or

$$\Delta P = 13.36 \times 10^9 \rho_p V_p^2 (MC)^{-2.81} \left( \frac{D_b}{D_d} \right)^{2.73} \left( 4.837 + \frac{L_d}{D_d} \right) \tag{16}$$

for  $27 \leq \pi_1 \leq 36$ ,  $1 \leq \pi_7 \leq 8$ ,  $2.36 \leq \pi_8 \leq 9.45$

Equation (15) was used to obtain predicted values of  $\pi_{12}$  which are plotted in Fig. 6 against observed values. The general model fit the observed data with an  $R^2$  of 0.974 and is valid for all  $\pi_1$ ,  $\pi_7$  and  $\pi_8$  values within the experimental ranges.

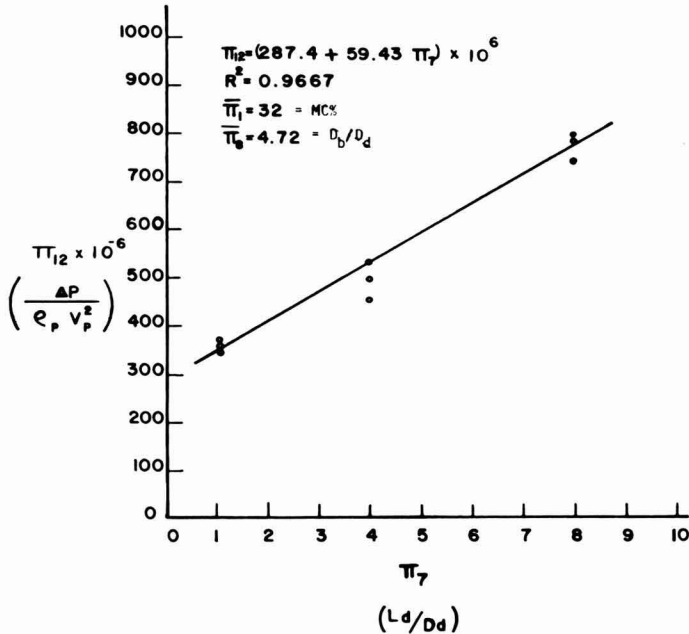


FIG. 4. RELATIONSHIP OF CAPILLARY DIE L/D TO EXTRUDER PRESSURE LOSS.

Equation (16) can be used to generate  $\Delta P$  data for varying moisture contents and flow rates ( $Q$ ). In most cases, the effects of temperature ( $T_p/T_i$ ) and time ( $\theta/\theta_{pe}$ ) would also have been measured. In generating  $\Delta P$  data, it will be necessary to generate values of  $\Delta P$  for various  $(L/D)_d$  ratios with constant  $D_d$  and  $V_p$  to obtain data that may be used to calculate Couette's die entrance correction factor McKelvey *et al.* 1957). This factor can then be used to obtain values of true shear stress at the capillary wall. After obtaining these sets of data, plots may be made to obtain values for the Rabinowitch correction (Rabinowitch 1929). With these corrections applied, viscosity calculations can be made and used to develop a material viscosity function of the flour dough for various ranges of shear rate, moisture content, temperature, time, etc.

Additional data were obtained to test the above proposed method of obtaining rheological data. Equation (16) was used to generate  $\Delta P$  vs  $Q$  data from which viscosity calculations were made using the procedure described above. Actual  $\Delta P$  versus  $Q$  data were obtained by experimental observation and viscosity values calculated from these data. Figure 7 is a plot of predicted vs observed viscosities for a defatted soy-flour cooked 90 sec at 177°C. The observed data fit the predicted with an  $R^2$  of 0.904

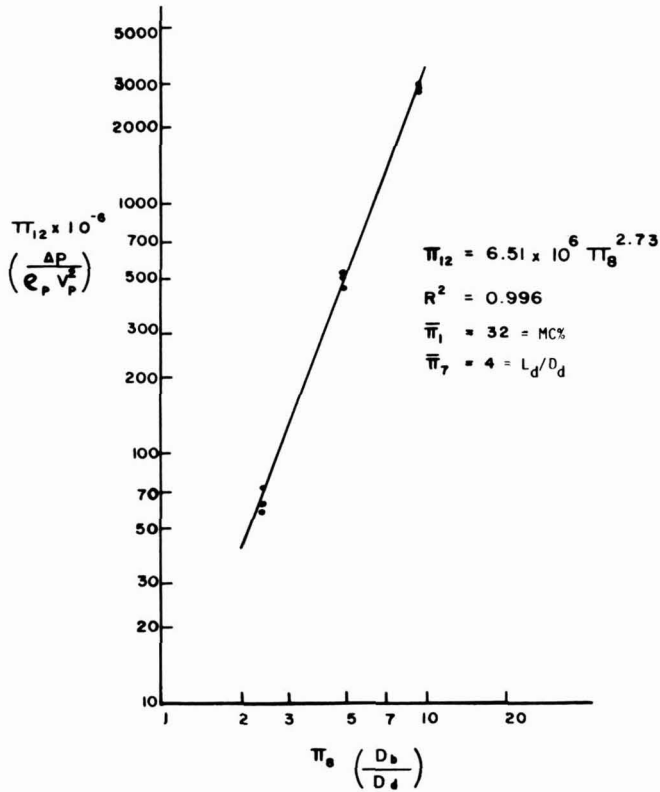


FIG. 5. EFFECT OF BARREL-TO-DIE DIAMETER RATIO ON  $\pi_{12}$  FOR CONSTANTS MC AND DIE L/D

thus indicating a reasonable degree of accuracy of the proposed procedure.

$\Delta P$  data obtained for various ranges of  $T_p/T_i$ ,  $\rho_p/\rho_i$ ,  $\theta/\theta_{pe}$ , and MC can be used to study the thermodynamic reactions occurring during extrusion and possibly relate these to two-phase extrusion heat and mass transfer phenomena.

### CONCLUSIONS

A capillary ram plant-protein extruder can be designed and used for studying rheological and thermodynamic properties of doughs. Dimensional analysis and Buckingham's Pi Theorem may be used to develop a mathematical model equation of which the constants may be determined

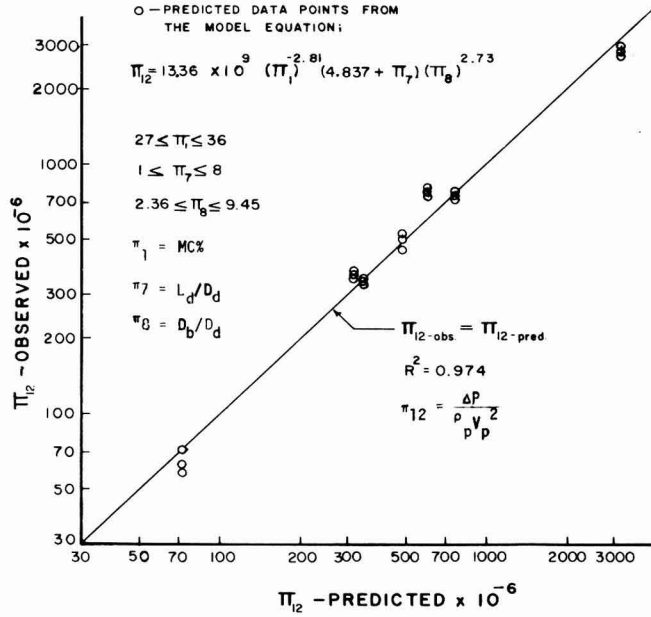


FIG. 6. RESULTS OF EXPERIMENTAL VALIDATION OF MODEL EQUATION

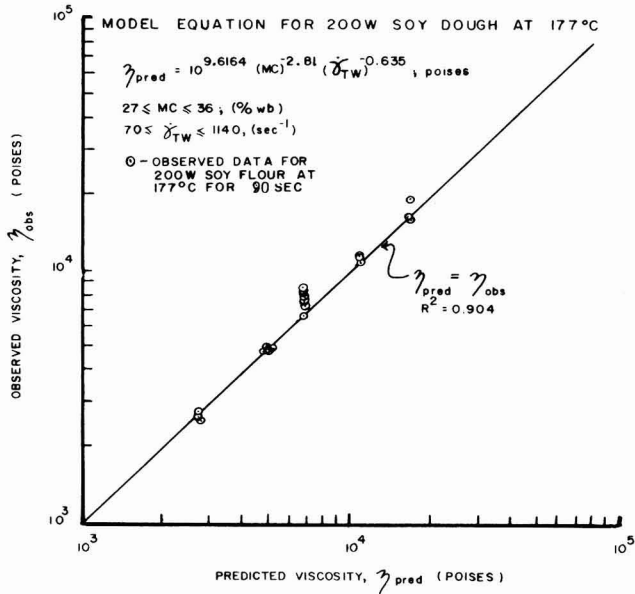


FIG. 7. RELATIONSHIP OF OBSERVED SHEAR VISCOSITY WITH THE PREDICTED VALUES USING MODEL-GENERATED  $\Delta P$  DATA



with minimal experimentation. It is proposed that this equation can be used to generate numerous sets of  $\Delta P$  versus  $Q$  data for various operating conditions. These data can be used to develop and study rheological material functions and thermodynamic models of various plant-protein doughs during cooking extrusion.

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# VISCOSITY OF AN INTERMEDIATE MOISTURE DOUGH

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## ABSTRACT

*A simple model was used to correlate extrusion dough viscosities with moisture, temperature and shear rate. The parameters were fit to the model for a pregelatinized corn flour dough. The effect of two additives, sodium stearyl-2-lactylate and a diacetyltartaric acid ester of monoglycerides, were also explored using the pregelatinized corn flour system. Moisture, extrusion temperature and sodium stearyl-2-lactylate levels were correlated to the viscosity of the pregelatinized corn flour dough.*

## INTRODUCTION

Harman and Harper (1974) found that the equations for flow rate and torque input to an extrusion screw's metering section, which had been derived for plastics extruders, could be applied to food extruders with sufficient accuracy to be useful for engineering design and scale up. The accurate application of these equations depends upon knowing the viscosity of the food dough. The viscosity of most food doughs is dependent upon shear rate, temperature and moisture. Food systems may be affected by the addition of emulsifying agents. These agents may also affect the viscosity of a dough system. The stimulus for this work was the lack of data on the viscosity of the intermediate moisture doughs from pregelatinized corn flour as affected by: shear rate, temperature, and moisture. The addition of two emulsifying agents, sodium stearyl-2-lactylate (SSL) and a diacetyltartaric ester of monoglycerides (DTEMMD), to the pregelatinized corn flour and water dough system was also studied.

The viscosity of the dough, which is an important parameter in the flow rate and torque equations for extruders, is dependent upon the moisture content, shear rate and temperature for each dough composi-

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tion. Although a number of papers have been written on the viscosity of high moisture (> 70%) food systems, only one paper was found on the viscosity of intermediate moisture doughs. Harper, Rhoades and Wanning (1971) used the following model to describe viscosity changes about a known base point for cooked cereal dough:

$$\eta = C_1 (du/dr)^{C_2} e^{C_3/T} e^{C_4MWB} \quad (1)$$

These authors used a straight tube viscometer to determine the viscosity of an 80% corn grits and 20% oat flour dough. The final equation with coefficients estimated using multiple regression technique was:

$$\eta = 3.76 \times 10^4 (du/dr)^{-0.49} (e^{2.480/T}) (e^{-0.079MWB}) \quad (2)$$

The correlation coefficient  $r = 0.96$

Their model reduced to the power law model at conditions of constant moisture and temperature.

### MATHEMATICAL MODEL

The model used for this work is derived from the power law, Eyring's kinetic theory of liquids and the semi-empirical mixing rule. The Ostwald-de Waele model, also known as the power law, is written as:

$$\tau_{yx} = \eta_p \left| \frac{du}{dy} \right|^n$$

When  $n$  equals 1, the equation reduces to Newton's law of viscosity with  $\eta_p$  equal to  $\mu$ . For values of  $n$  less than one, the fluid is pseudoplastic; and for  $n$  greater than one, the fluid displays dilatant behavior. Although the power law does not hold over extreme ranges of shear rate, it has been used to model flow behavior for many materials and has found many practical applications (Bird *et al.* 1960).

The Eyring kinetic theory of liquids predicts the relationship between viscosity and temperature;

$$\eta = K e^{b/T} \quad (3)$$

where  $b = \Delta^\circ E/R$ . The effect of moisture content on a liquid can be estimated from the semi-empirical logarithmic mixing rule,

$$\log \eta_{\text{mix}} = \sum_{i=1}^m x_i \log \eta_i \tag{4}$$

with  $x_i$  the mole fraction and  $\eta_i$  the viscosity of the  $i$ th species in the mixture. Use of the logarithmic mixing rule does not allow for interaction of chemical species as may be the case in many food systems. Despite this limitation, the logarithmic mixing rule has been used to describe variations in viscosity about a known viscosity point.

By using the power law which relates shear stress and shear rate for pseudoplastic materials, Eyring's kinetic theory of liquids and the semi-empirical logarithmic mixing law, the following model was written for food dough viscosity:

$$\eta = C_1 (\text{du/dr})^{C_2} e^{C_3/T} e^{C_4 \text{MDB}} \tag{5}$$

or in a linear form,

$$\ln \eta = \ln C_1 + C_2 \ln (\text{du/dr}) + C_3/T + C_4 \text{MDB} \tag{6}$$

This model reduces to the power law at a constant temperature and moisture with

$$\eta_p = C_1 e^{C_3/T} e^{C_4 \text{MDB}} \tag{7}$$

### METHODS

Pregelatinized corn flour (Lora-dex, Krause Milling Co.) was mixed with tap water to the desired moisture content and stored in moisture-tight plastic containers at room temperature for approximately 24 hr allowing the complete hydration of the starch molecules. The characteristics of the pregelatinized flour are given in Table 1. When corn oil was added to the dough, it was mixed into the corn flour in the liquid-solid blender prior to the addition of any water. In the later experiments, several food-emulsifiers were added to the corn dough. These emulsifiers were: Artodan SP 50, SSL, and Panodan DTEMD. These additives were dispersed in the water at 50 to 55 °C and added to the flour in this form. In all cases, just prior to extruding, the dough was particlized in the blender and duplicate moisture samples from each container of dough were taken to estimate the moisture content in each specific experimental run.

Table 1. Lora-dex #804 low density pregelatinized corn flour characteristics

Protein	6.5–8.5%
Fiber	0.4–0.6%
Water	<11.0%
Ash	0.4–0.7%
Fat	<0.6%
Starch	78–80%
Amylose	~20%
Amylopectin	~80%

The particlized dough was manually fed into the Brabender Plasticorder extruder having a 1.90 cm diameter barrel and a 20:1 L/D ratio, to obtain a constant feed rate and to overcome problems of bridging in the feed hopper on the feed section of the screw. A screw with a 3:1 compression ratio was used. The extruder was allowed to operate at least ½ hr prior to extruding samples to assure uniform heating of the screw and to obtain equilibrium conditions. The dough was heated as it passed through the first section of the extruder and was maintained at  $\pm 1^\circ\text{C}$  of the desired experimental run temperature in the metering and die zones of the extruder. This was accomplished by closely monitoring the dough temperature with thermocouples and adjusting the barrel temperature to obtain the desired dough temperature for the run. When the extruder reached steady state, as indicated by constant torque (fluctuation less than 10 on the scale) and pressure readings ( $\pm 6.9 \times 10^4$  Pa), data were recorded and samples were taken following the procedure outlined in Table 2. Two 1-min timed samples were taken and saved to determine the average extruder output rate. Various combinations of the variables — moisture, temperature, fat and additives — were studied as reported in Table 3. Viscosity was calculated by measuring the quantities in the Hagan-Poiseuille equation. The die section of the extruder was used as a straight tube viscometer for calculation of shear rate and shear stress. End effects in the die were corrected using the model presented by Bagley (1957) for the effective shear stress. The effective shear rate was calculated using a technique presented by Moodey (1931) for the expression of shear rate which is independent of fluid properties as modified by Metzner and Reed (1955).

#### EFFECT OF MOISTURE AND TEMPERATURE

Constants in the proposed viscosity model equation (Equation 6) were

Table 2. Order of data collection

---

(1) Record
a. Dough temperatures
b. Pressures
(2) Collect
a. Two 1-min timed samples
(3) Record
a. Dough temperatures
b. Pressures

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Table 3. Combination of extruder variables

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Temperature (°)	Moisture (% Wet Basis)	Fat (% Dry Basis)	Additives (% Dry Basis)	Range of Screw Speeds (rpm)
90	30	0	0	10–100
90	25	0	0	10–100
120	30	0	0	10–100
120	25	0	0	10– 60
120	22	0	0	10– 40
150	25	0	0	40–100
90	30	0	0.5 DTEMD	10–100
90	30	0	1.0 DTEMD	10– 80
90	30	0	0.5 SSL	10–100
90	30	0	1.0 SSL	20– 70
90	30	5	0	20–100
90	30	5	0.5 SSL	20–100
90	30	5	1.0SSL	20–100
90	30	5	0.5 DTEMD	20– 60
90	30	5	1.0 DTEMD	10– 50
90	35	5	0	20–100
90	35	5	0.5 SSL	40–120
90	35	5	1.0 SSL	40–120

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estimated using multiple linear regression. The resulting equation describes the effect of shear rate, moisture and temperature on the viscosity of the pregelatinized corn dough:

$$\eta = 0.0036 (\text{du/dr})^{-0.644} e^{4388/T} e^{-0.101\text{MDB}} \quad (8)$$

All of the variables: shear rate, moisture (dry basis) and temperature, were highly significant ( $p < 0.0001$ ) in the correlation of the dependent variable, viscosity. The resulting equation obtained had a very good fit

with the data ( $r = 0.987$ ). An increase in moisture content from 30% MWB to 35% MWB caused a decrease in viscosity of 40% and an increase in temperature from 90 to 120°C caused a decrease in viscosity of 50%. As the shear rate increases, viscosity decreases,  $\eta \propto (du/dr)^{-0.664}$ . The power law index of 0.356,  $n < 1$ , indicates that the dough system is highly pseudoplastic.

The equation obtained from the regression analysis in this study fits the changes in viscosity due to temperature very well. A plot of the actual data points and the calculated lines from the equation are presented in Fig. 1 for variations due to temperature. The fit indicates that the Eyring model for the temperature depression of viscosity is very helpful in describing the temperature effects on the cereal dough tested.

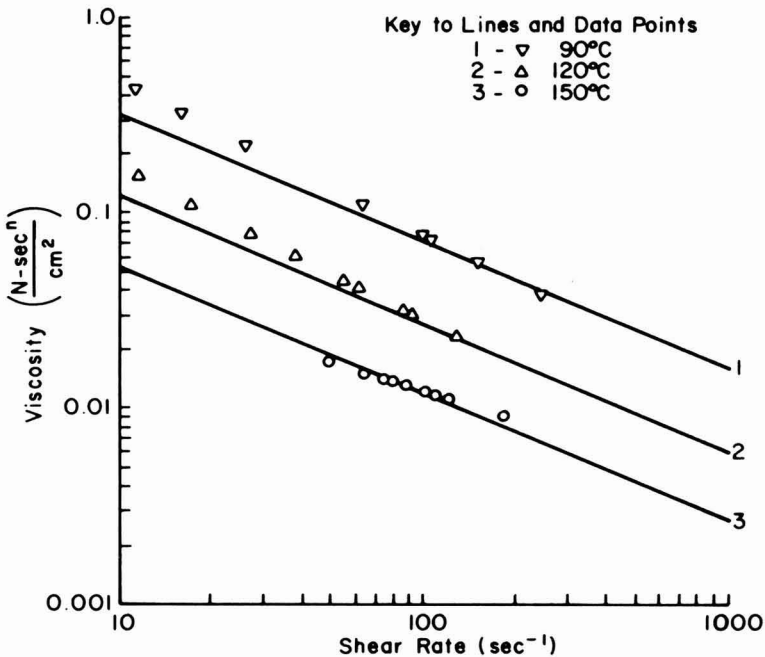


FIG. 1. EFFECT OF TEMPERATURE ON VISCOSITY AT A CONSTANT MOISTURE (25% MWB)

Since the model curves did not fit the data well at low and high shear rates, there appears to be an interaction between the temperature effect and the shear rate. Such an effect was not included in the model because it is not theoretically predicted and because, over the range of shear rates of interest, it is not needed to have a satisfactory fit of the data.

A plot of the actual data points for doughs having varying moisture



contents and corresponding regression lines are presented in Fig. 2. A change in the slope of the data points for different moistures can be observed and may be a reflection of the inadequacy of the logarithmic mixing model to describe viscosity contributions of moisture. For example, the model used does not account for the separate effects of free, bound or absorbed water that might be present in the system. The manner in which water exists in the dough system will undoubtedly affect the viscosity of the dough. The mixing model is based upon the addition of the proportion of a substance present multiplied by its characteristic viscosity. In the case of the dough system studied, the addition of water changes the actual viscosity of the dough by rehydrating the pregelatinized starch. Therefore, a model which accounts for the flour still present, the rehydrated starch and the free, bound and absorbed water may yield a more accurate description of the affect of adding water.

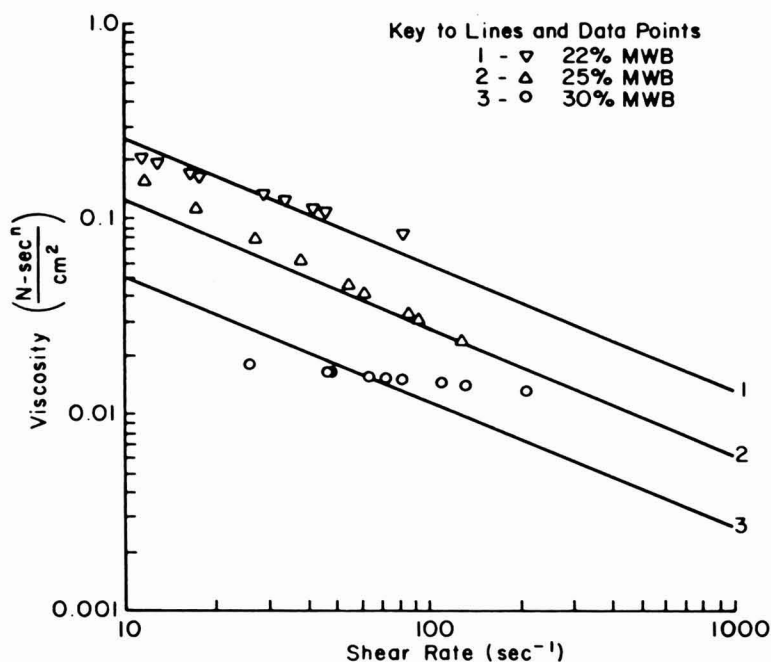


FIG. 2. EFFECT OF MOISTURE CONTENT ON VISCOSITY OF DOUGH AT A CONSTANT TEMPERATURE (120°C)

The dough in this experiment was allowed to rehydrate from 18 to 24 hr. In this experiment, it is not believed that time was a large factor affecting the dough viscosity because of the reproducibility of the viscosity data for varying hydration lengths.

The close fit of regression equations for both the temperature and moisture effects makes the proposed model appear to be a practical and useful method to correlate viscosities even though the quantity of free, bound and absorbed water needs further refinement. Further refinement is warranted if the model is to be applied over a wider range of conditions.

### EFFECTS OF SSL

When shear rate, moisture content (30% and 35% MWB), fat content (0% and 5%), and level of SSL (0, 0.5 and 1.0%) were regressed on viscosity (at a constant extrusion temperature of 90°C) using multiple linear regression, it was found that shear rate, moisture content and SSL had significant effects ( $p < 0.0001$ ) but that the effects of fat were not significant ( $p = 0.613$ ). Therefore, fat was not included in the final correlating equation:

$$\eta = 24.38 (du/dr)^{-0.648} e^{-0.0869MDB} e^{0.286SSL} \quad (9)$$

A good fit of the data was indicated for a value of  $r = 0.986$  in the final equation used to predict viscosity. There is internal consistency with the coefficients for shear rate (0.648) and moisture content ( $-0.0869$ ) being very close to those received in the first correlation reported of shear rate, moisture content and temperature (Equation 8).

The addition of SSL to corn dough increased the viscosity of the mixture regardless of the absence (Fig. 3) or presence (Fig. 4 and 5) of fat. The increase of viscosity with the addition of SSL is clearly seen in Fig. 5. In Fig. 3 it appears that the dough with 1.0% of SSL is less viscous than that with 0.5% SSL. This apparent reversal in trend is actually due to a slight difference in moisture content (29.92% MWB at 0.5% SSL and 31.22% MWB at 1.0% SSL). A similar slight reversal of lines and data points for dough without SSL and with 0.5% SSL can be seen also in Fig. 4. This also is due to a slight difference in moisture contents, 29.67% MWB for dough without SSL and 30.75% MWB for dough with 0.5% SSL.

An unexplained change in slope is noted in Fig. 5 for the data obtained for the corn dough with 0.5% SSL added. It is possible that this is due to experimental error and/or some unknown contamination which occurred during the mixing process. Unfortunately, duplicate data were not obtained for this particular set of conditions.

SSL has an anionic affinity for starch and protein molecules, and has been reported to form an insoluble complex with amylose (Krog 1971; 1973). The hydrophobic end of the SSL groups align themselves around

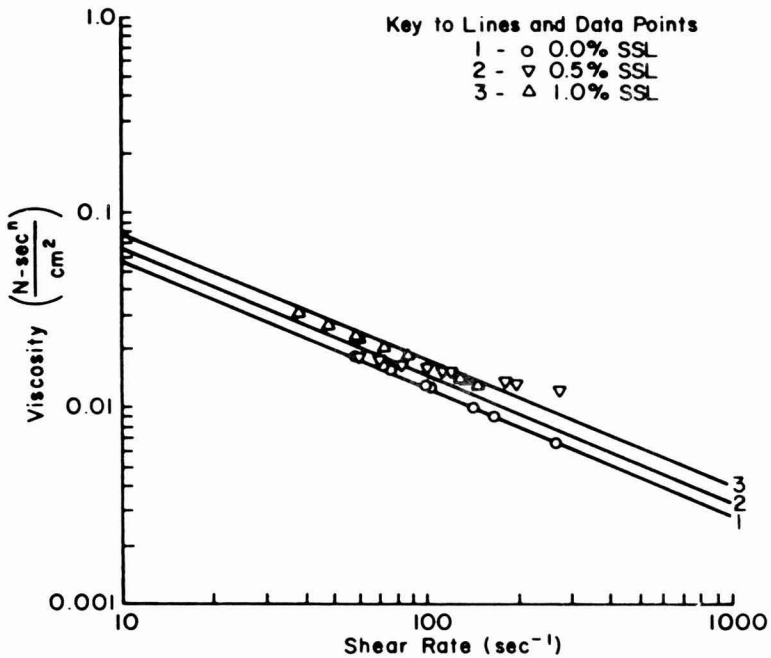


FIG. 3. EFFECTS OF SSL ON VISCOSITY AT 90°C, 30% MWB AND NO FAT ADDED

the starch molecule and the hydrophilic ends are attracted to the water in the system. In this manner, an insoluble complex is formed with the amylose in the corn starch and acts like a rigid coating around the starch molecules. If ungelatinized starch were used, a decrease in viscosity would be expected because the SSL coating on the molecule would prevent water from entering and becoming involved in the gelatinized reaction. But, since pregelatinized corn flour was used, the starch molecule has an increased ability to accept the water prior to the SSL coating the outside of the molecule; therefore, very little effect was seen by the addition of the SSL. In the case where pregelatinized flour is used, the SSL may have several effects on the dough system. The SSL may complex with the starch making the granule more rigid and stabilizing it against breakdown and decrease in viscosity (Krog 1973), or the SSL may tie up some of the free water present in the dough system, so that the water is not available to the system and its tendency to decrease viscosity is not observed. This phenomenon is supported by the observation of the dough characteristics; the addition of SSL made the dough appear to be drier than other doughs with the same moisture content. The addition of fat to this system has an insignificant effect because the major reaction is

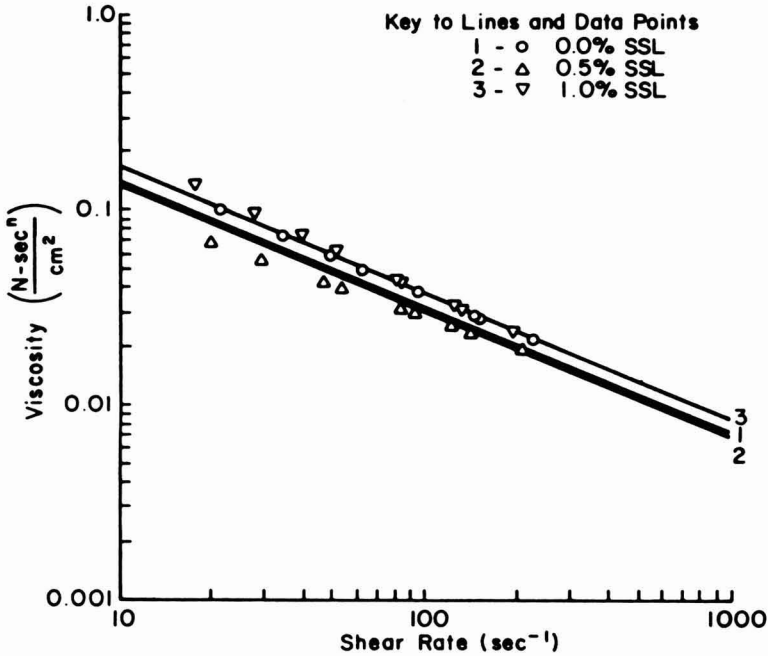


FIG. 4. EFFECTS OF SSL ON VISCOSITY AT 90°C, 30%  
MWB AND 5% FAT

between starch, water and SSL and not between water, fat and SSL. It is possible that since the effect of the fat is much smaller than that of the SSL, the SSL masks the fat's effects.

#### EFFECTS OF DTEMD

The effects of DTEMD (at a constant temperature of 90°C) on viscosity were analyzed treating moisture in three different ways. In the first regression, moisture content of the dough was considered to be a constant because of the very narrow range of moisture content between the dough samples (29.67 to 30.98% MWB), and shear rate, fat content and DTEMD content were independent variables. This regression resulted in an equation where shear rate and fat were significant ( $p < 0.001$ ) but DTEMD was only marginally significant ( $p < 0.04$ ). A second regression was run with moisture content considered an independent variable because in the earlier analysis it was observed that moisture content had a highly significant effect on viscosity. The second set of results showed that the DTEMD level was not significant ( $p = 0.404$ ). The coefficient for

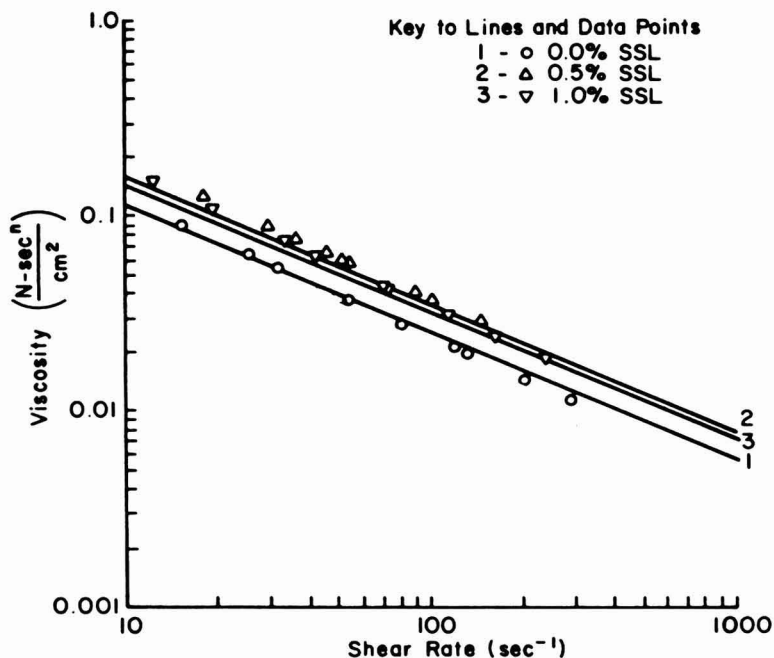


FIG. 5. EFFECTS OF SSL ON VISCOSITY AT 90°C, 35% MWB AND 5% FAT

moisture content in this run was  $-0.183$  which is similar to that found for the moisture content ( $-0.101$ ) in doughs without DTEMD. It was felt that the moisture coefficient estimated by the regression of DTEMD, fat and moisture on viscosity was questionable because of the narrow range of moisture variation. Assuming the moisture effect was similar to the effect estimated in the corn dough without DTEMD, a third regression was performed where the coefficient for moisture was specified at  $-0.1011$  and shear rate, fat and DTEMD content were independent variables regressed against viscosity. In this case, DTEMD was not significant ( $p \leq 0.110$ ), and so it was not included in the final equation:

$$\eta = 52.72 (du/dr)^{-0.717} e^{-0.101MDB} e^{0.059FAT} \quad (10)$$

DTEMD is a diacetyltartaric acid ester of monoglycerides and the monoglyceride portion of the molecule gives DTEMD an affinity for protein. Therefore, DTEMD does not greatly affect the viscosity of the corn flour-water-fat system because it does not interact with the starch present in the system which largely determines the final viscosity. Krog (1973) has shown that DTEMD has little effect on viscosity of starch-water systems and that its effect is highly pH and ion concentration

dependent. DTEMD acts as an emulsifier between protein-water systems. The diacetyltartaric acid portion of the molecule has an affinity for starch, but the monoglyceride portion also gives the molecule an affinity for protein; therefore, a dicetyltartaric acid without the monoglyceride portion might make a more effective additive in a system which has a high starch content.

### CONCLUSIONS

The power law can be used to accurately correlate shear rate effects on the viscosity of intermediate corn doughs. Eyring's theory of liquid viscosity adequately predicts the effect of temperature on the pregelatinized corn dough system. The logarithmic mixing model describes the effects of moisture content sufficiently to be a practical and useful method to correlate moisture content to viscosity over narrow ranges even though the quantity of free, absorbed and bound water is not precisely represented. The additive SSL caused an increase in viscosity, and fat did not significantly affect viscosity when SSL was present while the additive DTEMD did not significantly affect viscosity.

### NOMENCLATURE

C	constant, dimensionless
D	diameter of screw, cm
DTEMD	diacetyltartaric ester of monoglycerides
$du/dr$	shear rate, $\text{sec}^{-1}$
$^{\circ}E$	activation energy, kcal/mole
K	constant in power law
L	length of screw, cm
MDB	moisture dry basis, %
MWB	moisture wet basis, %
n	constant in power law
SSL	sodium stearyl-2-lactylate
T	absolute temperature, $^{\circ}k$
x	mole fraction
$\eta_p$	power law coefficient, $N\text{-sec}^n/\text{cm}^2$
$\eta$	non-Newtonian viscosity, $N\text{-sec}^n/\text{cm}^2$

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# ENGINEERING ANALYSIS OF SOY DOUGH RHEOLOGY IN EXTRUSION

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## ABSTRACT

*Effects of moisture level, temperature and shear rate on the rheological properties of defatted soy dough during extrusion were studied on a Brabender laboratory extruder. Entrance pressure loss through the extruder capillary die as well as viscosity of the cooked and compressed soy dough were expressed as a function of added moisture, shear rate and temperature through multiple regression analysis. Process conditions involved in this study were (a) temperature from 100 to 160°C; (b) added moisture from 22 to 32% and (c) shear rate from 50 to 10,500 sec<sup>-1</sup>. Data were collected at steady state. It was found that in the shear rate range of 50 to 1,000 sec<sup>-1</sup> and temperature range of 100 to 160°C, the viscosity of the cooked and compressed soy dough with 32%, 25% and 22% added moisture ranged from 10,000 to 500 poise and 20,000 to 200 poise and 20,000 to 1,000 poise respectively.*

## INTRODUCTION

Texturization of vegetable protein by extrusion is one of the most common ways to produce intermediates for various meat analogs. This

process utilizes either viscous dissipation heat generated through the rotation of a screw or heat applied through an extruder barrel or both simultaneously.

In traditional plasticating extrusion, solid polymer is fed to the extruder hopper in the shape of pellets, chips, beads or in other forms. The polymer is then conveyed and melted along the channel between the barrel and the screw. Generally, a plasticating screw can be visualized as having 3 functional sections of solid conveying, compression and metering; however, each screw section can perform all 3 functions simultaneously (Chung 1970). Similar to plasticating extrusion, the conventional food extrusion process can also be visualized as having three zones. Protein material, such as soy flour and water, are generally fed to the solid conveying zone at room temperature and then compressed forward down the channel between extruder barrel and screw. This movement creates a large amount of frictional heat which initiates the unfolding of the protein material (Vinogradov and Linnell 1971). The proteinaceous mass is further forced and oriented through extruder die. The process is usually associated with high pressure created by the compression of materials and resistance against the extruder die. The cooked and compressed mass is thrust through a die into the atmosphere. When it emerges from the die, the super heated water flashes off from the extrudate and subsequently creates a porous and laminated structure.

There is limited information available on rheological properties of a proteinaceous mass in extrusion. However, polymer rheology in plasticating extrusion has been extensively studied (Tadmor and Klein 1970; Ballenger *et al.* 1971; Mendelson 1976). Modeling for solid conveying in plasticating extrusion has been reported by Darnell and Mol (1956), Chung (1970), Tadmor and Klein (1970), and Klein (1976). Harman and Harper (1974) performed their modeling study by feeding pregelatinized corn flour with water. It is now well known that the internal surfaces of the barrel and the screw in an extrusion process can be at a temperature above the polymer melting point. Tadmor and Klein (1970) developed an excellent series of photographs showing the progressive melting of polyethylene along the screw. Through ultrastructural investigation, Aguilera *et al.* (1976), reported that the protein cells of soybean grits were disrupted and oriented into strands during the last turns of the screw in the thermoplastic extrusion.

Unlike polymers in traditional plastic extrusion, soy flour is a far more complex biosystem in which some components can easily be activated to initiate interactions by the combined effects of water, heat, pressure and shear, thus potentially making the system even more complicated. Two major fractions of defatted soy flour are carbohydrate and protein. Reducing sugars and starch are reportedly seldom found in mature soybeans (Smith and Circle 1972). The principle soluble carbohydrates of de-

fatted soybean flakes are sucrose, raffinose, stachyose and verbascose. Some of the sugars' melting points are lower than the extrusion temperature of 160°C. Arabinan and arabinogalactan are the insoluble carbohydrates existing in soy flour systems. Information regarding the reaction of these polysaccharides under extrusion condition is not available. However, it was reported (Mercier 1975) that there is no degradation of starch within an extrusion temperature range of 95 to 225°C. It is possible that arabinan and arabino-galactan may also be nondegradable under extrusion conditions up to 160°C. This potentially may minimize the destruction of lysine due to its reaction with reducing sugar in the extrusion process. Polysaccharides may influence the rheological characteristics of cooked soy dough by mixing with the protein molecules. However, protein is considered to be the influential component with respect to the functionality of the system.

It is generally recognized that viscosity data is critical in extrusion modeling and that viscosity has a great deal of influence on extruder and die design (Carley 1963; Chung 1970; Klein 1976), extrusion stability, and texture of the product. The accuracy of the viscosity prediction may determine the success of the extrusion process.

The object of this paper is to provide true apparent viscosity of defatted soy dough as a function of moisture, shear rate and temperature in extrusion process.

## MATERIALS AND METHODS

### Experimental Procedure

Soyafluff 200W from Central Soya was used for this study. The typical composition of this material is moisture — 6.5%, protein — 53%, fat — 1%, crude fiber — 3%, ash — 6% and carbohydrate — 30.5%. The flour was mixed with 22%, 25% and 32% (w/w) of added water in a Hobart mixer for about 5 min to form a soy dough. The dough was then pelletized with a modified Hobart chopper into small pellets about 0.3 to 0.4 cm both in length and diameter. The added water along with the original moisture in the flour brought final moisture in these pellets to a level of about 25.5%, 28.5% and 35.5% respectively. Pellets were then fed into the extruder hopper through a Vibra Screw feeder. The extrusion rate was adjusted by changing the RPM of both the extruder screw and the Vibra Screw simultaneously so that the hopper throat was always filled with pellets, thus maintaining a "full feed" condition.

Maintaining a full feed condition is important in this experiment for providing consistent retention time which improves the stability of the process. The homogeneous distribution of moisture through the dough

may affect the homogeneity of the texture and body strength of the extrudate. The pelletization of the soy dough prior to extrusion improves the moisture distribution while the Vibra Screw provides a steady feed to the system.

In this study, a Brabender extruder with a barrel diameter of 1.918 cm ( $L/D = 20$ ) was used. This unit is fully equipped with temperature controllers, torque, temperature and pressure recorders, a motor of variable RPM, Dynisco pressure and temperature transducer (Model TPT-432A). Four Brabender capillary dies of  $4 \times 30$  (mm),  $3 \times 30$ ,  $2 \times 30$  and  $1 \times 30$  with  $L/D$  ratio of 7.5, 10, 15 and 30 respectively were used. A smooth barrel and a general purpose screw with a compression ratio of 3:1 were employed for this study. The screw was manufactured with solid conveying, compression and metering zones with pitch numbers of 10, 5 and 5 respectively. During the experiment, temperature of solid conveying zone was set at 50 °C. Compression zone, metering zone and die head were set at the same temperature for each experiment. Soy dough pellets with a fixed moisture level were extruded with the temperature of 100, 130 and 160 °C. Experimental data were collected when feed rate, temperature, pressure and torque of the process were at steady state.

The Dynisco transducer was used to monitor temperature and pressure simultaneously at the port 45 mm from die face. It was found that pressure drop was undetectable when there was no die in the channel of die head. Therefore, the observed pressure value in this study was considered as the sum of entrance and capillary pressure drop. The extrusion rate was obtained by weighing samples of extrudate collected from die exit for one minute. Moisture content of extrudate was determined by using a Cenco moisture balance. The actual extrusion rate was calculated on a dry matter basis by referring to the moisture levels of both inlet pellets and outlet extrudates. The volume flow rate was then determined from the actual extrusion rate divided by the corresponding density of the cooked and compressed mass.

The density of the mass at each different temperature and moisture level was determined directly from inside of the die head at each condition. It was calculated from the weight difference of the mass filled die head and the empty die head divided by the void volume of the empty die head. The cooked and compressed soy mass after the last turns of the screw became a semi-transparent type of mass with a color close to brass yellow. It is likely that the protenaceous mass at this stage was in a melt-like state. Consequently, in the text, it will be referred to as melt-like mass, or simply, the mass.

During the preliminary study, various attempts of feeding soy flour and water simultaneously into the extruder failed. It was found that steam generated from the injected water caused bridging of flour in the throat of the extruder. This experience was also reported in the cot-

tonseed meal extrusion by Taranto *et al.* (1975). The feeding problem in this experiment was eliminated by mixing water and flour followed by pelletizing into pellets. This step provides a good moisture distribution in the feed and control of the feed rate.

The effect of moisture content and pH of soy flour on the physical-chemical properties of the extrudate is important. For a bench scale study, soy flour from a newly opened 50-lb bag is recommended. The opened bag should be stored in double plastic bags in a proper container with desiccant. The moisture content of newly opened flour falls in the range of 5 to 7%, and the pH of a 5% flour suspension (w/v) in distilled water is about 6.6. However, the moisture level of flour may quickly elevate to approximately 10% and a pH of about 6.4 if exposed to air. This may shift the titration curve for the flour and affect the characteristics of the extrudate.

Variation of soy dough density with pressure was also investigated by using an Instron Universal Testing Machine. The bulk density of loose, particulated Soyaflo 200W dough (22, 25 and 32% added moisture) ranges from 0.45 to 0.61 g/cc. These values increase with pressure and reach a plateau of about 1.33 g/cc when the pressure is higher than 21 kg/cm<sup>2</sup> (300 psi). The melt-like density with 22 to 32% added moisture measured directly from the interior of the extruder die head immediately after extrusion was found consistent with data from the Instron tester under similar pressure at room temperature.

### Calculation Procedure

Laminar flow of Newtonian fluids in a horizontal, round capillary of constant cross section under isothermal condition is described by the Hagen-Poiseuille equation. This equation may be presented as shear stress ( $\tau_w$ ) and shear rate ( $\dot{\gamma}_w$ ) at the capillary wall and viscosity ( $\mu$ ) being the ratio of the two.

$$\tau_w = \mu \dot{\gamma}_w$$

The shear stress at the wall is

$$\tau_w = \frac{R \Delta P_o}{2L}$$

The shear rate at the wall is

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

When short capillary flow of non-Newtonian fluids such as molten polymers is studied, the above equations are used with Bagley correction (1957) on the shear stress and with Rabinowitsch correction (Klein and Marschall 1968) on the shear rate. The modified equation is shown as follows:

$$\frac{R\Delta P_{\text{cap}}}{2L} \times 980660 = \mu \times \frac{4Q}{\pi R^3} \times \frac{1}{4} \left[ 3 + 1 \left/ \frac{d(\ln \tau_w)}{d(\ln \dot{\gamma}_w)} \right. \right]$$

Where:

- R = inside radius of capillary, cm  
 $\Delta P_{\text{cap}}$  = pressure loss within the capillary, kg/cm<sup>2</sup>  
 L = capillary length, cm  
 980660 = conversion factor for kg/cm<sup>2</sup> to dyne/cm<sup>2</sup>  
 $\mu$  = viscosity, poise  
 Q = bulk volumetric flow rate, cm<sup>3</sup>/sec

$$\frac{1}{4} \left[ 3 + 1 \left/ \frac{d(\ln \tau_w)}{d(\ln \dot{\gamma}_w)} \right. \right] = \text{Rabinowitsch correction factor}$$

$$\frac{R\Delta P_{\text{cap}}}{2L} \times 980660 = \text{shear stress at the capillary wall, dyne/cm}^2$$

$$\frac{4Q}{\pi R^3} = \text{shear rate at the wall, sec}^{-1}$$

Data analysis started with plotting the relationship of observed pressure loss ( $\Delta P_o$ ) and shear rate ( $\dot{\gamma}_w$ ) on log-log scale. The entrance pressure loss ( $\Delta P_e$ ) was obtained by extrapolating the plot of  $\Delta P_o$  versus the ratio of capillary length and diameter (L/D) to L/D = 0. The entrance pressure loss was then plotted against the shear rate on a semi-log scale.

The actual capillary pressure loss was calculated from the observed pressure drop ( $\Delta P_o$ ) less the entrance pressure drop ( $\Delta P_e$ ). The corrected shear stress and the viscosity value were then calculated. Empirical models were developed after programs of VISC and VISPAC from Scientific Products and Research, Inc., Highland Park, N.J. to implement the Rabinowitsch correction and to apply multiple regression analysis on the data. The actual capillary pressure loss and viscosity were expressed as the function of shear rate, temperature and added moisture. In some cases, viscosity was shown only as a function of shear rate and temperature. In the shear rate range of 50 to 1000 sec<sup>-1</sup>, power law was applied to describe flow behavior of the melt-like mass.

## RESULTS AND DISCUSSION

In the treatment of the rheological data, the method used by Bagley

(1957) and Metzger and Brodkey (1963) enables the calculation of end correction for capillary flow of non-Newtonian molten polymers. This method illustrates the correction of entrance effects and allows the comparison of data obtained from using different capillaries. Diagrams in Fig. 1 demonstrate the relationship of observed pressure drop and shear rate at 160°C with various capillary dies. The plot of observed pressure versus L/D of the capillary at several constant shear rates is shown in Fig. 2. The entrance pressure drop at each shear rate is shown by extrapolating the line to L/D = 0. The entrance pressure drop is then plotted versus shear rate as shown in Fig. 3.

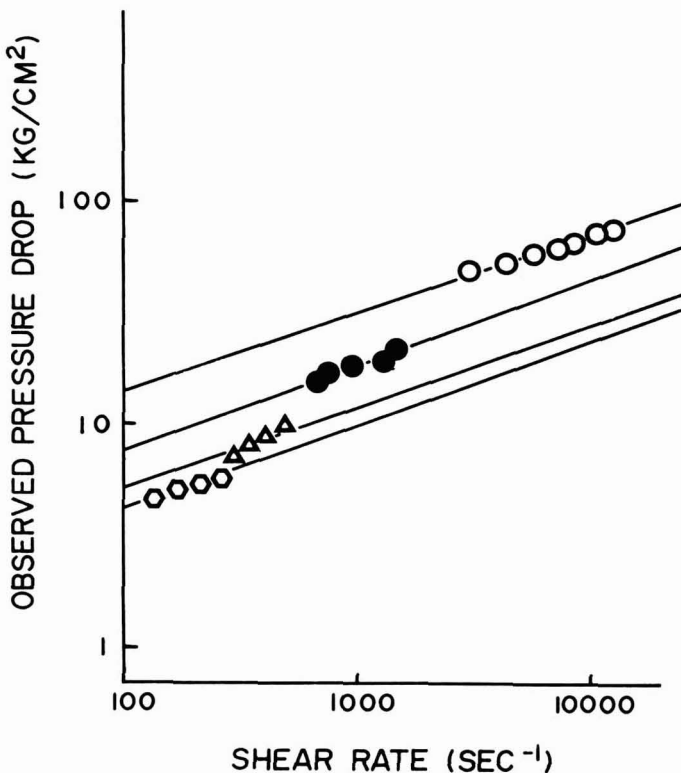


FIG. 1. RELATIONSHIP OF THE OBSERVED PRESSURE DROP  $\Delta P_o$  (KG/CM<sup>2</sup>) TO SHEAR RATE  $\dot{\gamma}_w$  (SEC<sup>-1</sup>) AT VARIOUS CAPILLARY L/D AT 160°C (25% ADDED MOISTURE.)

- L/D = 30
- L/D = 15
- △ L/D = 10
- ⬡ L/D = 7.5

It is known that at extremely low flow rate, a polymer fluid behaves as

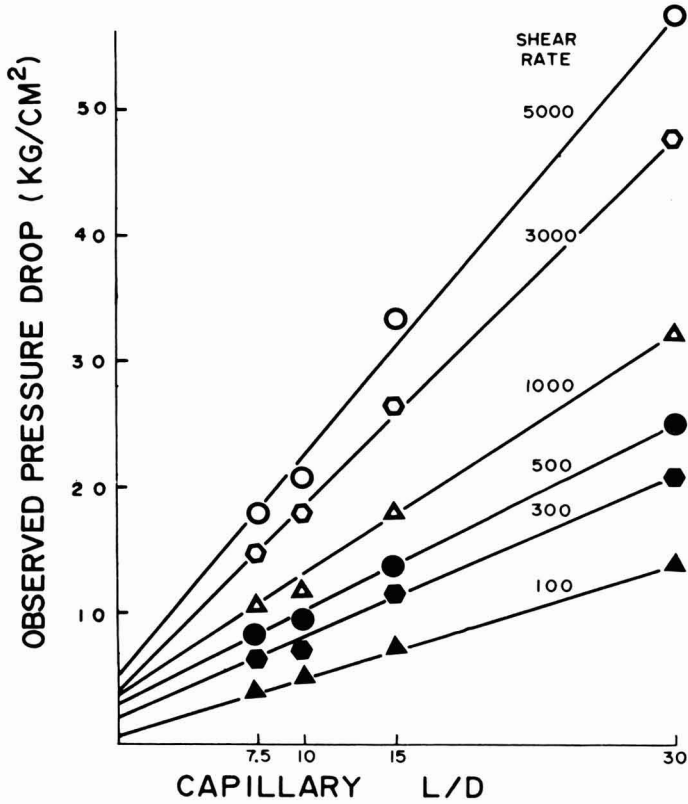


FIG. 2. RELATIONSHIP OF THE OBSERVED PRESSURE DROP  $\Delta P_o$  (KG/CM<sup>2</sup>) TO CAPILLARY L/D AT VARIOUS SHEAR RATES (SEC<sup>-1</sup>) AT 160°C (25% ADDED MOISTURE)

a Newtonian fluid (Weissberg 1962). White (1973) indicated that at a higher extrusion rate streamlines were distorted into a spiral shape at the entrance of capillary and resulted in an increase of entrance pressure loss. Results shown in Fig. 3 demonstrate an increase in entrance pressure drop as the shear rate increases.

It was reported that the elevation of the temperature in a polymer system may provide sufficient energy for the polymer to overcome molecular bonding forces and move to higher modes of molecular motions, such as a rotational and translational movement of chain ends and chain segments (Williams 1971). When the temperature of a protein system increases, it will induce an increase in entropy in the system and encourage the peptide chain to unfold into a random coil form (Huang and Rha 1974). It was reported (White 1973) that linear polymers, in general, exhibit little side group interaction and little resistance to the



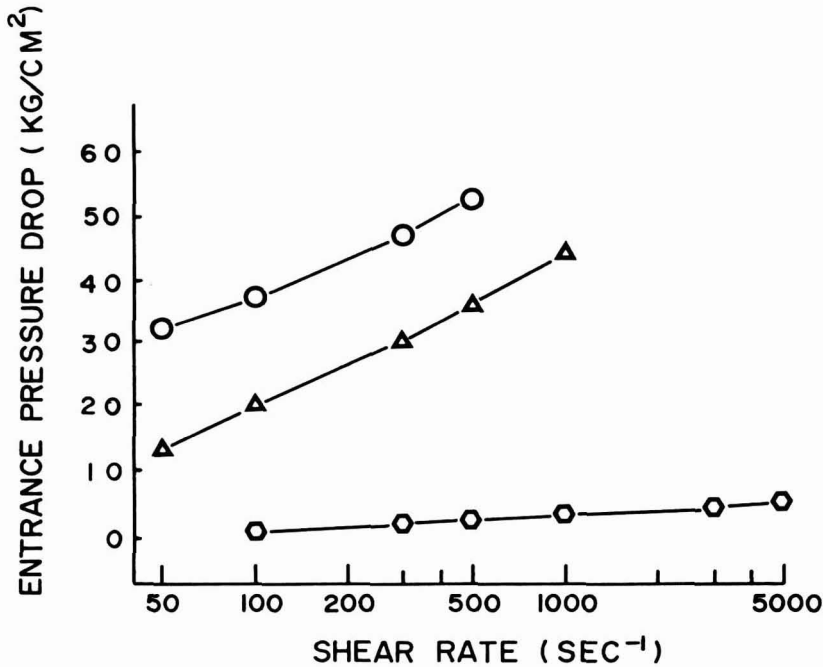


FIG. 3. RELATIONSHIP OF THE ENTRANCE PRESSURE DROP  $\Delta P_e$  (KG/CM<sup>2</sup>) TO SHEAR RATE  $\dot{\gamma}_w$  (SEC<sup>-1</sup>) AT VARIOUS TEMPERATURES WITH 25% ADDED MOISTURE

- 100°C  
 △ 130°C  
 ⬡ 160°C

capillary entrance; however, branched polymers tend to show a higher resistance to the capillary entrance and result in a higher entrance pressure loss. Extrusion at various temperatures reveals a relationship, as shown in Fig. 3, of decreasing entrance pressure loss as the temperature increases. It suggests that the effects of extrusion heat on the unfolding of soy protein and on the capillary entrance pressure loss are similar to the characteristics of conventional polymers.

The interactions between water and proteins have been reported by Lumry (1973) and Von Hippel and Wong (1965). Results shown in Fig. 4 indicate that entrance pressure loss decreases with higher moisture levels. It is likely that the way water plasticizes protein dough during extrusion process is the same as that of solvents to thermoplastics — the higher the solvent content, the less the flow resistance of the system.

This extrusion study reveals that entrance pressure loss is affected by shear rate, temperature and moisture. An expression to describe the response of entrance pressure loss as a function of these three indepen-

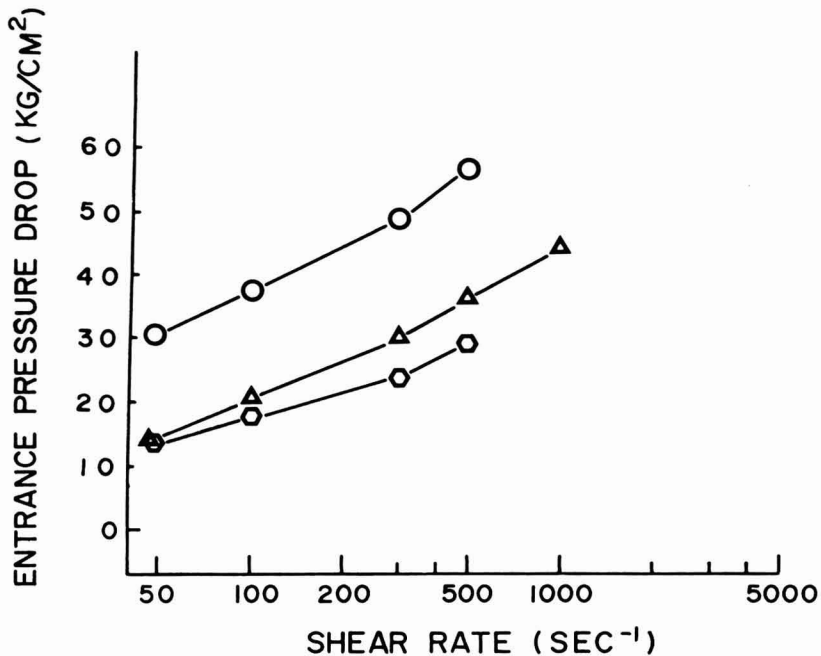


FIG. 4. RELATIONSHIP OF THE ENTRANCE PRESSURE DROP  $\Delta P_e$  (KG/CM<sup>2</sup>) TO SHEAR RATE  $\dot{\gamma}_w$  (SEC<sup>-1</sup>) AT 130°C AT VARIOUS MOISTURE LEVELS

- 22% added moisture
- △ 25% added moisture
- ⬡ 32% added moisture

dent variables was developed. The multiple regression analysis, with a multiple correlation coefficient of 0.951 is shown in the first equation in Table 1.

Most liquid and molten polymers showed an exponential decrease in viscosity with rising temperature (Ballenger *et al.* 1971; Medelson 1976). Soy dough under extrusion conditions also demonstrates the same phenomena, as shown in Fig. 5, 6 and 7. Viscosity data for melt-like mass with the same moisture level were analyzed through VISC and VISPAK programs from Scientific Process and Research, Inc., Highland Park, N. J., to implement the Rabinowitsch effect and to develop a model showing viscosity as a function of shear rate and temperature (Table 2). The multiple correlation coefficient for the viscosity model of the mass with 32, 25 and 22% added moisture are 0.994, 0.998 and 0.991, respectively. The equation which consists of six terms, enables the prediction of rheological data within the domain of the experiment. A set of shear rates ranging

Table 1. Coefficients in both equations<sup>a</sup> of entrance pressure loss  $\Delta P_e$  (Kg/Cm<sup>2</sup>) and viscosity  $\mu$  (poise) as a function of shear rate  $\dot{\gamma}_w$  (sec<sup>-1</sup>), temperature T (°C/100) and added moisture H (% added water/100)

Y	A <sub>0</sub>	A <sub>1</sub>	A <sub>11</sub>	A <sub>12</sub>	A <sub>13</sub>	A <sub>2</sub>	A <sub>22</sub>	A <sub>23</sub>	A <sub>3</sub>	A <sub>33</sub>	Multiple Correlation Coefficient
$\Delta P_e$	6.5891	-0.1137	-0.0815	0.1550	1.9751	10.2296	-3.7440	-8.8186	-77.9908	147.9340	0.951 <sup>b</sup>
$\mu$	10.2997	-1.4234	-0.0729	0.4207	1.7546	6.2089	-3.7372	5.5858	-53.0685	72.1603	0.979 <sup>c</sup>

<sup>a</sup>  $\log Y = A_0 + A_1 \log \dot{\gamma}_w + A_{11} \log^2 \dot{\gamma}_w + A_{12} T \log \dot{\gamma}_w + A_{13} H \log \dot{\gamma}_w + A_2 T + A_{22} T^2 + A_{23} T H + A_3 H + A_{33} H^2$  where A<sub>0</sub>, A<sub>1</sub>, A<sub>11</sub>, A<sub>12</sub>, A<sub>13</sub>, A<sub>2</sub>, A<sub>22</sub>, A<sub>23</sub>, A<sub>3</sub>, and A<sub>33</sub> are empirical constants  
<sup>b</sup>, <sup>c</sup> valid for the range  $55 \leq \dot{\gamma}_w \leq 10500$ ,  $1.0 \leq T \leq 1.6$  and  $0.22 \leq H \leq 0.32$ .

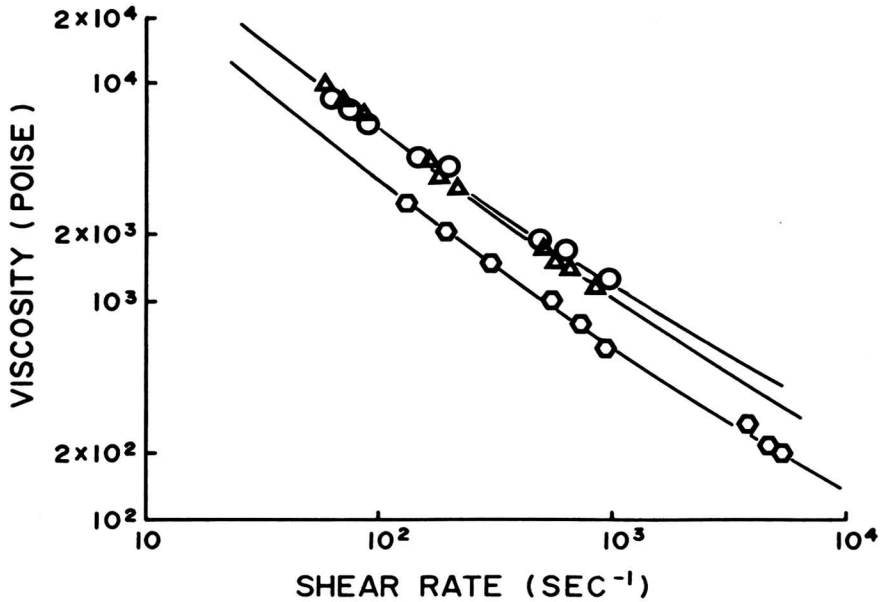


FIG. 5. VISCOSITY CURVES OF SOY FLOUR (SOYAFLUFF 200W) WITH 32% ADDED MOISTURE

○ 100°C

△ 130°C

⬡ 160°C

— Line from mathematical Model

from 50 to 100  $\text{sec}^{-1}$  and the corresponding shear stress calculated from the model were collected and fitted with the power law to examine the flow behavior (Table 3).

Viscosity curves of the melt-like mass with 32% added moisture at 130°C (Fig. 5) is similar to that at 100°C. The power law coefficients are functions of temperature. The effect of temperature on the viscosity between 130 and 160°C is more pronounced than that between 100 and 130°C. This result suggests that the mass below 130°C may be structurally different from those above 130°C. Based on the coefficient  $n$  of power law, the melt-like mass with the added moisture of 32% shows a pseudoplastic (shear-thinning) flow characteristic when the temperature is above 100°C.

Viscosity data with 25% added moisture is shown in Fig. 6. This viscosity at 100°C drops drastically with shear rate and the curve crosses over that of 130°C. At this added moisture level the melt-like mass still behaves as a pseudoplastic material in the temperature range of 100 to 160°C.

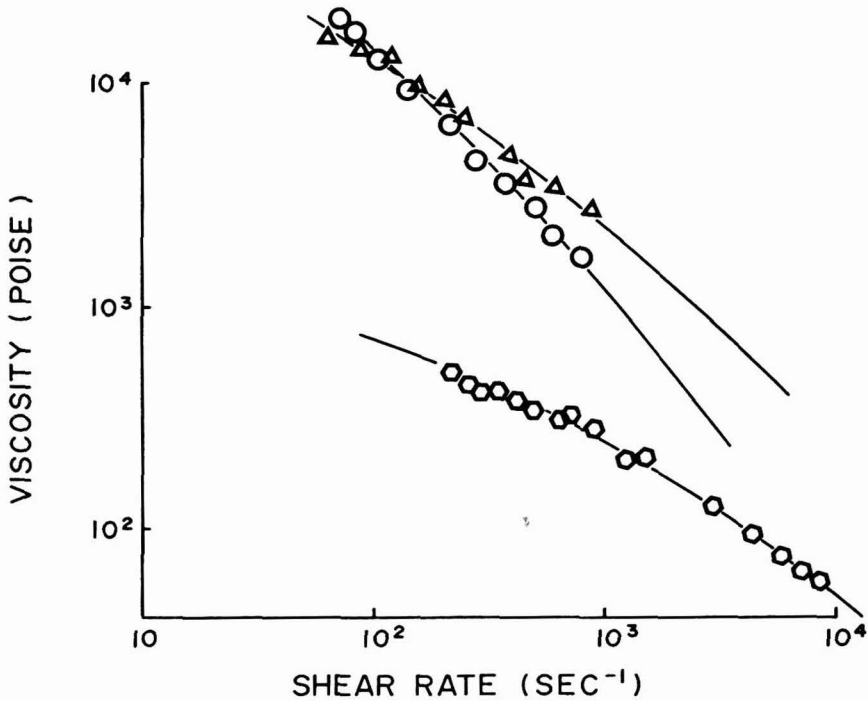


FIG. 6. VISCOSITY CURVES OF SOY FLOUR (SOYAFLUFF 200W) WITH 25% ADDED MOISTURE

○ 100°C

△ 130°C

⬡ 160°C

— Line from Mathematical Model

Figure 7 shows that the viscosity curve at 100°C with 22% added moisture crosses over both curves at 130 and 160°C. The behavior of the melt-like mass at 100°C and 130°C is also affected by the low moisture level. The power law became inadequate to describe the flow characteristics of the mass at these two temperatures within the specified shear rate range. Correlation coefficients for these two cases became as low as 0.245 and 0.632 respectively as shown in Table 3.

In the shear rate of 50 to 1000  $\text{sec}^{-1}$  and the temperature range of 100 to 160°C, the viscosity of soy melts with 32, 25 and 22% added moisture ranged from 10,000 to 500 poise, 20,000 to 200 poise and 20,000 to 1,000 poise respectively. A set of 81 data points calculated from the viscosity equations in Table 3 was analyzed by multiple regression, expressing the viscosity of the melt-like mass as a function of shear rate, temperature and added moisture. A viscosity model containing 10 terms is shown as the second equation in Table 1 with a multiple correlation coefficient of

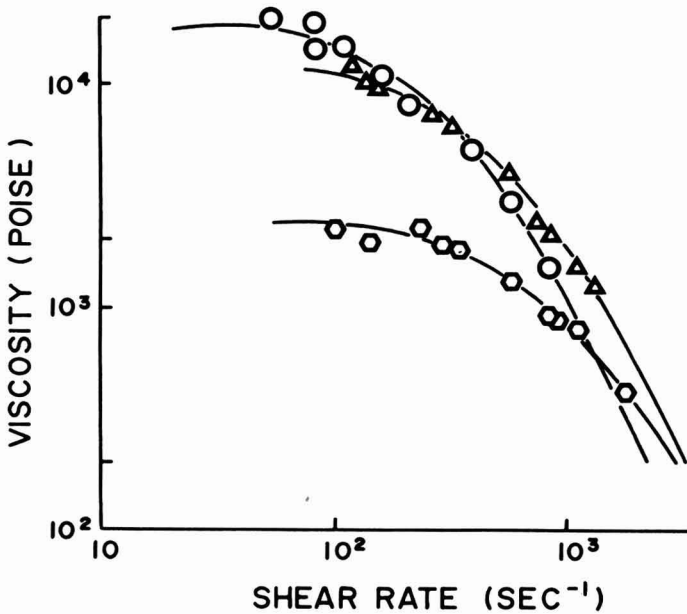


FIG. 7. VISCOSITY CURVES OF SOY FLOUR (SOYAFLUFF 200W) WITH 22% ADDED MOISTURE

○ 100°C  
 △ 130°C  
 ⬡ 160°C

— Line from Mathematical Model

0.979. It is possible to have less than 10 terms for the regression equation when stepwise regression technique is applied. However, for the purpose of accuracy as well as the interpretation of the response surface, all ten terms are included. Response surface analysis on this true apparent viscosity model will be reported elsewhere.

Results from this study suggest that in soy dough extrusion water functions as a plasticizer to lubricate and soften the protein globules making them moldable. The cohesiveness of the dough system may be reduced by the increasing of water content in the system. Both water and heat are necessary to unfold protein molecules in extrusion. The intramolecular disulfide bonds of protein may possibly be exposed by water and heat to form active sulfhydryl ( $-SH$ ) groups and ready for further intermolecular reactions. The degree of unfolding and interaction of proteins during extrusion may be governed by added water, process temperature, shear and pressure. These factors may also control the rheological properties of proteinaceous mass as well as the extrudate body strength and texture. Proper control of these factors may potential-

Table 2. Coefficients in the equation<sup>a</sup> of viscosity  $\mu$  (poise) as a function of shear rate  $\dot{\gamma}_w$  (sec<sup>-1</sup>) and temperature T (°C/100)

Added Moisture	A <sub>0</sub>	A <sub>1</sub>	A <sub>11</sub>	A <sub>12</sub>	A <sub>2</sub>	A <sub>22</sub>	Multiple Correlation Coefficient
22%	1.9153	0.6256	-0.5815	1.1560	3.9976	2.9588	0.991 <sup>b</sup>
25%	-1.1733	-1.6475	-0.0930	1.0297	13.8805	-6.9624	0.998 <sup>c</sup>
32%	4.0761	-1.0016	0.0616	-0.0378	2.7291	-1.1817	0.994 <sup>d</sup>

<sup>a</sup> $\log \mu = A_0 + A_1 \log \dot{\gamma}_w + A_{11} \log^2 \dot{\gamma}_w + A_{12} T \log \dot{\gamma}_w + A_2 T + A_{22} T^2$  where A<sub>0</sub>, A<sub>1</sub>, A<sub>11</sub>, A<sub>12</sub>, A<sub>2</sub> and A<sub>22</sub> are empirical constants  
<sup>b</sup>Valid for the range 55 ≤  $\dot{\gamma}_w$  ≤ 1200 and 1.0 ≤ T ≤ 1.6  
<sup>c</sup>Valid for the range 70 ≤  $\dot{\gamma}_w$  ≤ 10500 and 1.0 ≤ T ≤ 1.6  
<sup>d</sup>Valid for the range 60 ≤  $\dot{\gamma}_w$  ≤ 5210 and 1.0 ≤ T ≤ 1.6

Table 3. Coefficient of power law equation<sup>a, b</sup> of soy melt with various added moisture

Added Moisture (%)	Temperature (°C)	K	n	Correlation Coefficient	Flow Characteristics of Melt
32	100	288260	0.19	0.980	Pseudoplastics
	130	286259	0.18	0.975	Pseudoplastics
	160	177883	0.16	0.975	Pseudoplastics
25	100	783551	0.13	0.980	Pseudoplastics
	130	230750	0.34	0.985	Pseudoplastics
	160	2988	0.65	0.995	Pseudoplastics
22	100	1105646	0.05	0.245	---
	130	159029	0.40	0.632	---
	160	6711	0.75	0.964	Pseudoplastics

<sup>a</sup> $\dot{\gamma}_w = K \gamma_w^n$  where K and n are empirical constants  
<sup>b</sup>Shear rate ranging from 50 to 1000 sec<sup>-1</sup>

ly allow one to create a highly desirable extrudate for further applications. Further studies of the proteinaceous mass during extrusion by using ultra-structure examination techniques along with physical and chemical analysis is highly recommended.

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## MEETINGS

### NOVEMBER

Nov. 5—9: **FOOD AND DAIRY EXPO '78**. Anaheim Convention Center, Anaheim, Calif. W. Dibbern, Dairy & Food Industries Supply Assn., 5530 Wisconsin Ave., Washington, DC 20015.

Nov. 5—9: **ENERGY TECHNOLOGY CONFERENCE AND EXHIBIT**. Albert Thomas Center, Hyatt Regency, Houston, TX 77001. Bruce Ross, Public Relations Department, ASME United Engineering Center, 345 East 47th Street, New York, NY 10017.

Nov. 7—9: **FALL MEETING OF RESEARCH & DEVELOPMENT ASSOCIATES FOR MILITARY FOOD AND PACKAGING SYSTEMS, INC.** U. S. Army Natick Research and Development Command, Natick, Mass. Col. Merton Singer, R&D Associates, 90 Church St., Room 1315, New York, NY 10007.

Nov. 12—16: **71st ANNUAL MEETING OF AMERICAN INSTITUTE FOR CHEMICAL ENGINEERS**. Miami Beach, FL. Verle N. Schrodt, Monsanto Company, 800 North Lingrude Blv., St. Louis, MO 63166.

Nov. 30—Dec. 1: **SHORT COURSE ON CONTROL OF WASTEWATERS IN THE FOOD INDUSTRY**. Holiday Inn O'Hare/Kennedy, Chicago, IL. Sponsored by Food & Nutrition Press, Inc., 265 Post Road West, Westport, CT 06880.

### DECEMBER 1978

Dec. 18—20: **WINTER MEETING OF AMERICAN SOCIETY OF AGRICULTURAL ENGINEERS**. Palmer House, Chicago, IL. R. R. Castenson, American Society of Agricultural Engineers, 2950 Niles Road, St. Joseph, MI 49085.

### AUGUST 1979

Aug. 27—31: **SECOND INTERNATIONAL CONGRESS ON ENGINEERING AND FOOD — FOOD PROCESSING ENGINEERING 1979**. Helsinki University of Technology, Helsinki, Finland. Dr. J. Larinkari, P.O. Box 244, SF-00131 Helsinki 13, Finland.



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HASSON, E. P. and LATIES, G. G. 1976. Separation and characterization of potato lipid acylhydrolases. *Plant Physiol.* 57, 142-147.

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