

A Comparison of Inlet- and Exit-Region Flow of Viscoelastic Fluids in Capillary Tubes

D. R. OLIVER, *Chemical Engineering Department, University of Birmingham*, W. MACSPORRAN, *School of Postgraduate Studies in Chemical Engineering, University of Bradford*, and B. M. HIORNS, *Chemical Engineering Department, University of Birmingham, England*

Synopsis

It is shown, in confirmation of earlier work, that when a viscoelastic liquid flows into a capillary tube, a definite fraction of the inlet pressure drop is required to overcome effects of elastic origin. This pressure drop, ΔP_E , is proportional to the same power of shear rate as the steady-state first normal stress difference measured by means of the jet thrust method. The values of ΔP_E are some 1.6–8.6 times higher than the values of the first normal stress difference, the amount depending on the fluid in use but not on the elastic power-law exponent m . The importance of the extensional mode of deformation in entry region flow is discussed. It is shown by examining dilute polymer solutions after varying periods of degradation that changes in ΔP_E are closely related to changes in the turbulence suppression characteristics of the solutions. The extensional mode of deformation is presumably important in both types of experiment. Both *n*-pentanol and a mineral oil, tested in the same small-diameter capillary tubes as the polymeric solutions, give well-defined values of ΔP_E , which are compared with previously published normal stress figures. The mineral oil is shown to develop particularly high stresses in elongational flow, though the viscous stresses were also higher than for the polymeric solutions used. Previous evidence of the existence of shear elasticity in organic liquids is listed and the potential importance of the case of mineral oil is emphasized.

INTRODUCTION

The inlet pressure drop is larger for a viscoelastic fluid than for a corresponding purely viscous liquid. This effect was noted in 1948 by McMillen.¹ Subsequently, Bagley et al.,^{2,3} following Philippoff and Gaskins,⁴ separated the viscous and elastic components of the end correction and showed how the elastic component varied with the molecular characteristics of the polymer in use. A careful examination of the subject was made by La Nieve and Bogue,⁵ who listed the inlet corrections and plotted that part of the inlet pressure drop due to elastic effects against the tube wall shear rate. Steady-state normal stress differences measured in a rheogoniometer were included on the diagrams. For each elastic liquid used, the "elastic" inlet pressure drop and the steady-state normal stress difference depended on the same power of shear rate, the former values being some 2–6 times

larger than the latter. This ratio appeared to increase, for various liquids, with decreasing values of the power of the shear rate.

The above technique may be applied, for higher shear rates, by using normal stress data obtained by the jet thrust method.⁶⁻⁸ For sufficiently long tubes and high exit velocities, flow in the exit region is approximately steady state and the normal stresses deduced from jet thrusts form a satisfactory extension of data obtained in a rheogoniometer.^{6,8} This behavior contrasts with that observed near the inlet. Recent evidence suggests that the (viscoelastic) fluid which finally enters the tube is confined within a narrow-angled cone upstream of the inlet.^{9,10} The fluid within the cone is both sheared and stretched and the system is self-adjusting; if the flowrate rises or the hole size is reduced, the extensional strain rate will also rise, but the cone angle is free to fall in order to minimize the rise of strain rate. The flow pattern is only part-specified, but it is possible to set the upper and lower limits of the extensional strain rate under given flow conditions.⁹ The flow pattern in the entry region of the tube itself may be distorted, axial cores of high velocity being reported in some cases.^{11,12} These effects may have important bearing on the magnitudes of the "viscous" and "kinetic energy" terms for the inlet region.

The object of the present work was to apply a technique of the type used by La Nieve and Bogue to both fresh and degraded polymer solutions and other liquids, comparing the "elastic" inlet pressure with the first normal stress difference obtained by the jet thrust method. The present work differs from that of La Nieve and Bogue in that the kinetic energy term, rather than the viscous term, is of paramount importance.

FUNDAMENTALS

The overall pressure drop for flow of a liquid through a capillary tube is quoted by La Nieve and Bogue⁵ as

$$\Delta P_{TOT}/\rho = \Delta(KE) + \underbrace{\Delta E + F_E}_{\Delta P_E/\rho} + \underbrace{F_U + F_I}_{\text{entrance friction}} + F_D + F_0 \quad (1)$$

kinetic	elastic	entrance	devel-	exit
energy	energy	friction	oped	friction
			fric-	
			tion	

The terms designated by F indicate irreversible dissipation; it may be noted that the term F_E is included under "elastic effects" to allow for the dissipative loss due to any unusual flow patterns caused by the elasticity. The reader is referred to the original paper⁵ for a full discussion of the terms in eq. (1).

Several workers have shown experimentally that a plot of the total capillary pressure drop against L/D is linear at constant shear rate.^{3,4,5} An example from the present work is shown in Figure 1. The pressure drop

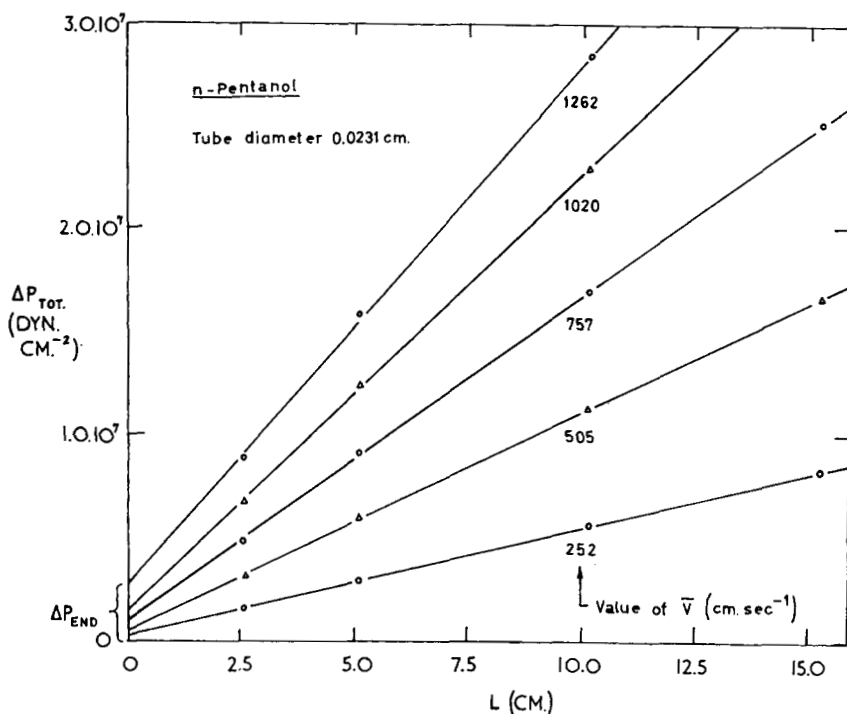


Fig. 1. Relationship between ΔP_{tot} and tube length for n-pentanol in tube of diameter 0.0231 cm.

ΔP_{END} is that pressure required to force the fluid into the capillary entrance; for this case the terms F_D and F_0 disappear, eq. (1). The term F_I relating to the viscous dissipation within the first few diameters of the tube, due to profile rearrangement, should be small relative to the kinetic energy term¹³ and negligible if the lines shown in Figure 1 are exactly straight.

The viscous loss upstream of the entrance, F_U , will be taken as that calculated by Weissberg for creeping Newtonian flow toward a thin orifice plate:^{5,14}

$$\Delta P_U = \rho F_U = 3\mu Q/2R^3 \quad (2)$$

The kinetic energy term $\Delta(K.E)$ will be taken as equivalent to two velocity heads, the value corresponding to a free jet of Newtonian fluid. (See "Discussion.")

EXPERIMENTAL

Apparatus

An accurately controlled liquid flow rate from an Instron rheometer was used in both jet thrust and pressure drop measurements. The method of making jet thrust measurements has been fully described elsewhere.^{7,8,15}

Pressure drop measurements were made, as in Oliver and Macsporrán,¹⁶ by means of a calibrated Bourdon test gauge connected upstream of the capillary in use. The temperature was $23.0 \pm 1.0^\circ\text{C}$.

The square-ended stainless-steel capillaries were made by brazing hypodermic tubing, supplied by Accles and Pollock, into 0.952-cm diameter stainless steel rods. All capillaries were calibrated by water flow under carefully controlled temperature conditions. The dimensions of the tubes are given in Table I.

TABLE I
Dimensions of Capillary Tubes

Diameter, cm	Length, cm
0.0231	2.54, 5.08, 10.16, 15.24,
0.0322	2.54, 5.08, 8.89, 15.24
0.0330	0.635, 1.91, 5.08
0.0358	1.27, 2.54, 5.08, 7.62, 10.16
0.0409	1.27, 2.54, 5.08, 7.62, 10.16
0.0452	2.54, 5.08, 10.16, 15.24
0.0594	0.635, 1.91, 5.08
0.129	1.83, 3.15, 5.61, 10.81, 21.45

Liquids Used

The physical properties of the liquids used are listed in Table II. The dilute polymeric solutions appeared to be Newtonian in steady flow (shear rate range 10^3 – 10^5 sec^{-1}) but the more concentrated solutions exhibited pseudoplastic behavior. The solvent was water.

TABLE II
Liquids Used

Liquid	Viscosity, cp, 20°C	Density, g/ml, 20°C
<i>n</i> -Pentanol	3.82	0.814
Light mineral oil (OM 33)	49.2	0.882
0.01% Polyox	1.40 ^a	1.00
0.01% ET597	1.70 ^a	1.00
0.05% ET597	2.1(10^5 sec^{-1})	
	2.9($2 \times 10^4 \text{ sec}^{-1}$)	1.00
0.10% ET597	2.7(10^6 sec^{-1})	
	3.8($2 \times 10^4 \text{ sec}^{-1}$)	1.00

^a Fresh, carefully prepared solutions. On degradation viscosity falls toward 1.00 cp.

Polyox is a trade name for poly(ethylene oxide), a water-soluble polymer manufactured by Union Carbide Ltd. The coagulant grade in use had a molecular weight of several million. ET 597 (now replaced by AP 273) is a polyacrylamide manufactured by Dow Chemical Co.; the molecular weight distribution is rather broad, with a mean molecular weight of approxi-

mately 2×10^6 . These values may fall owing to oxidation or sustained mechanical shear (see "Discussion").

The degradation of the 0.01% Polyox and ET597 solutions was studied by pumping the liquid in a closed loop, 2.13 m long and 1.91 cm in diameter, using a No. 12 Stuart-Turner centrifugal pump. The liquids were tested when freshly made up and after periods of 2, 4, 6, 8, 12, and 24 hr of circulation in the loop system.

RESULTS

A typical set of data, showing the relative magnitudes of the total endpressure and the correction terms, is given in Table III. For the polymer solution, the "kinetic energy" term is much larger than the "viscous" term, but their combined value is always less than one third of the total endpressure. Figure 2 shows the combined data for three ET597 solutions, of concentration 0.1%, 0.05%, and 0.01%, compared with normal stress values measured by the jet thrust method.^{17, 18}

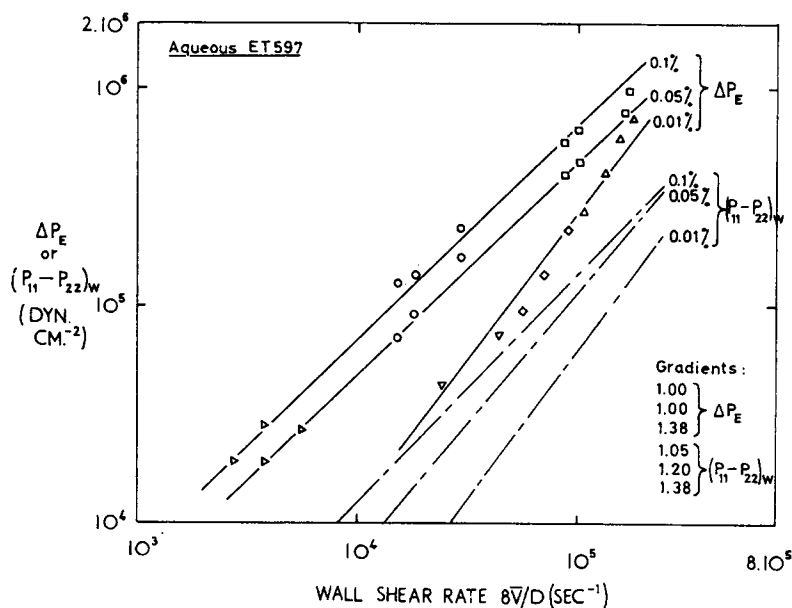


Fig. 2. Values of ΔP_E and $(P_{11} - P_{22})_w$ plotted against $8\bar{V}/D$ for various ET597 solutions. Tube diameters: (Δ) 0.0231 cm; (\diamond) 0.0322 cm; (\square) 0.0330 cm; (∇) 0.0452 cm; (\circ) 0.0594 cm; (\triangleright) 0.129 cm.

For the mineral oil, the two correction terms are of similar magnitude, the total endpressure being greatly in excess of their combined value. Figure 3 shows the values of ΔP_E for *n*-pentanol and mineral oil compared with previously published normal stress data.

TABLE III
 Values of ΔP_{END} , ΔP_{KE} , and ΔP_U for 0.1% ET597 and a Light Mineral Oil

Tube diam., cm	ΔP_{END} , dyn/cm ²	ΔP_U , dyn/cm ²	ΔP_{KE} , dyn/cm ²	\bar{V} , cm/sec	$8\bar{V}/D$, sec ⁻¹	ΔP_E , dyn/cm ²	
0.1% ET597	0.0330	1.45 × 10 ⁶	5.4 × 10 ³	4.9 × 10 ⁵	706	1.7 × 10 ⁵	9.6 × 10 ⁵
		8.3 × 10 ⁵	3.3 × 10 ³	1.8 × 10 ⁵	424	1.0 × 10 ⁵	6.5 × 10 ⁵
	0.0594	6.9 × 10 ⁵	2.7 × 10 ³	1.2 × 10 ⁵	354	8.6 × 10 ⁴	5.7 × 10 ⁵
		2.8 × 10 ⁵	1.3 × 10 ³	4.7 × 10 ⁴	217	2.9 × 10 ⁴	2.3 × 10 ⁵
		1.6 × 10 ⁵	7.9 × 10 ²	1.7 × 10 ⁴	131	1.8 × 10 ⁴	1.4 × 10 ⁵
Light mineral oil	0.0409	1.4 × 10 ⁵	6.5 × 10 ²	1.2 × 10 ⁴	109	1.5 × 10 ⁴	1.3 × 10 ⁵
		3.7 × 10 ⁵	2.3 × 10 ⁴	3.6 × 10 ⁴	202	4.0 × 10 ⁴	3.1 × 10 ⁵
	0.0409	2.4 × 10 ⁵	1.8 × 10 ⁴	2.3 × 10 ⁴	162	3.2 × 10 ⁴	2.0 × 10 ⁵
		1.9 × 10 ⁵	1.4 × 10 ⁴	1.3 × 10 ⁴	121	2.4 × 10 ⁴	1.6 × 10 ⁵
		6.9 × 10 ⁴	9.2 × 10 ³	5.8 × 10 ³	81	1.6 × 10 ⁴	5.4 × 10 ⁴
	3.5 × 10 ⁴	4.5 × 10 ³	1.4 × 10 ³	40	8.0 × 10 ³	2.9 × 10 ⁴	

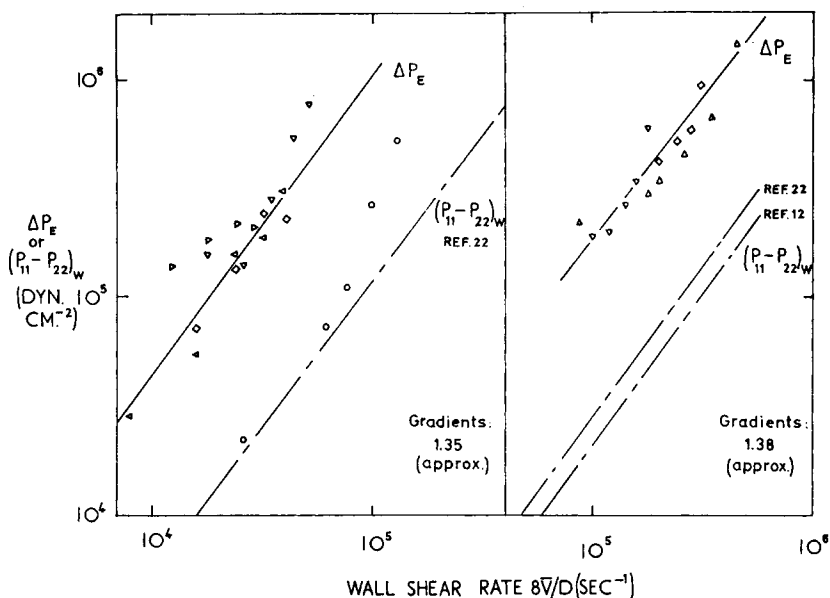


Fig. 3. Values of ΔP_E and $(P_{11} - P_{22})_w$ plotted against $8\bar{V}/D$ for mineral oil and n-pentanol. Mineral oil—tube diameters: (\diamond) 0.0322 cm; (\triangleright) 0.0358 cm; (\triangleleft) 0.0409 cm; (∇) 0.0452 cm; (\circ) 0.0594 cm. n-Pentanol—tube diameters: (Δ) 0.0231 cm; (\diamond) 0.0322 cm; (∇) 0.0452 cm.

Figures 4 and 5 show how the behavior of 0.01% Polyox and 0.01% ET597 changes with progressive degradation of the polymer molecules. The endpressure measurements show a continuous change with increased time of degradation, whereas the normal stress (jet thrust) results suggest a rapid initial loss of elastic properties as degradation proceeds, with little further change in the later stages.

DISCUSSION

Figure 1 illustrates a limitation of the method described in this paper. Extrapolation is an inaccurate process and when the intercept is small, errors are magnified. The larger intercepts are normally subject to errors of the order $\pm 10\%$ and the smaller ones, to errors approaching $\pm 50\%$. In the present work, the viscous end correction (ΔP_U) is always one or two orders of magnitude smaller than the total end correction and if ΔP_U should be in error by 16%,* ΔP_E will be changed by less than 2%. The kinetic energy term, however, sometimes accounts for half the inlet pressure drop and if ΔP_{KE} is underestimated, then ΔP_E will be made to appear artificially large. The figure of two velocity heads should be satisfactory for dilute solutions and liquids having flow behavior indices close to unity (parabolic velocity profile). The kinetic energy flux has also been calculated

* As suggested in Weissberg.¹⁴

for a pseudoplastic fluid having $n = 0.80$ and found to be 5% lower than that appropriate to a fluid with a parabolic velocity profile. Applied to the case of 0.1% ET597 solution (see Table III), the use of the more refined correction would raise ΔP_E by an amount which is generally less than 2%.

The jet velocity is reduced by the "jet swell" phenomenon. Most of the work done against elastic forces is dissipated irreversibly near the tube inlet, but there is a residual steady-state tensile stress at the exit which reduces

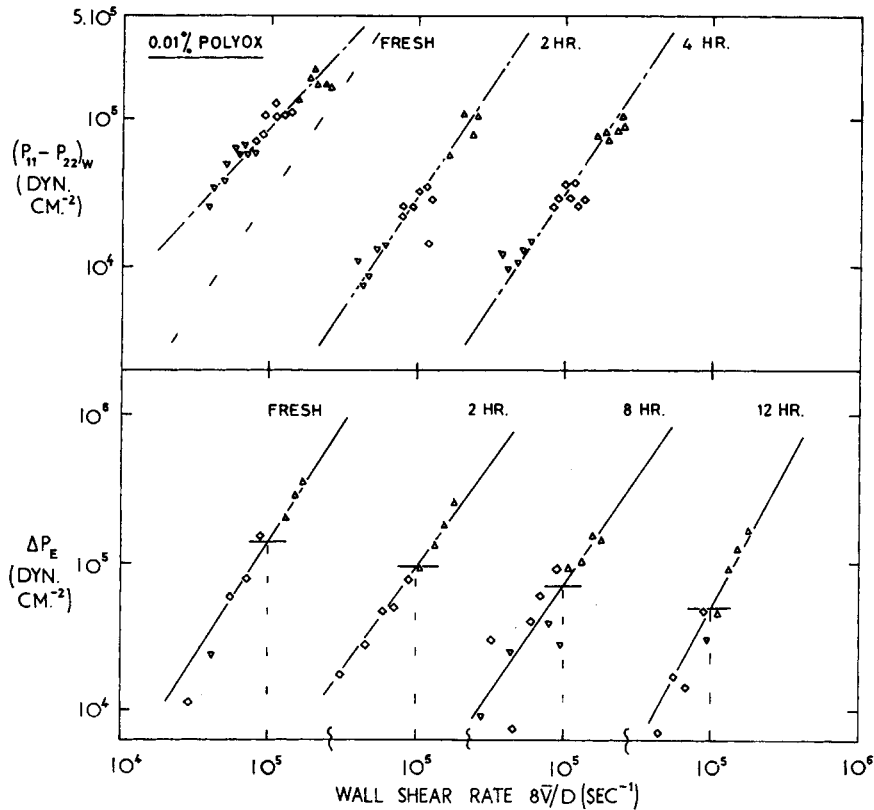


Fig. 4. Values of ΔP_E and $(P_{11} - P_{22})_w$ for fresh and degraded 0.01% Polyox. Tube diameters: (Δ) 0.0231 cm; (\diamond) 0.0322 cm; (∇) 0.0452 cm.

the jet velocity and kinetic energy flux. According to Newton's third law of motion, the same stress is helping to draw liquid out of the tube; some of the work done against elastic forces is thus recovered and not lost as heat. The magnitude of this effect may be estimated as follows: Since the respective values of the kinetic energy flux and momentum flux are

$$\text{kinetic energy flux} = \frac{\pi R^2 \rho V_j^3}{2} = Q \cdot \frac{\rho V_j^2}{2} \quad (3)$$

$$\text{momentum flux (thrust)} = \pi R^2 \rho V_j^2 = Q \cdot \rho V_j \quad (4)$$

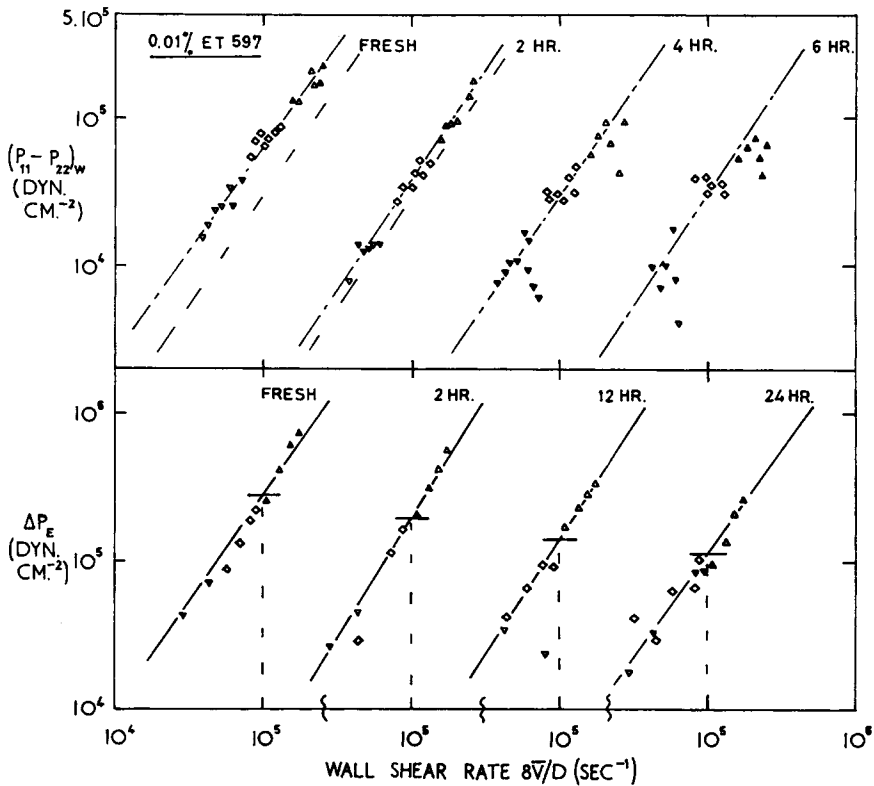


Fig. 5. Values of ΔP_E and $(P_{11} - P_{22})_w$ for fresh and degraded 0.01% ET597. Tube diameters: (Δ) 0.0231 cm; (\diamond) 0.0322 cm; (∇) 0.0452 cm.

where V_j is the final (relaxed) jet velocity, jet thrust data¹⁷ may be used in order to calculate the amount by which the kinetic energy flux differs from the theoretical value ($\pi R^2 \rho \bar{V}^3$ for parabolic velocity profile). These calculations show that, for liquid ejected from a tube of diameter 0.0330 cm, the kinetic energy flux is some 40%–60% below the theoretical value for 0.1% ET597 and 20%–30% below for 0.01% Polyox. It may be implied that between 12% and 25% of the work done against elastic forces at the tube inlet is recoverable at the exit*; if the two effects were separated, the work done at the inlet would be higher, by approximately these percentages, than the values obtained by grouping the effects together.

The data shown in Figure 2 confirm the conclusion of La Nieve and Bogue⁵ that the elastic pressure drop and the first normal stress difference are governed by the same power of the shear rate and are thus proportionally related. The proportionality constant (α_3 as previously quoted⁵) has values of 4.9, 3.8, and 4.5 at the shear rate of 10^5 sec⁻¹ for 0.1%, 0.05%, and 0.01% ET597, respectively. These lie within the range of values quoted

* This applies only to the specific cases mentioned.

in the earlier paper.⁵ However, at the higher shear rates used in the present experiments, the exponent m in the equations

$$(P_{11} - P_{22}) = K_1(\dot{\gamma})^m \quad (5)$$

$$\Delta P_E = K_2(\dot{\gamma})^m \quad (6)$$

is significantly higher than in the earlier work. This has been noted previously.^{6,8} It may also be observed that there appears to be little correlation between α_3 and m , although the value of α_3 varies from fluid to fluid.

The question of the existence of shear elasticity in organic liquids remains somewhat controversial. Reiner¹⁹ gave results which indicate that toluene developed normal stresses at shear rates of $8 \times 10^5 \text{ sec}^{-1}$ (though experiments with gases in the same type of apparatus gave questionable results).^{20,21} Jet thrust measurements by Oliver^{7,12,22} gave strong indications of the presence of shear elasticity in fluids such as glycerol, mineral oil, *n*-pentanol, and toluene at shear rates of the order 10^5 sec^{-1} . Turbulence suppression experiments in small capillary tubes gave similar indications,¹⁶ while the jet thrust behavior of *n*-pentanol in very short tubes is difficult to explain without recourse to the assumption that the liquid has viscoelastic properties.¹² Recently, Rein et al.²³ have used an oscillating quartz crystal viscometer to show that viscoelastic behavior occurs in toluene at shear rates of $5 \times 10^6 \text{ sec}^{-1}$.

The present measurements using *n*-pentanol and a mineral oil (Fig. 3) are the first to be made on these fluids in which a stretching type of flow is used. The endpressure (ΔP_{END}) is considerably in excess of the combined viscous and kinetic energy terms; the gradient of the line relating ΔP_E and shear rate is again comparable with that of the line relating first normal stress difference and shear rate.^{12,22} The proportionality constant α_3 is 8.5 for mineral oil and 8.6 or 6.4 for *n*-pentanol depending on which normal stress data is used.^{12,22} The respective values of the gradient m are 1.35 and 1.38. The implication of these figures is that the above liquids are elastic and relatively more resistant to the stretching mode of deformation than the polymeric solutions. Nevertheless, the values of ΔP_E developed by 0.1% ET597 solution, of apparent viscosity about 3 centipoise, are comparable with those developed by the mineral oil, of apparent viscosity 49.2 centipoise. The elasticity of the oil is still largely masked by viscous effects, particularly at low rates of deformation. At higher deformation rates, however, the contribution from elastic forces will increase and, owing to the freedom of the fluid to select its own lines of approach in the present experiment, the current results may underestimate the effect.

Measurements using fresh and degraded polymer solutions are useful in showing that ΔP_E changes continuously with increasing time of degradation, thus indicating that a real property of the fluid is being measured (Figs. 4 and 5). The corresponding steady-shear normal stresses fall rapidly during the first 2-hr period of degradation, with little change thereafter. It is significant that turbulence suppression experiments on the same solutions follow the trend of the ΔP_E measurements (Fig. 6). For

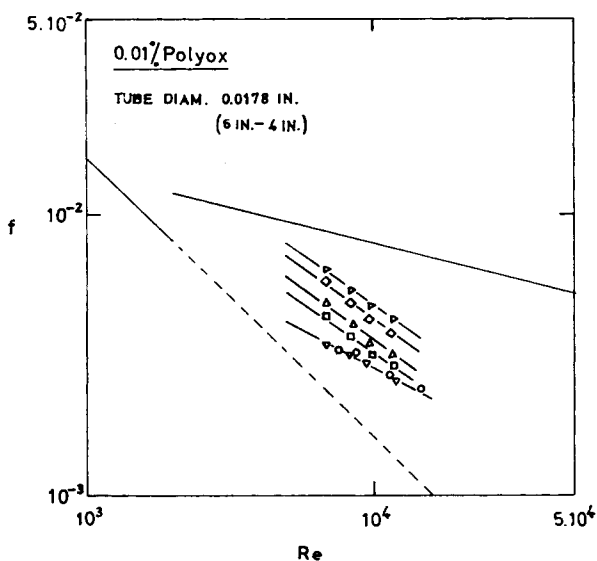


Fig. 6. Friction factor Reynolds number relationship for 0.01% Polyox at various stages of degradation: (○) fresh solution; (▽) 2 hr degradation; (□) 4 hr degradation; (△) 6 hr degradation; (◇) 12 hr degradation; (▷) 24 hr degradation.

0.01% Polyox solution, the friction factor gradually rises with increased time of degradation (though in this case little change occurs in the first 2 hr). Since the steady-shear normal stress undergoes a very rapid change in the first 2 hr of degradation, it is clear that effects are occurring in the turbulence suppression experiment which cannot be fully explained in terms of steady state behavior. This is not surprising in view of the fact that the deformational modes in the sublayer region are essentially of "extensional" type.²⁴ Equally, a simple experiment on extensional flow should not be expected to provide complete information for the prediction of turbulence suppression effects.

An important difference between the behavior of 0.01% Polyox and 0.01% ET597 is shown by the endpressure measurements. The values of α_s for the slowly degrading Polyox are respectively 1.6, 3.2, 2.4, and 1.7 while for ET597 they are 4.6, 5.0, 4.3, and 3.8. The latter solutions are more resistant to extensional deformations, despite the fact that carefully prepared, fresh Polyox solution gives higher normal stress figures than the polyacrylamide solution. Similar trends were observed in earlier endpressure data, extracted from information intended for a different purpose.¹⁷ Turbulence suppression measurements show ET 597 to be more effective as an additive than Polyox after varying periods of degradation, though the performance of freshly prepared solutions is comparable.

It should eventually be possible to explain degradation phenomena in terms of molecular theory. The early theories of Rouse²⁶ and Zimm,²⁷ in which the molecules were considered as noninteracting, segmented flexible chains, have been extended by several workers to include the possibility of

molecular entanglements (reviewed in Meister and Biggs²⁸). For high entanglement densities and high shear rates, forces may develop which permanently break individual molecules, but the molecules may also form rotating clumps of semipermanent nature. The high shear conditions to which the present solutions were subjected should produce these effects, though the irreversible nature of the changes suggests the importance of molecular breakdown as a controlling mechanism. The reduction of molecular chain length will have direct bearing on the elastic properties of the solution.²⁸ A molecular explanation for the viscoelastic behavior of organic liquids may prove more elusive; most experimental evidence suggests that high shear rates ($>10^5 \text{ sec}^{-1}$) or rapid extensional flows are necessary for deviation from Newtonian behavior to be detected.

After a moderate period of degradation, both polymeric solutions give normal stress data which is rather scattered, a limited number of points having negative values. The better points lie close to a line (shown dotted) previously quoted as representing the normal data for water.¹⁵ The scatter of all these data is caused by the fact that the jet thrust differs from the theoretical value by an amount which is only slightly greater than the experimental error; a measure of doubt must therefore surround these results.

It should be mentioned that La Nieve and Bogue⁵ attempted to correlate their endpressure data with normal stress data evaluated from Bagley's results on jet expansion of polyethylene melts. (A similar attempt was made by Savins²⁵ using data for concentrated sodium carboxymethyl cellulose solutions.) The jet expansion method gave unreasonably low normal stress differences; certain difficulties with jet solidification were noted. However, in view of the present satisfactory results obtained by measuring jet thrusts at fairly high momentum flux, it seems as though serious errors are present in the jet expansion method when low speed jets are used in conjunction with a Metzner-type analysis. If the jet velocity of a molten polymer is increased, however, serious instabilities develop and melt fracture is eventually encountered.

It has been stated that the present data, some of which has been omitted for brevity, does not suggest the existence of any correlation between the proportionality constant α_3 and the power law exponent m , eq. (5). It seems likely that fluids differ considerably in their ability to resist extensional deformation and that this may not be predictable, in a simple way, from steady shear data. In order of increasing α_3 , the present data suggest the following ranking: sodium carboxymethyl cellulose solution, Polyox solution, ET 597 solution, *n*-pentanol, and mineral oil. The high resistance of mineral oil to extensional deformation, if confirmed, will have great bearing on lubrication problems. It is apparent that an urgent need exists to develop new, more refined techniques for the study of extensional flows of different liquids.

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Notation

D	diameter of capillary tube.
ΔE	difference between elastic energy per unit mass in the inlet and exit regions, respectively.
f	Fanning friction factor.
F	irreversible energy dissipation per unit mass of liquid. Subscripts refer to: D = fully developed flow in tube; E = loss associated with unusual flow patterns due to elasticity; I = loss associated with development of velocity profile inside tube entrance; U = loss associated with converging flow upstream of tube entrance; 0 = loss in down-stream reservoir.
K_1	constant in eq. (5).
K_2	constant in eq. (6).
$\Delta(KE)$	difference between kinetic energy per unit mass in inlet and exit regions, respectively.
L	length of capillary tube.
m	elastic power law exponent defined by eq. (5).
ΔP_{END}	total of pressure endeffects at the tube inlet.
ΔP_E	inlet pressure loss associated with elastic effects.
ΔP_U	viscous pressure loss associated with converging flow upstream of tube entrance.
ΔP_{KE}	pressure drop required to develop kinetic energy of liquid in tube.
ΔP_{TOT}	total pressure drop across capillary tube system.
$(P_{11} - P_{22})$	first normal stress difference (subscript w refers to tube wall conditions of shear in jet thrust experiment).
Q	volumetric flow rate of liquid.
R	radius of capillary tube.
Re	Reynolds number ($\bar{V}D\rho/\mu$).
\bar{V}	mean velocity of liquid in tube.
V_j	final relaxed velocity of jet.
α_3	proportionality constant between ΔP_E and $(P_{11} - P_{22})_w$
γ	shear rate in liquid ($\dot{\gamma}_w = 8\bar{V}/D$ for Newtonian liquid).
μ	viscosity of liquid.
ρ	density of liquid.

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