

Extrudate Swell and Texture of PS, LDPE, ABS, PVC Melts and their Blends in Extrusion Capillary Flow using a Magnetic Die

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ABSTRACT: The extrudate swell behavior and extrudate texture of various thermoplastic melts, namely, polystyrene (PS), low-density polyethylene (LDPE), acrylonitrile-butadiene styrene (ABS) copolymer, poly(vinyl chloride) (PVC), and their blends, were examined with a magnetic die system in a constant-shear-rate capillary rheometer at a shear rate range $5\text{--}28\text{ s}^{-1}$ and a temperature range $170\text{--}230\text{ }^{\circ}\text{C}$. The extrudate swell results obtained from the magnetic die were then compared with those produced by a nonmagnetic die. The results showed that the extrudate swell increased with shear rate, but decreased with temperature. In a pure polymer system, up to 25% increase in the extrudate swell was observed with the application of the magnetic field to the PS melt, and the effect decreased in the order $\text{ABS} > \text{LDPE} > \text{PVC}$. The extrudate swell changes were associated with the changes in rheological properties of the melts.

The extrudate textures of the ABS and PVC melts were improved by the magnetic field. In PS/LDPE or PS/ABS blend, it was found that the magnetic die resulted in higher values of the extrudate swell than the nonmagnetic die for all blends, the magnetic effect being less as the LDPE or ABS content was increased. For PS/LDPE system, the extrudate swell of the PS melt did not change much with addition of 20% LDPE, but slightly decreased at the LDPE loading of 40%. At higher LDPE loadings, the extrudate swell increased towards the value of the pure LDPE melt. For PS/ABS system, the extrudate swell ratio progressively decreased with increasing ABS content. © 2002 Wiley Periodicals, Inc. *J Appl Polym Sci* 86: 509–517, 2002

Key words: swelling; rheology; extrusion; magnetic field; processing

INTRODUCTION

There are the elastic stresses generated during the transfer of the polymer melt from the reservoir into the capillary die, and as a result of the normal stresses generated by the effect of the shear gradient in the capillary, these elastic stresses affect the orientations of the polymer molecules.¹ It is generally assumed that the elastic stresses begin to decay by some viscous relaxation mechanism as the melt flows down the capillary. If the flow channel is sufficiently long, the elastic forces will eventually be completely dissipated. However, in engineering practice, the flow channels are not sufficiently long and the recovery of the normal stresses is not complete, which has an important implication in extrudate swell as the melt emerges the die exit. Extrudate swell is an important variable that determines the size and quality of the extruded polymer products and the design of polymer processing equipment, for example dies. The extrudate swell is

also used for assessing the polymer elasticity during extrusion.²

Extrudate swell is clearly of importance in processes in which the degree of the swelling is known and can be predicted. For instance, in pipe extrusion, the extrudate swell can be controlled by adjusting the extruder-screw speed or the speed of the haul-off unit. Previous work in the field has been mainly directed towards the experimental investigation of the swell of viscoelastic fluids rather than the theoretical aspects of the problem. These experimental studies were primarily concerned with developing relationships between the operating conditions or molecular structure and the swell observed. Extrudate swell has been widely studied, mostly being performed and explained in terms of the elastic recovery at the die exit in the capillary rheometers, the extrudate swell of a polymer melt being found to vary with molecular weight and structure, shear rate, melt and die temperatures, die characteristics (L/D ratio, die size, geometry), and fillers.

Orbey and Dealy² examined the effects of die geometry and extrusion velocity on parison swell for three HDPE blow-molding resin, using different geometries (straight, convergent, and divergent dies) of annular dies. They found that the equilibrium swell occurred

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after 5–8 min. The diameter and thickness swells were strongly dependent on die design. The convergent die gave the highest diameter swell, whereas the divergent one showed the lowest, the latter involving the degree of molecular orientation while flowing in the dies. The actual value of the swell depended on the rheological properties, especially the response of the melt to the shearing and elongational deformations in the dies. Swan et al.³ measured the thickness and diameter swells as a function of time of a polymer melt using an annular die, the measurement being made with a moving camera being focused on the die exit. The effects of die temperature, flow rate, molecular weight distribution, and die gap on the swelling were of interest. They found that the swell was slightly affected by changes in melt temperature and flow rate, but strongly changed with molecular distribution. The latter was related to the relaxation time of the polymer molecules. The effect of varying the die entrance angle and the die length on extrudate swell in a capillary rheometer was studied by Eggen and Hinrichsen.⁴ They also proposed a mathematical model to predict extrudate swell and compared the calculated results with the experimental ones; a good agreement being found. The proposed calculations were based on analysis of rates and stresses corresponding to the elongational and shear flow components. Liang⁵ investigated the extrudate swell behavior of two blow-film extrusion grade thermoplastics, polypropylene (PP) and low density polyethylene (LDPE) that were melt-blended in a screw extruder, with a capillary rheometer. The extrudate swell measurement was the determination of polymer weight for a given length of the extrudate. The results suggested that the die-swell ratio (B) of the blends decreased approximately linearly with increasing temperature and the die L/D ratio. With increasing wall shear stress, the end pressure losses and B increase nonlinearly with addition of the weight percentage content of PP. This phenomenon can be attributed to the elastic stored energy amount in the melt and its transition due to the viscoelasticity difference between the two phases. Later work by Liang^{6,7} studied the extrudate swell behavior in the capillary flow of LDPE/HDPE blend melts at a temperature range 180–200 °C with a shear rate of 25–120 s⁻¹. It was observed that the extrudate swell ratio increased linearly with increasing shear rate, but decreased progressively with L/D ratio of the dies used. At a low shear rate, the temperature sensitivity of the melt extrudate swell was more significant than that at a high shear rate. In addition, Liang has established a relationship between the exit pressure losses and extrudate swell (B value) and proposed an expression for description of the B and exit pressure drop (P_{exit}) relationship during long die flow of viscoelastic fluids. Work by Sombatsompop and Thongsang⁸ used a direct technique to determine the swelling of PVC

and its blends with recycled PVC pipes in a capillary rheometer with a wide range of die temperatures and recycled PVC loading. The swelling increased with die temperature and recycled PVC loading, the effects concerning increased gelation level with increasing temperature. Later, the swelling of natural rubber (NR) as a function of extrusion time in a circular die of the capillary rheometer was also determined.⁹ The magnitude of the swelling ratio of NR decreased with extrusion time, which involved the flows developed in the rheometer, residence time, and the temperature rise during the flow. The last factor also played an important role in the changes in die swell ratio, especially for a large die diameter.¹⁰

An understanding of the interaction between the molecular conformations and orientations and magnetic field is important for processing or potential applications and for determining characteristics of the material¹¹ because the extrudate swell is related to the molecular orientations and the degree of extrudate swell should be affected by the magnetic field. In this paper, the extrudate swell of various thermoplastic melts (PS, LDPE, ABS, and PVC, and their molten blends) was examined at the die exit of a constant shear-rate capillary rheometer using a magnetic die, and the results were then compared with those produced by a nonmagnetic die for given test conditions. This is the *first time* that a magnetic die was introduced in a capillary rheometer. The findings in this paper clearly offered significant practical aspects as to how to control or alter the swell ratio of the extrudate *without* changing process/processing parameters.

EXPERIMENTAL

Raw materials

Four polymers, including amorphous and semicrystalline polymers, were used to study the extrudate swell in the capillary rheometer and are listed in Table I.

Polymer blend system

The work was also extended to investigate the extrudate swell behavior in polymer blend systems. Two blending systems were chosen, PS/LDPE and PS/ABS. The blending compositions were varied from 0 to 100% w/w. The polymers were melt-blended with a twin screw extruder (Haake PolyLab-Rheomex CTW 100P). The blending temperature profiles on the extruder were 180, 190, 200, and 210 °C from hopper to die zones. The screw rotating speed used was 80 rpm and a three-strand die (each strand with a 3 mm diameter) was used, the produced extrudates being passed through a water bath before undergoing the pelletization unit to produce polymer blend granules.

TABLE I
List of Polymers

Polymer	Grade/supplier
Polystyrene (PS)	Styron 656D 267/Siam Polystyrene Company Ltd. (Thailand)
Low-density polyethylene (LDPE)	LDPE-LD1905F/Thai Polyethylene Company Ltd. (Thailand)
Acrylonitrile butadiene styrene copolymers (ABS)	PORENE MH1-0105001/Thai ABS Company Ltd. (Thailand)
Polyvinyl chloride (PVC)	Y5911BLA/Thai Plastics & Chemicals Company Ltd. (Thailand)

It should be noted that for consistency and comparison purposes, all pure polymers also underwent this blending process prior to further use.

Experimental apparatus

The constant shear rate rheometer was used as used in a previous work.⁹ The barrel, with a 26-mm diameter and 145-mm length, was designed so that the die system could be easily changed. In this work, two circular dies with the same dimension ($L/R = 60/3$) were used, one being a permanent magnetic die and the other being a nonmagnetic die. The magnetic die was achieved by applying the magnetic field with an intensity of 126 milli-tesla or 1.26×10^3 Gauss to the die, the intensity being measured at the die entry region. It should be noted that these two dies were made of the same material; therefore, the interfacial effect between the melt and the die wall in this case can be negligible. A small pressure hole was located between the two die locations to detect die entrance pressure drop occurring, the entrance pressure being taken using the Pin-Spring pressure sensor.¹² The apparatus temperature was controlled using a Eurotherm 018 temperature controller.

Extrudate swell measurement

The extrudate swell ratio (B) of the polymer melts was directly measured by calculating the ratio of diameter of the extrudate to that of the die, the extrudate diameter being based on the size of the extrudate diameter in the fully swollen (~ 2 in. away from the die exit)⁹ as shown in eq. 1.

$$B = \frac{\text{diameter of extrudate}}{\text{diameter of die}} \quad (1)$$

The textures of the extrudates were visualized with a digital camera. It should be noted that the materials used in this stage had experienced the twin-screw extrusion process as detailed earlier. In this article, the effects of test temperature and shear rate on the extrudate swell for various polymer melt systems were also considered, the test temperature range used being 170–230 °C, depending on the type of polymers used, and the shear rate ranging from 5 to 28 s^{-1} .

In this article, the percentage change in the extrudate swell (ΔB) due to the applied magnetic field can be obtained by the differences in the extrudate swell ratios obtained from nonmagnetic die (B_n) and magnetic die (B_m), as expressed in eq. 2:

$$\% \text{change in } B \text{ due to magnetic } (\Delta B) = \frac{B_m - B_n}{B_n} \times 100 \quad (2)$$

Rheological properties

Flow curves (relationship between wall shear stress and wall shear rate) of the thermoplastic melts, using the same dies and test conditions as were used for extrudate swell measurements, were also established, and the calculations can be obtained elsewhere.⁹

RESULTS AND DISCUSSION

Pure molten polymer system

Extrudate swell ratio

Figures 1–4 show plots of extrudate swell and shear rate for different extrusion temperatures for PS, LDPE, ABS, and PVC, respectively, using the magnetic and

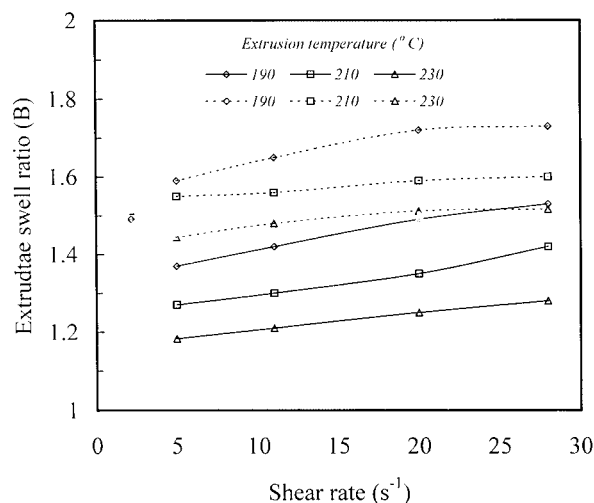


Figure 1 Extrudate swell ratio and shear rate for the PS melt at different test temperatures (solid line: nonmagnetic die; dashed line: magnetic die).

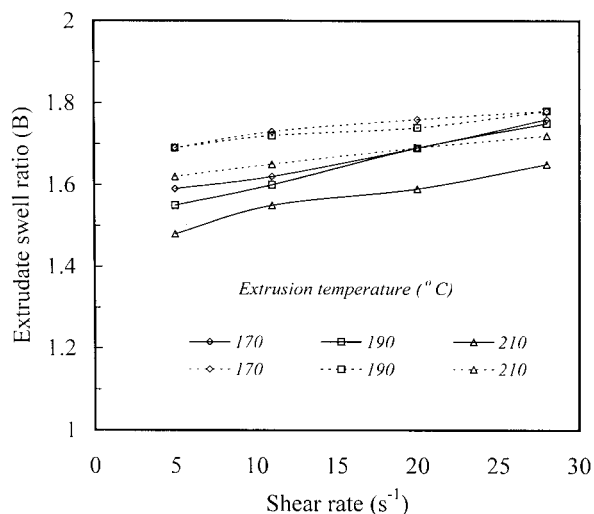


Figure 2 Extrudate swell ratio and shear rate for the LDPE melt at different test temperatures (solid line: nonmagnetic die; dashed line: magnetic die).

nonmagnetic dies. For a given test condition, each polymer melt gave different degrees of swelling due to the differences in the molecular characteristics.^{5,11} Generally, for both dies, the extrudate swell increased with shear rate but decreased with temperature. The increase in extrudate swell with shear rate was due to an increase in applied stresses accompanied by a decrease in stress relaxation time to the polymer molecules, whereas the decrease of extrudate swell with extrusion temperature resulted from an increase in the viscous component in the molecules and thus easier molecular relaxation. In all cases, the effect of extrusion temperature on the change in the extrudate swell was more pronounced than that of shear rate. For the temperature effect, the sensitivity of the extrudate

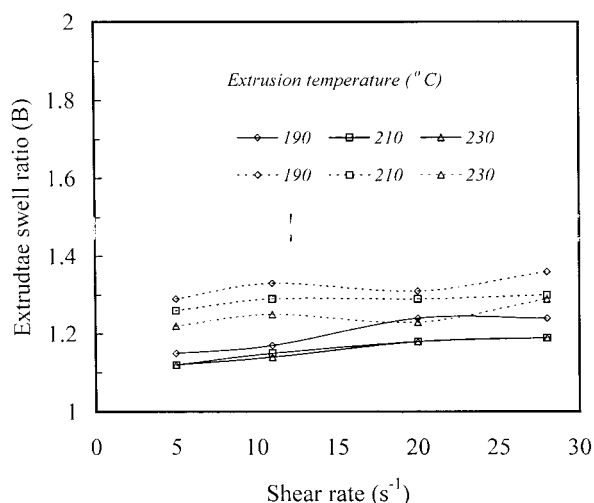


Figure 3 Extrudate swell ratio and shear rate for the ABS melt at different test temperatures (solid line: nonmagnetic die; dashed line: magnetic die).

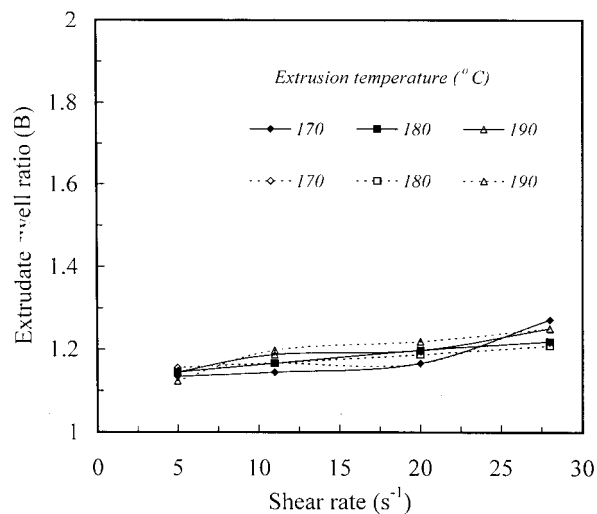


Figure 4 Extrudate swell ratio and shear rate for the PVC melt at different test temperatures (solid line: nonmagnetic die; dashed line: magnetic die).

swell decreased in the order PS→LDPE→ABS→PVC. It was interesting to note that the nonpolar polymers like PS and LDPE had higher swell ratio than polar polymers such as ABS and PVC; the reasons for this difference are explained later in this paper.

Considering the magnetic field effect, Figures 1–4 clearly indicate the magnitude of the differences observed between the data obtained with the magnetic and nonmagnetic dies. In general, the extrudate swell ratio from the magnetic die was greater than that from the nonmagnetic die, the differences varying with molecular structure, extrusion temperature, and shear rate. The percentage differences (ΔB) in the degree of the extrudate swell for all polymer melts between magnetic and nonmagnetic dies are listed in Table II. The values of the percentage differences in the extrudate swell between the two dies decreased in the order PS→ABS→LDPE→PVC. The differences in the results decreased as the shear rate was increased. For LDPE melt, most data of the difference in the extrudate swell were relatively small and were close to the experimental errors (the error of the extrudate size measurement being in the range $\pm 3\%$). No measurable differences were observed for the PVC melt. In the case of PS and ABS melts, the increase in extrudate swell due to the magnetic field was relatively high (10–25% for PS and 5–13% for ABS), these values being greater than the experimental errors. The explanation to these findings was the changes in the rheological properties of the melts upon the application of the magnetic field. The flow curves of PS, LDPE, ABS, and PVC melts produced by the two dies at 190 °C are shown in Figure 5. The results produced using the nonmagnetic and magnetic dies were very similar except for the PS melt. For a given wall shear rate, the wall shear stress of the PS melt produced by the

TABLE II
Percentage Differences (ΔB) in the Extrudate Swell Ratio between Magnetic and Nonmagnetic Dies for PS, LDPE, ABS, and PVC Melts at Various Test Temperatures and Shear Rates

Shear Rate (s^{-1})	PS			LDPE			ABS			PVC		
	190 °C	210 °C	230 °C	170 °C	190 °C	210 °C	190 °C	210 °C	230 °C	170 °C	180 °C	190 °C
5	16	22	25	6	9	10	12	13	9	2	— ^a	— ^a
11	16	20	23	6	8	7	13	12	9	2	— ^a	1
20	15	18	16	4	3	6	6	9	5	— ^a	— ^a	2
28	13	13	10	1	2	4	9	9	8	— ^a	— ^a	— ^a

^a No difference or negative value.

magnetic die was much greater than that produced by the other die. Therefore, the latter could be the reason for the increase in the extrudate swell due to the magnetic field for the PS melt.

It was postulated in this work that the increase in the wall shear stress (and extrudate swell) of the PS melt due to the application of the magnetic field was associated with the increases in the existing electron delocalization and a distortion of the electron clouds, which were induced by the applied magnetic field, within the benzene rings side-groups in the molecules. These results were then thought to cause an increase the shear stresses and a change in molecular orientations during the flow.¹³ On molecular relaxation at the die exit, the increased shear stresses were dissipated elastically and the molecular recoiling process occurred, thus causing the increased swelling of the extrudate. This view could also be applied to explain the differences in the extrudate swell due to the magnetic die used in ABS melt, but it was not the case for LDPE and PVC melts. This difference suggested that because of the absence of the benzene groups, the LDPE and PVC molecules were not induced by the magnetic fields.¹⁴

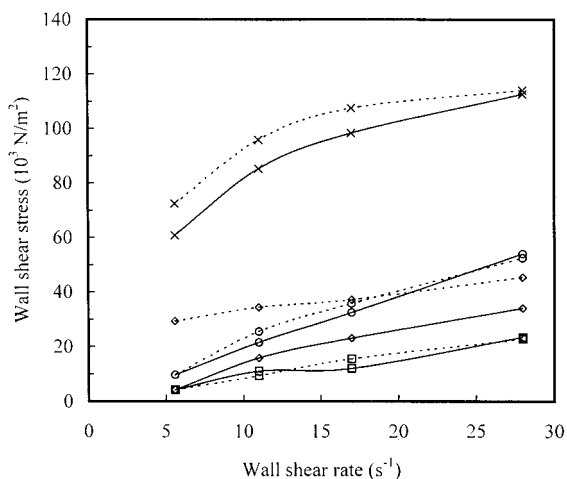


Figure 5 Flow curves for PS (diamond), LDPE (square), ABS (circle), and PVC (cross) melts at 190 °C (solid line: nonmagnetic die; dashed line: magnetic die).

Extrudate texture

The extrudate textures for PS, LDPE, ABS, and PVC melts produced by the two dies (nonmagnetic and magnetic) at 190 °C and shear rate of $28 s^{-1}$ are shown in Figure 6. There were no observable differences in the extrudate textures for PS and LDPE melts, the extrudate surfaces being relatively smooth (Figure 6a and b). This result may be because the applied stress did not exceed the critical tensile stresses of PS and LDPE melts. For the ABS and PVC extrudates, the extrudates generated by the magnetic die had better surface textures (smoothness) than those produced by the nonmagnetic die (Figures 6c and d). Without the magnetic field, the extrudate surface shows the shark-skin (rough surface) characteristic, suggesting that the applied stress exceeded the critical shear stresses of the ABS and PVC melts. Because both ABS and PVC are quite strong polar polymers, when the magnetic field was applied to the molecules, the polarities and subsequently the dipole–dipole interactions between atoms in the ABS and PVC molecules were thought to increase. These interactions would result in an increase in the critical tensile stresses of the melts, and the surface roughness of the extrudates would then be minimized.¹⁵ The differences in the degree of surface roughness between ABS and PVC melts were associated with their critical tensile stresses and the stresses applied to the molecules. It should be noted that in all cases, no melt distortion (or melt fracture) occurred for the shear rate range used.

Blended molten polymer system

Extrudate swell ratio

The extrudate swell ratio of PS melt blended with various LDPE contents for three different shear rates at 190 °C using the two dies is shown in Figure 7. For any given shear rate and material composition, the extrudate swell ratio obtained by the magnetic die was different from that obtained by the nonmagnetic die, the extrudate swell with the magnetic die being relatively greater. The differences decreased with increasing LDPE content. This result was related with the

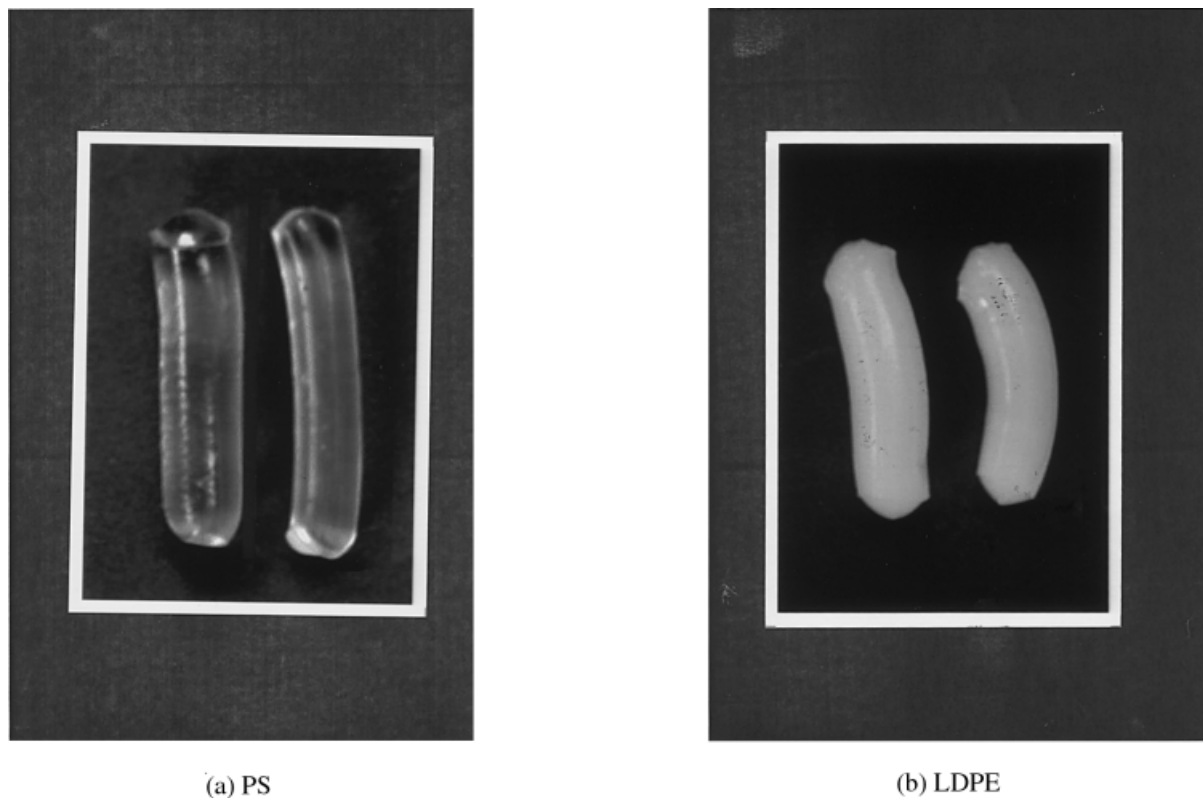
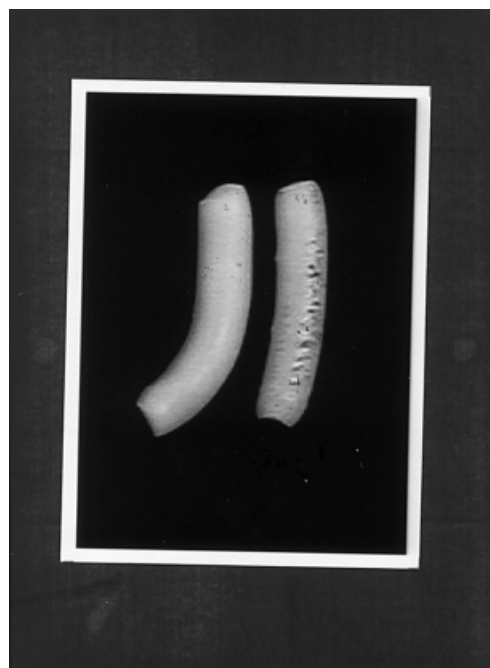


Figure 6 The extrudate textures for different polymers at 190 °C with a shear rate of 28 s⁻¹ (left: magnetic die; right: nonmagnetic die).

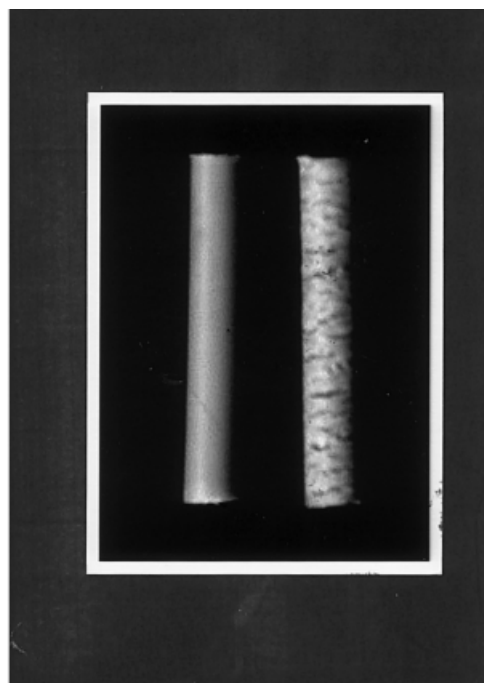
results from Figures 1 and 2; that is, the magnetic field had relatively small effect for the LDPE, but a large effect for the PS. In Figure 7, the extrudate swell ratio of the blend did not change much with 20% LDPE loading, but a slight decrease was seen at 40% loading. It was interesting to observe that the values of the extrudate swell ratio at 40% LDPE loading was lower than that of the pure PS and LDPE melts for both dies. This result may be because the PS and LDPE blend is incompatible, and the small content of LDPE molecules added to the PS melt may interfere with the molecular movements (mobilities) and abilities to disentangle of the PS melt during the flow in the capillary. If this explanation was true, there should be some changes in the molecular mobility related properties. A supplemental work was carried out to follow the change in the glass-transition temperature (T_g) of the PS incorporated with LDPE using a differential scanning calorimetry (DSC) technique (using a heating rate of 2 °C/min); the experimental procedure is found in a previous work.⁸ The T_g results of the PS melt are listed in Table III. The T_g of the PS shifted by 5 °C with 40% LDPE melt, suggesting that the molecular mobility of the PS molecules became more restricted with the presence of the LDPE molecules, which would lead to the decrease in the extrudate swell. However, at high LDPE loading (60–80%), the extrudate swell ratio progressively increased towards

the swell ratio of the pure LDPE melt, the swelling behavior being dominated by LDPE melt characteristic. The rheological properties of PS/LDPE blends are shown in Figure 8. The wall shear stress did not change much with LDPE content for all shear rates used.

The PS and ABS melts were used as a compatible blend in this work, and the extrudate swell results are shown in Figure 9. The extrudate swell ratio progressively decreased with increasing ABS content. Again, the extrudate swell ratio with the magnetic die was greater than that with the nonmagnetic die for all blends, the differences being less when the ABS content was increased. It should be emphasized that at higher ABS content, application of the magnetic field resulted in a considerable decrease in the swell ratio. This result would probably be caused by an increased polarity of the blend due to the presence of the ABS. According to the earlier discussion of swelling behavior of pure PVC and ABS melts, an increase in the polarities in the molecules gave rise to an increased critical shear stresses with a decreased extrudate swell. These results can be substantiated by the rheological results of PS/ABS blends in Figure 10, which illustrates that the wall shear stress increased with ABS content for all shear rates used. In this respect, it would be interesting, but beyond the scope of this paper, to study the effects of the ABS polarities on the



(c) ABS



(d) PVC

Figure 6 (Continued from the previous page)

extrudate swell ratio (i.e., varying the monomer contents) on the application of magnetic field.

Extrudate texture

During the experiment, there were no observable differences in the extrudate textures for PS/LDPE and PS/ABS blends from both dies used, the extrudate surfaces being relatively smooth for all cases. Thus, no further discussion was required in this stage.

In summary, the findings in this article clearly had practical implications in that the extrudate swell in the extrusion process changed with the application of the magnetic die. For quantitative benefit, the size of the extrudates could be altered by the magnetic field. If this procedure was applied to the process, no changing of any process/processing parameters would be required (Note: In practice, adjusting one process or processing parameter will automatically change other parameters, and this makes the process more difficult to control). For qualitative benefit, the texture of the extrudates, especially with the strong polar molecules such as PVC and ABS, could be improved by the use of the magnetic die.

CONCLUSION

A magnetic die was used to investigate the extrudate swell at the die exit in a capillary rheometer for PS,

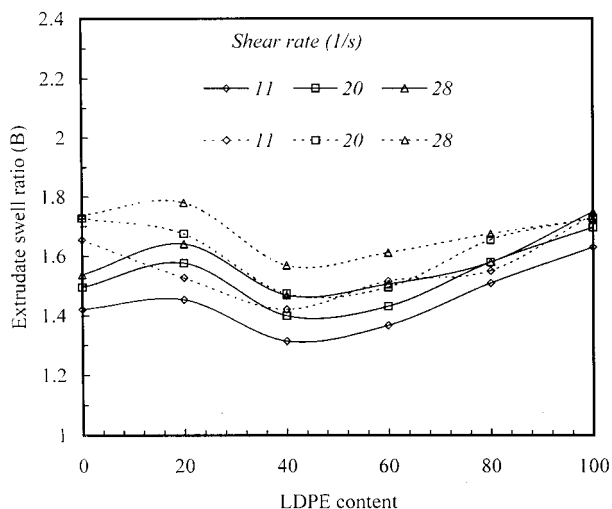


Figure 7 Extrudate swell ratio of PS melt blended with various LDPE contents at different shear rates at 190 °C (solid line: nonmagnetic die; dashed line: magnetic die).

TABLE III
Values of the Glass-Transition Temperatures of PS in PS/LDPE Blends Obtained by DSC Technique

Blend Composition (PS:LDPE)	T _g (°C)
100 : 1	100
80 : 20	102
60 : 40	105
40 : 60	104
20 : 80	103

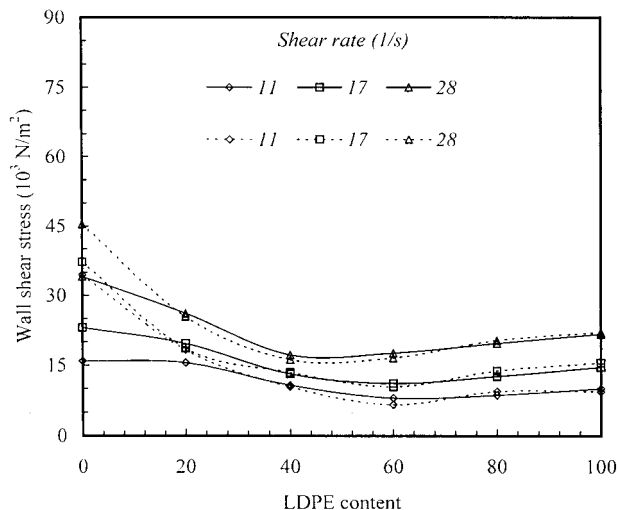


Figure 8 Rheological properties of PS melt blended with various LDPE contents at different shear rates at 190 °C (solid line: nonmagnetic die; dashed line: magnetic die).

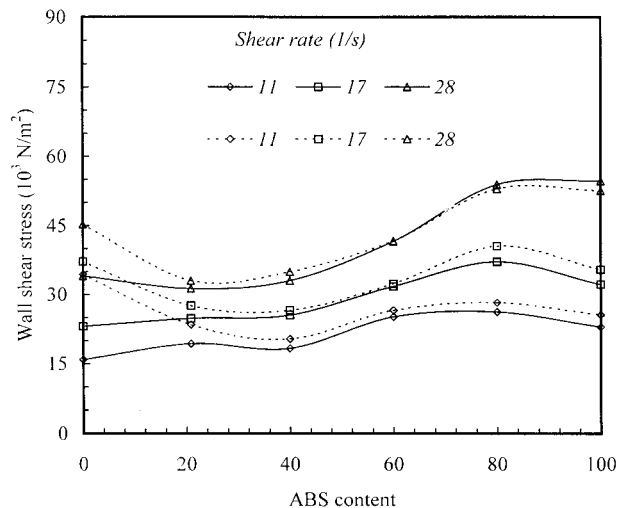


Figure 10 Rheological properties of PS melt blended with various ABS contents at different shear rates at 190 °C (solid line: nonmagnetic die; dashed line: magnetic die).

LDPE, ABS, and PVC melts and their blends. The following should be noted:

- The extrudate swell increased with shear rate, but decreased with extrusion temperature.
- For a pure polymer system, using the magnetic die resulted in a significant increase in the swelling ratio of the extrudates (up to 25% for PS melt and 13% for ABS melt) as compared with the nonmagnetic die, this involving the increases in the existing electron delocalizations and the distortion of the electron clouds within the benzene rings, the shear stress during the flow, and molecular orientations. The percentage differences in the

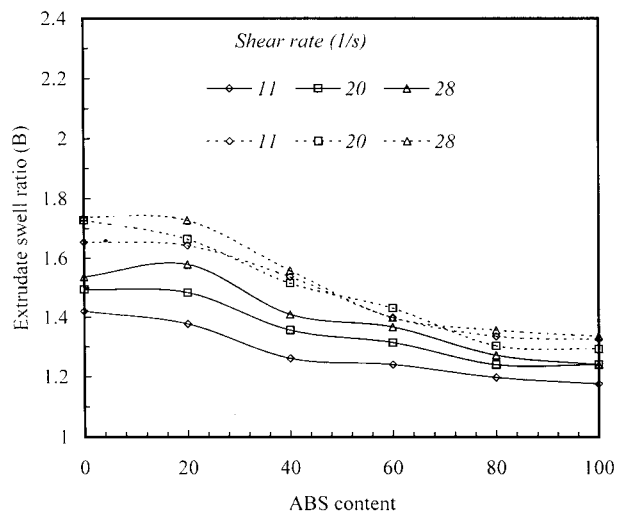


Figure 9 Extrudate swell ratio of PS melt blended with various ABS contents at different shear rates at 190 °C (solid line: nonmagnetic die; dashed line: magnetic die).

extrudate swell due to the magnetic field decreased in the order PS→ABS→LDPE→PVC melts.

- The extrudate textures of the ABS and PVC melts were improved with the use of magnetic die, this being associated with the increases in the polarities, dipole–dipole interactions, and the critical tensile stresses of the melts.
- In PS/LDPE or PS/ABS blend, using the magnetic die gave rise to higher values of the extrudate swell ratio than the nonmagnetic die for all blends, the magnetic effect being less pronounced as the LDPE or ABS content was increased. For PS/LDPE system, the extrudate swell of the PS melt did not change much with addition of 20% LDPE, but slightly decreased at the LDPE loading of 40%. At higher loadings, the extrudate swell increased towards the value of the pure LDPE melt. In the PS/ABS system, the extrudate swell ratio progressively decreased with increasing ABS content, this being more pronounced with the use of the magnetic die.

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