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Effect of Processing Conditions and Die Design on Die Drool Phenomenon for HDPE Polymer Melt

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Abstract

In this work, the die drool phenomenon analysis has been performed for HDPE polymer melt by using specific type of experimental set-up. It has been revealed that the thermally induced degradation occurring inside the processing equipment may leads to HDPE melt viscosity/elasticity enhancement, which promotes unwanted material accumulation at the end of the extrusion die. It has been found that for particular processing conditions, shaped die, flared die or the use of hard chrome instead of stainless steel can reduce die drool phenomenon for HDPE.

Introduction

Die drool, also called die build-up, die bleed or plateout, is undesirable spontaneous accumulation of polymer melt at the exit edges or open faces of extrusion die during melt extrusion of polyolefins, PVC, or filled polymers. This unwanted phenomenon can occur in all common extrusion processes (film blowing, pipe or profile extrusion, fiber spinning, film casting etc.) and after it arises, the productivity of extrusion line rapidly decreases from following basic reasons. Firstly, small amount of accumulated and usually degradated material sticks to the extrudate at the die exit region which cause depreciation of the final product. In the case of film casting or extrusion of solid profiles is possible to solve this problem only by strip the accumulated material from the die exit region. However, in the case of film blowing, pipe, cored profile extrusion or wire sheathing the die drool arises not only at the outer exit region but also at the inner one thereby the first clean opportunity is impossible. Therefore, in these cases the extrusion line is necessary to periodically switch off and after cleaning the die which is uneconomical and time consuming.

The fundamental problem of die drool phenomenon is to understand and describe the formation principle of this phenomenon. In the open literature, several factors have been found to support the formation of this instability. They include pressure fluctuations in screw [1], volatitilies, low molecular fractions of the polymer, fillers, poor dispersion of pigments [2], die swell [1, 3], processing near degradation temperature [4], dissimilar viscosities in blends [5], die design [6, 7, 8] etc.

At the present time, two basic types of die drool phenomenon are known: external and internal. It has been recently found that the external type of die drool phenomenon for metallocene type of LLDPE polymer is predominantly driven by the extrudate free surface rupture and suction effect due to negative pressure generated at the die exit region [9, 10] which can be suppressed by the die exit angle modification [9, 10]. On the other hand, the internal type of die drool phenomenon can be caused by the flow induced fractionation occurred before the polymer melt leave the extrusion die which is not fully understood yet [11]. Thus, the effect of processing conditions and die design on this unwanted flow phenomenon has been investigated in this work for unfilled HDPE extrusion through annular extrusion die.

Experimental

Material characterization

In this work, HDPE Tipelin FS 450-26 (TVK, Hungary) polymer, widely used in the film extrusion was chosen for the experimental research. The basic material characteristics are provided in Table 1 and [12]. In order to evaluate the HDPE polymer thermal stability, firstly, polymer pellets were added and melted in the Rosand RH7-2 control speed capillary rheometer equipped by capillary die having 16mm length and 1mm radius. After each 10 minutes of the rest, the constant shear rate ($80 \, \text{s}^{-1}$) has been applied and the equilibrium capillary pressure drop has been measured. This procedure was repeated 30 times to cover 5 hour degradation time period after which the extruded polymer has been collected. This procedure was applied for two different melt temperatures T = 210 °C and T = 260 °C. Effect of processing temperature and time on the extrusion pressure for the tested HDPE sample is provided in Figure 1. It is clearly visible that the extrusion pressure rise occurs during the time suggesting that temperature induced degradation leads to molecular weight increase of the tested polymer and the effect is much more pronounced for the higher temperature.

Die drool measurements

The die drool experiments were carried out on a conventional Brabender Plasti – Corder 2000 single-screw extruder with diameter D = 30 mm and L = 25 D (standard single-thread screw with compression ratio 4:1, and lengths of zones: feed L_1 = 10D, compression L_2 = 3D, metering L_3 = 12 D) which was included in the laboratory extrusion line. The schematic view of laboratory extrusion line is depicted in Figure 2. This line consists of extruder with four heating zones, transition annular part, specially designed extrusion die, which is introduced in [9, 10], photo camera placed near the die exit, and draw-off mechanism. In our experiments, two different processing conditions were used. In the first case, the extruder zones (from the hopper to the die) were heated to T_1 = 80°C, T_2 = 200°C, T_3 = 240°C and T_4 = 260°C whereas in the second case, third and fourth extruder zone temperatures were decreased to T_3 = T_4 = 210°C by keeping the annular tube (connecting die and extruder) and die exit temperature in both cases constant, T_5 = T_6 = 150°C. Die drool accumulation has been investigated for both temperature profiles along the screw and constant mass flow rate (1.1 kg.hr⁻¹). In this study, following extrusion dies were utilized: straight die, shaped die, flared die manufactured from stainless steel and straight die manufactured from hard chrome material (see Figure 5 for more detail). For each individual test, barrel, screw and all parts of the die have been perfectly cleaned to ensure the reproducibility of the performed measurements.

Results and discussion

The character of the die drool phenomenon for HDPE within the time has been investigated for the following processing conditions at which the die drool intensity was found to be the highest: temperature profile along the screw: $T_1 = 80^{\circ}\text{C}$, $T_2 = 200^{\circ}\text{C}$, $T_3 = 240^{\circ}\text{C}$, $T_4 = 260^{\circ}\text{C}$; annular tube (connecting die and extruder) and die exit temperature $T_5 = T_6 = 150^{\circ}\text{C}$; mass flow rate = 1.1 kg.hr⁻¹. The obtained results are depicted in Figure 3. It is clearly visible that the accumulated material takes a torus shape along the exrudate surface rather than flakes or powder as in the case of external mLLDPE die drool phenomenon [9-10]. Moreover, no surface defects occur during the HDPE die drool accumulation which lead us to the conclusion that rupture of the extrudate free surface at the die exit is not driving factor in this case (unlike for mLLDPE [9-10]).

The effect of temperature profile along the screw channel on the die drool intensity is visualized in Figure 4 for constant mass flow rate. It is nicely visible that the last two screw zone temperature increase from 210°C to 260°C causes significant die drool intensity increase even if the annular tube (connecting die and extruder) and die exit temperatures remains to be unchanged, equal to 150°C.

This behavior can be explained by the increased HDPE melt viscosity/elasticity due to thermally induced degradation (as showed in the performed rheological analysis) promoting flow induced fractionation of low molecular weight component of HDPE, which is in good correspondence with recent work on molecular weight fractionation during polymer processing [11] and theoretical study of internal die drool phenomenon [10]. In more detail, the studied die drool phenomenon can be understood through originally Busse's hypothesis [13] stating that during the flow, larger (highly elastic) chains acquire more elastic energy near the wall than small molecules which generates thermodynamic force tending to increase the concentration of small molecules at the wall (area of the high stress), and of the larger chains at the centerline (area of the low stress). In the light of this hypothesis, (where die drooled material can be viewed as the low molecular weight component of HDPE), increased die drool phenomenon through melt elasticity enhancement can be explained by the increased thermodynamic force driving molecular weight fractionation intensity during the flow.

The effect of die design and die material on the die drool phenomenon for HDDPE polymer melt has also been

investigated in this work. It was found that for the particular processing conditions and given time period, the stainless steel shaped die (22.2% die drool reduction), stainless steel flared die (46% die drool reduction) or the use of hard chrome straight die (20.2% die drool reduction) reduces die drool phenomenon with respect to reference stainless steel straight die. Die drool shape for the reference stainless steel straight die and the most stabilizing flared die is visualized in Figure 6.

Conclusion

In this work, the die drool phenomenon analysis has been performed for HDPE polymer melt by using specific type of experimental set-up. It has been revealed that the temperature increase at the end of the extruder, by keeping annular tube (connecting die and extruder) and die exit temperature unchanged, leads to die drool intensity increase. It has been proved that this type of the internal die drool phenomenon is caused by the thermally induced HDPE degradation enhancing melt elasticity and thus promoting the flow induced fractionation of the HDPE melt. It has also been found that for particular processing conditions, shaped die, flared die or the use of hard chrome instead of stainless steel can reduce die drool phenomenon for HDPE.

Acknowledgement

The support of the projects by the Ministry of Education CR (KONTAKT ME08090, MSM 7088352101) are gratefully acknowledged.

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Property	HDPE Tipelin
	FS 450-26
Density [g.cm ⁻³]	0.945
$M_{\rm w}$ [g.mol ⁻¹]	212,300
$M_{\rm n}$ [g.mol ⁻¹]	22,430
$M_{\rm z}$ [g.mol ⁻¹]	1,045,000
$M_{\rm w}/M_{\rm n}$ [-]	9.465
DSC peak melting point [°C]	130.3
DSC heat of fusion [J.g ⁻¹]	190.3
Crystallinity [%]	64.9

Table 1: Basic characterization of chosen polymer.

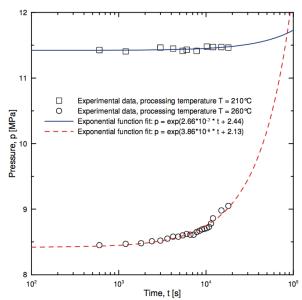


Figure 1. Effect of processing temperature on the time dependent extrusion pressure for tested HDPE Tipelin FS 450-26 sample.

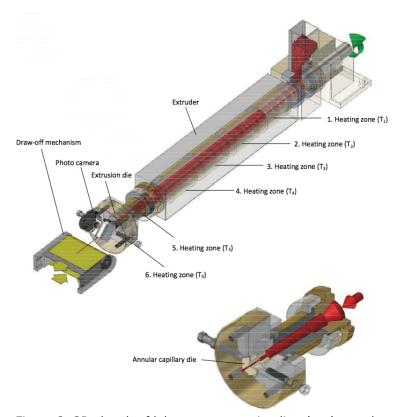


Figure 2. 3D sketch of laboratory extrusion line (top) together with detail view of the extrusion die (bottom).

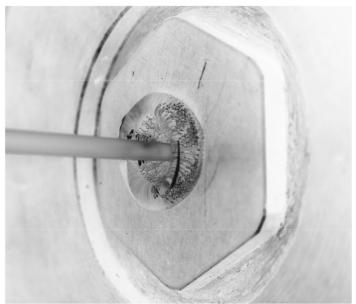


Figure 3. Die drool visualization at the die exit for the tested HDPE sample after 15 min of extrusion (temperature profile along the screw: $T_1 = 80^{\circ}\text{C}$, $T_2 = 200^{\circ}\text{C}$, $T_3 = 240^{\circ}\text{C}$, $T_4 = 260^{\circ}\text{C}$; annular tube (connecting die and extruder) and die exit temperature $T_5 = T_6 = 150^{\circ}\text{C}$; mass flow rate = 1.1 kg.hr⁻¹).

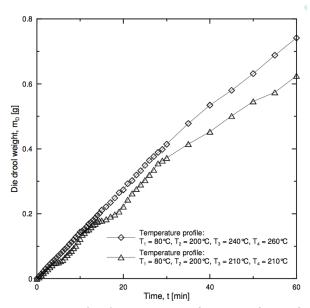


Figure 4. Die drool amount as a function of time for two different temperature profiles along the screw; mass flow rate = 1.1 kg.hr^{-1} , annular tube (connecting die and extruder) and die exit temperature $T_5 = T_6 = 150^{\circ}\text{C}$.

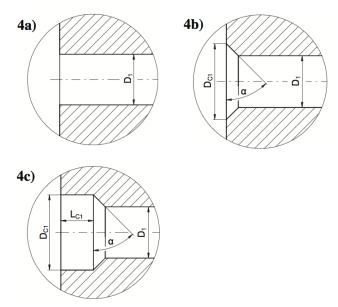


Figure 4. Investigated capillary dies, $D_1=1.6$ mm, alpha=45°. 4a) Straight die; 4b) Shaped die, $D_{C1}=2$ mm; 4c) Flared die, $D_{C1}=2$ mm , $L_{C1}=12$ mm.

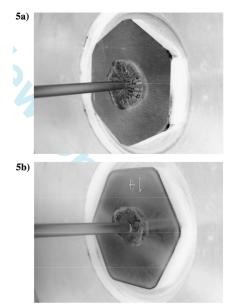


Figure 5. Visualization of die drool phenomenon at the die exit after 10 minutes of extrusion. 5a) Stainless steel straight die; 5b) Stainless steel flared die (temperature profile along the screw: $T_1 = 160^{\circ}$ C, $T_2 = 165^{\circ}$ C, $T_3 = 170^{\circ}$ C, $T_4 = 170^{\circ}$ C; annular tube (connecting die and extruder) and die exit temperature $T_5 = T_6 = 170^{\circ}$ C; mass flow rate = 0.9 kg.hr⁻¹).

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