DIE DESIGN EFFECT ON INTERNAL DIE DROOL PHENOMENON

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Abstract

In this work, the effect of die exit design on the *internal die drool* phenomenon occuring during extrusion of HDPE has been experimentaly investigated. It has been revealed, that firstly, the effect of flared length and die exit angle on the *internal die drool* intensity during extrusion of HDPE has non-monotonic character and secondly, flared dies are more stabilizing in comparison with chamfered dies.

Introduction

During extrusion process, there is a tendency for some of the extruded polymer materials to adhere to exit edges or open faces of extrusion die from which the extruded material emerges. The material so deposited on the die exit, can build up into a large compact usually degraded mass or can form drips, flakes or powder which frequently break away from the die, adhere perseveringly onto extruded product surface and thus damage it. This effect is in extrusion art defined as undesirable spontaneous accumulation of polymer melt at the die exit face and it is termed like "die drool", "drooling", "die lip build-up", "die bleed", "die plate-out", "die deposit", "die drip" or "die moustache" and the accumulated material is generally named "drool" [1, 2]. It is generally accepted that there are two types of the *die* drool phenomenon; "external die drool phenomenon", which is generated just at the end of the die due to the extrudate free surface creation and negative pressure generation (causing the suction effect) [2, 3] and "internal die drool phenomenon", which is initiated inside the processing equipment as the results of degradation [4] or flow induced molecular weight fractionation [5]. In fact, there is number of material, processing and die design based parameters promoting and suppressing the *die drool* phenomenon and some of them have been recently documented such as pressure fluctuations in screw [6], volatiles, low molecular fractions of the polymer, fillers, poor dispersion of pigments [8], die swell [4, 6], processing near degradation temperature [7, 9], dissimilar viscosities in blends [10], broadening molecular weight distribution [11-12], increasing melt elasticity [5, 7],

shark skin [2, 3], slip-stick phenomenon [5, 12-13], or abrupt corners at the die lips [3, 14, 15] have been found to promote *die drool*. On the other hand, chain branching increase [5], polymer processing aids addition [10], using [16] ceramics dies or dies with PTFE chemically/physically locked in die wall [8], silicon rubber coated surface of extrusion die [13], hard chrome dies [17], flared [3, 14, 15] or chamfered [3] die exits, have been found to reduce this phenomenon. With respect to the stabilizing role of the die design, it was initially believed that flared dies are so effective due to occurrence of stress undershoot inside flared section [1]. However, Ding et al. [18] mathematically modeled stress field inside the flared section and they concluded that stress undershoot is not the main reason for suppressing accumulation of *drool* mass at the die lips. They have suggested that the history of the stresses upstream of the exit, not just their instantaneous values at die lips, governs the *die drool* reduction in flared dies. Recently, Chaloupková and Zatloukal [3] were able to correlate stabilizing efficiency of die exit chamfering, die opening and die exit flaring with negative pressure, pressure gradient and normal component of the pressure gradient during extrusion of the metallocene based LLDPE at which the external die drool has occurred.

In this work, experimental analysis has been performed for extrusion dies at which chamfer angle and flared length were systematically varied in order to understand the role of die design for the internal type of *die drool* phenomenon as well as to explore the knowledge about parameters which could allow more efficient die design optimization.

Experimental

In this work, well stabilized unfilled virtually linear HDPE polymer melt (*HDPE Liten FB 29 E2009 3220 4479*, extrusion grade, Unipetrol RPA, Czech Republic, material characterization is summarized in our previous work [12]) has been used. The *internal die drool* measurements were performed on specially designed extrusion line equipped by replaceable capillary, which has already been used in our previous studies [5, 7, 12]. The line was consisted of conventional *Plasti – Corder*

2000 model (Brabender, Germany) single-screw extruder with diameter D = 30 mm and L = 25D (standard singlethread screw with compression ratio 4:1, and lengths of zones: feed $L_1 = 10D$, compression $L_2 = 3D$, metering L_3 = 12D), transition annular part, specially designed annular extrusion die, photo camera Canon 600D model (Canon, Inc., Japan) with resolution of 18 Mpx equipped with Canon macro lens EF 100mm placed near the die exit for die drool visualization and finally draw-off mechanism. The *die drool* experiments were performed as follows. Extruder zones (from the hopper to the die) were heated to $T_1 = 150^{\circ}$ C, $T_2 = 155^{\circ}$ C, $T_3 = 160^{\circ}$ C and $T_4 = 160^{\circ}$ C, respectively by keeping the annular tube (connecting die and extruder) and die exit temperature constant, $T_5 = T_6 =$ 160°C. The low exiting temperature 160°C for the HDPE melt was chosen to achieve highly pronounced fractionation effect due to high efficiency of melt to store elastic energy as well as to suppress any thermal/oxidative degradation. Further, mass flow rate (MFR) was chosen 750g.hr⁻¹ (apparent shear rate 673 s⁻¹) to ensure that the flow condition lies above the *slip-stick* phenomenon, i.e in the "superflow" regime at which the *die drool* intensity is the highest as shown in [12]. In order to measure fractionated (*drooled*) polymer mass accumulated at the die exit, the following methodology was used. The extruder was stopped after 10 minutes of extrusion and the accumulated material was carefully manually removed from the die lip by a tweezer, weighted on a sensitive analytical balance and the procedure was repeated three times for each capillary to calculate standard deviation. Before each set of three independent 10 minutes tests (for given capillary), barrel, screw and all parts of the die have been disassembled and perfectly manually cleaned. Finally, die drool intensity has been expressed in dimensionless form through buildup ratio BR (introduced by Gander and Giacomin in [1]):

$$BR = \frac{B}{\dot{m}} \tag{1}$$

where \dot{m} is total mass flow rate of extruded polymer melt and \dot{B} means buildup rate:

$$\dot{B} = \frac{B}{t_{\rm e}} \tag{2}$$

where *B* is the mass of accumulated *die drool* material on the die exit face and t_e is total extrusion time of each test (10 minutes in our case). To evaluate die exit design effect on accumulated *drool* mass following stainless steel capillaries were used. The <u>straight capillary</u> with abrupt die exit edge angle of 0° having diameter $D_1 = 1.6$ mm and L/D ratio of 9.375 (see Figure 1a) and its two modifications: <u>chamfered die</u> (see Figure 1b) with three different die exit angles ($\alpha = 15^\circ$, 30° and 45°) and <u>flared</u> <u>die</u> (see Figure 1c) with four different flared lengths ($L_{C1} = 1$ mm, 2mm, 5mm, 8mm) and one fixed die exit angle ($\alpha = 45^{\circ}$). For both modified dies, the outer diameter D_{C1} was kept to be 2mm.

Results and Discussion

The effect of die exit angle and flared length (expressed here in the dimensionless form as the flared length, L_{C1} , divided by the capillary length, L = 15 mm, i.e. by L_{C1}/L ratio) on the dimensionless buildup ratio is provided in Figure 2a and Figure 2b, respectively. As can be seen, this dependence has interestingly strongly nonmonotonic character and secondly, flared dies are more stabilizing in comparison with chamfered dies. In more detail, there is one minimum for 15° die exit angle (representing reduction of accumulated drool mass intensity by 56%) and one minimum at 2/15 dimensionless flared length (for which drool mass reduction is the highest, equal to 97%). The possible explanation of non-monotonic buildup ratio vs. die exit angle dependence could be understood through "detachment point" and "effective length", L_{eff.}, location as depicted in Figure 3. Here the detachment point represents location at which extrudate detaches metal die (due to the melt pressure/normal stresses at the die exit, adhesion at metal wall/flowing melt interface and extensional stress induced by the extrudate draw off) whereas the *effective length*, L_{eff} , is the distance in which fractionated low molecular weight species are in touch with the extrudate free surface as well as with the die wall. Clearly, if the L_{eff} is high, there is high probability that the low molecular weight fractioned polymer is effectively removed from the die exit region by the moving extrudate (because it happend under high pressure) and only small portion of it remains at the die exit face. This could happen for the most stabilizing case (i.e. die exit angle equal to 15° as visualized in Figure 3b). On the other hand, a decrease in the die exit angle down to 0° or increase it up to 30° or 45° could continuously decrease the $L_{\rm eff.}$ down to zero resulting to the extreme situation at which the low molecular weight fractionated polymer is not removed from the die exit region and only its intensive accumulation takes place at the die exit face resulting in intensive die drool phenomenon.

The flow situation inside flared section can also be viewed as the special case of two-layer coextrusion (see Figure 4) at which the low molecular weight species (formed due to flow induced fractionation) represents the low viscosity skin and the main polymer the high viscosity core (flow viscosity curves measured on Advanced Rheometrics Expansion System ARES 2000 for both layers are provided in Figure 5). Due to abrupt channel opening at the die exit, low enough pressure, normal stresses, adhesion and high extensional stress from draw off *"interface expansion"* could take place

during which the low molecular weight layer decelerates, expands in thickness and core layer starts to be separated from the skin layer (detachment point) far away from the die exit (effective length is high) at which low molecular skin layer becomes intensively dragged with the core extrudate layer out of the die, thus suppressing its building up at the die exit face. Thus, increase in the *effective length*, L_{eff} , for given flared die leads to more intensive drool suppression. It seems that for the given HDPE polymer melt and chosen processing conditions $L_{\rm eff}$ has maximum at 2/15 dimensionless flared length due to low pressure and low normal stresses, which increase the effect of the extensional stress from the draw-off on the flow field in the upstream direction, i.e. the detachment point is moved in the upstream direction too, which is stabilizing. On the other hand, for the longer dimensionless flared sections (5/15, 8/15), detachment point presumably starts to move closer to the die exit (i.e. $L_{\rm eff}$ decreases) due to increasing pressure and the normal stresses in flared section resulting in smaller efficiency of the extensional stress from the draw-off to influence the flow field in the upstream direction i.e. the position of the detachment point is moved more closer to the die exit, which is destabilizing.

Conclusion

It has been revealed experimentally, that the effect of die exit angle and flared length on the internal die drool intensity during extrusion of HDPE has non-monotonic character. It has been found that the optimum value for the die exit angle and dimensionless flared length is 15° and 2/15, respectively. It has been proved that flared dies are more stabilizing in comparison with chamfered dies. In more detail, 56% and 97% reduction in accumulated drool mass has been achieved with respect to straight die having abrupt die exit edge angle of 0° for the optimum value of die exit angle and dimensionless flared length, respectively, for the HDPE material and given processing conditions. It has been suggested that suppresion mechanism of the internal die drool phenomenon through die exit modification can be understood throught the balance between the melt pressure/normal stresses at the die exit, adhesion at metal wall/flowing melt interface and extensional stress induced by the extrudate draw off, which can lead to flow situation at which low molecular weight species are effectively removed from the die exit region by the moving extrudate and only small portion of them remains at the die exit face. This work suggests that for the simulation based die design optimization with respect to internal die drool phenomenon, fully viscoelastic constitutive equation together with proper boundary conditions (including adhesion forces between die wall and polymer melt and considering the extensional stresses due to extrudate draw off) should be utilized in

order to precisely determine the location inside the die at which free surface of the extrudate starts to occur.

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Figure 1: Detailed section views of capillary exit regions: (a) straight capillary, (b) chamfered die, (c) flared die.



Figure 2: Effect of die exit design on dimensionless buildup ratio: (a) die exit angle effect, (b) dimensionless flared length effect.



Figure 3: Effect of die exit angle on extrudate detachment and accumulation process of low molecular weight mass for (a) 0° , (b) 15° , (c) 45° dies.



Figure 4: Visualization of the low viscosity skin (die drool layer) and high viscosity core (main polymer) inside and outside the die for the optimum 2/15 dimensionless flared length.



Figure 5: Frequency dependent complex viscosity for the die drool (skin) and main polymer (core) samples.