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Molding via Rheological Control During Plastication and Purging

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Purging an injection molding machine is a vital procedure for every molder when switching from one resin formulation to another or when simply shutting down the machine. Complete removal of primary resin and hang-up of degraded material is necessary to achieve good production runs. The purpose of this study was to examine the dependence of rheology of a commercial purging compound on parameter settings during plastication of the shot. The results have been used to research the purging efficiency when purging materials such as polyamide-6,6 which is immiscible with the base resin of the purging compound. The results of the study showed that the purging efficiency depends on the flow induced morphology of the multi-phase system in the nozzle and on the content of undissolved gas in each purge shot. The latter proved to be controllable by the parameter settings during shot build-up. Purging of the screw depends on the viscosity and melting point of the materials used.

Keywords purging, purging compound, polyamide-6,6

Introduction

Although the improvement of injection molding machines and molding techniques can improve productivity and quality, there remain some problems that reduce productivity during molding machine purging when color or material changes are required. The efficiency of purging of an injection molding machine depends on the purging material or compound and on the process conditions. As a result, a great deal of literature has been published (1–10), that compares different materials or describes the superiority of a certain compound. However, purging efficiency should also depend on the rheology of the primary resin, purging compound, and follow-up material, as well as the operating procedure. This becomes especially true, when two-phase and multi-phase systems exist. Therefore, it is surprising that there is no available literature on the rheological background of purging.

The purpose of this study was to determine the rheological behavior of a hybrid purging compound and its dependence on processing parameters such as screw speed, back pressure,

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barrel temperature, and soak time. A closer look at purging of polyamide was made, because of difficulties in purging the material due to its small processing window and its immiscibility with HDPE (high density polyethylene). The results of this research should be used to improve the removal of primary resin by the purging compound as well as the removal of the purging compound itself by the follow-up material. This investigation also forms the background for future work-the purging of hot runner manifolds.

An improvement in purging will lead to real economic benefits. On the one hand, faster purging saves labor and machine cost and on the other hand, more efficient purging saves material and reduces scrap parts.

Materials

The materials used in this study are purging compound, polyamide -6,6, black color concentrate, manganese dioxide (MnO_2) and gold wire. The purging compound, PurgexTM 3056 Plus is a commercially available purging compound consisting of 82.5% PE as a base-resin, 7.5% PurgexTM 527 and 10% modified PE. PA-6,6 has hygroscopic properties and had to be dried prior to processing. The drying conditions: drying temperature ($^{\circ}\text{C}$) and drying time (h) for the resin are 80°C and 5 h, respectively. Rheological data for PA-6,6 material was found in the Moldflow[®] materials data base (11). Figure 1 shows the shear rate – viscosity relationship for PA-6,6.

The temperature dependence of the viscosity at a shear rate of 1,000 1/s for HDPE as base resin of PurgexTM 3056 Plus, and polyamide-6,6 is shown in Figure 2.

DOE to Determine the Rheology of PurgexTM 3056 Plus

PurgexTM 3056 Plus is one of the hybrid purging compounds that combines physical and chemical cleaning actions. The key point to accomplish both, is the incorporation of a

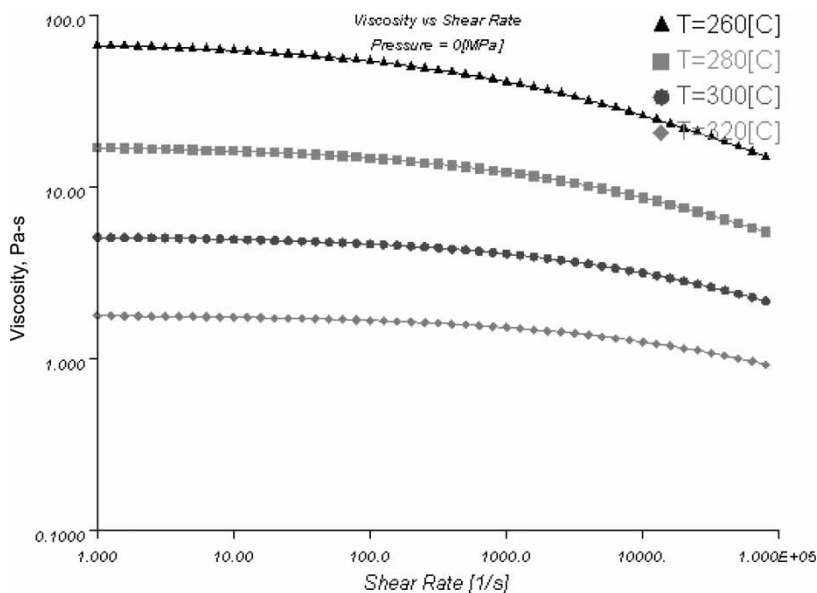


Figure 1. Shear rate dependence of the apparent viscosity (y-axis) for PA-6,6.

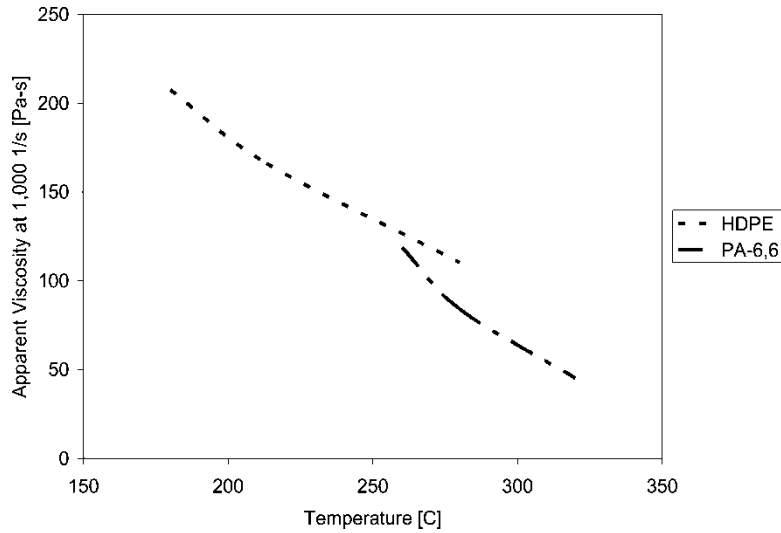


Figure 2. Temperature dependence of viscosity at a shear rate of 1000 1/s for HDPE (Purgex™), and PA-6,6.

foaming agent and the degree of foam formation. Thus, the rheology of Purgex™ 3056 Plus is difficult to determine, and the processing conditions have a large influence on the gas content in the melt and on the rheology of the purging compound. To determine the effect of certain injection molding process variables on the degree of foaming and thus, on the rheology of Purgex™ 3056 Plus, a full factorial design with four factors on two levels was performed. This design led to $2^4 = 16$ different process combinations. The chosen factors were back pressure, screw speed, barrel temperature, and soak time. Back pressure and screw speed were chosen because both have a significant influence on the amount of shear heating generated within the purging compound. Barrel temperature was chosen because the onset of gas liberation by the foaming agent is temperature dependent. As fourth factor, soak time was chosen because many different recommendations for soak time can be found in the literature, but a rheological dependence has not yet been specified. Table 1 reports the settings used for the low and high level of each factor.

Evaluation of Purging Efficiency

The efficiency of purging was evaluated by following the material change using MnO_2 as tracer in the primary resin. Therefore, MnO_2 powder was melt compounded into the

Table 1
Level settings for each factor

Level	Back pressure [MPa]	Screw speed [rpm]	Barrel temperature [°C]	Soak time [min]
Low (–)	0	90	280	0
High (+)	0.345	150	320	5

primary resin. A certain amount of the MnO_2 masterbatch was run through the injection molding machine and a soak time was applied to simulate a long production run with material hang-up and degradation in the plasticating unit. After the soak, different purging procedures and materials were used to remove the primary resin and potential hang-up. The purge was collected in strand form by injecting into a water bath. As a result, two centimeter long samples could easily be cut out of the purge shots. In addition, the position of each sample could be exactly determined by a cumulative mass balance. The cut-out samples were weighed and placed into plastic vials to be neutron activated in a nuclear reactor. A high purity Germanium detector was used to measure the activity of the samples and a gold wire activated with each sample was used to normalize for different activation conditions. By using commonly known nuclear science equations, the amount of manganese in a sample could be calculated from the activity of each sample. Plots of manganese concentration in the purge-outs via time and cumulative mass allowed one to evaluate the efficiency of the purging procedure.

Purging Procedure

Purging trials were performed to evaluate the purging efficiency of a resin change. As a primary resin, either a polycarbonate or polyamide-6,6 masterbatch with MnO_2 as tracer were used. To purge the primary resin Purgex™ 3056 Plus or the follow-up, neat resin was used. For purging with Purgex™ 3056 Plus, the purging procedure as recommended by Neutrex and also an alternate procedure were used. The alternate procedure was based on the results of the DOE (design of experiments) performed to determine the effects of back pressure, screw speed, barrel temperature, and soak time on the relative viscosity of the purging compound.

A purging agent or neat follow-up material was added to the preconditioned injection molding machine. With a purging agent, a certain purging procedure was followed. With a neat resin, the tracer masterbatch was purged from the system with follow-up material only. After each shot, the nozzle tip was cleaned with copper gauze.

Neutrex Procedure (12)

Neutrex recommends to use one and a half times the machine's barrel capacity of purging compound. That means the injection molding machine used an amount of 1.25 lbs (567 g) Purgex™ 3056 Plus. A screw speed of 120 rpm was used for melt plastication. Back pressure was set to a value of zero MPa. An injection speed of 25.7 mm/s was used to ensure the formation of a strand in the water bath. The injection pressure was set to 6.9 MPa. The maximum shot size of 120 mm (122 cm^3) was used.

After loading the purging compound into the hopper, four shots were made and ejected. The fifth shot was plasticated, but not ejected until 5 minutes of soak time passed. Further shots were made to clear the remaining Purgex™ from the barrel. Immediately after the hopper emptied out, the follow-up resin was added to the hopper to avoid air from entering the barrel, as recommended by Neutrex. When purging the polycarbonate masterbatch, polycarbonate was added as a follow-up resin and when purging polyamide-6,6, it was used as secondary resin. Using the follow-up resin purging was continued until 30 shots had been made.

Table 2
Parameter settings for the Purgex™ shots

Shot number	Relative viscosity	Screw speed [rpm]	Back pressure [MPa]
1–3	High	150	0.345
4–	Low	90	0

Alternate Procedure

This strategy first uses a high viscosity purging with high shear stress at the wall to scour and push most of the primary resin from the machine. Afterwards, a low viscosity purging compound with a high degree of foaming should allow the purging melt mass to reach into dead spots and hang-up zones to remove all hang-up that could lead to black specks and streaking.

The results of the DOE showed that the relative viscosity of Purgex™ 3056 Plus is controllable by the process parameter settings. Therefore, the first three Purgex™ shots were made with parameter settings that led to a high relative viscosity. The subsequent shots were made with low viscosity settings. The settings of screw speed and back pressure for the shots with Purgex™ 3056 Plus are reported in Table 2.

A second difference vs the standard procedure was to eliminate the soak time. All shots were made immediately one after the other. The DOE results showed that soak time had neither a significant influence on the relative viscosity nor on the melt temperature.

A third difference from the standard procedure was to empty out the plasticating unit of purging compound completely before adding the secondary material. The reason was to avoid back-mixing of purging compound with the follow-up resin and thus avoid increasing the amount of follow-up material and time required to remove the purging material.

Results and Discussion

DSC scans were performed to determine the decomposition temperature of the foaming agent masterbatch, Purgex™ 527. Figure 3 shows an overlay of two DSC cycles of the same sample.

As one can see, two endothermic peaks are observed for the first cycle. These peaks are no longer detected for the second cycle of the sample. It was concluded, that during the primary heating of the sample, an irreversible process occurred. This irreversible process is most probably the decomposition of the foaming agent. The onset temperature of the decomposition reaction was detectable at 70°C as shown in Figure 4.

The second endothermic peak could be a further decomposition or phase change of products of the primary decomposition reaction. A decomposition of the foaming agent at 70°C would also explain the storage recommendations for Purgex™ 3056 Plus. The manufacturer recommends storage below 66°C (13). The significance of this relatively low decomposition temperature for processing is the possibility that gas can escape out of the feed throat. If the foaming agent decomposes before the resin starts melting, the liberated gas cannot be dissolved in the melt. However, it could escape backwards through the deep channel of the solids conveying zone in the screw because a melt plug

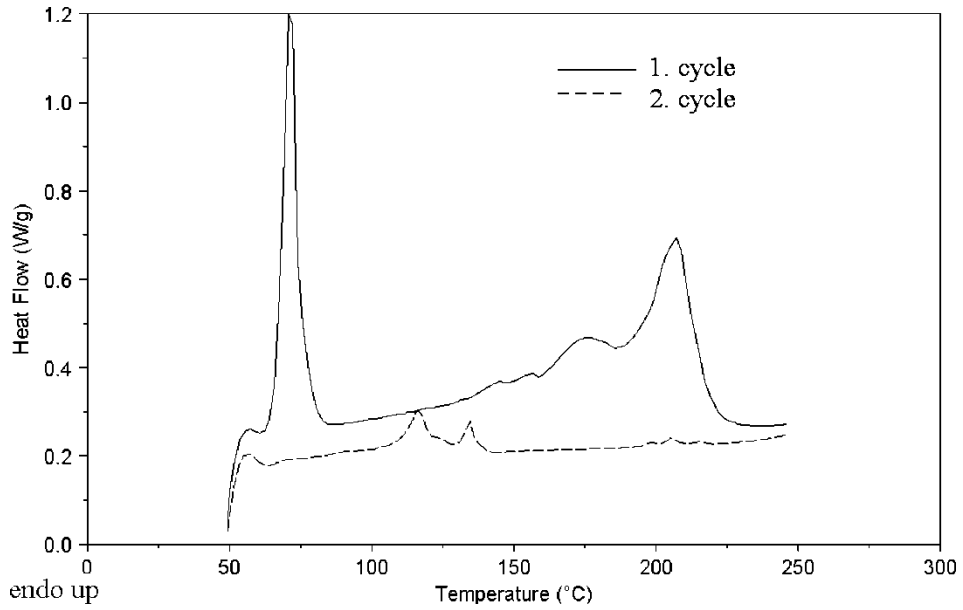


Figure 3. Overlay of two heat cycles for the same sample of PurgeX™ 527.

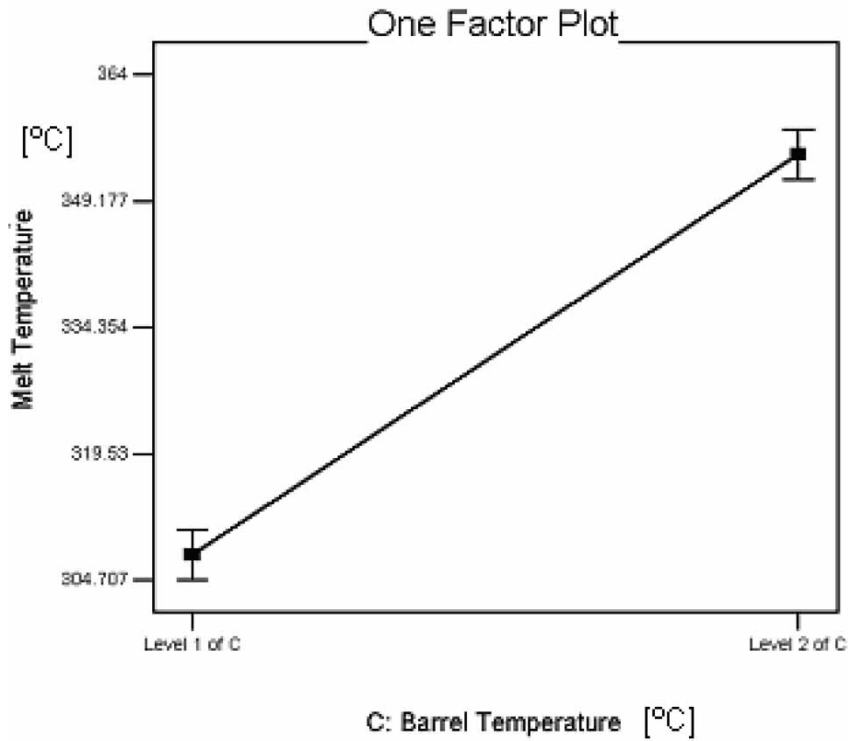


Figure 4. Effect of barrel temperature on melt temperature (°C), (Level 1 of C = 280°C and Level 2 of C = 320°C).

has not yet formed. Possibly, the amount of dissolved gas in the melt can be controlled by the processing conditions.

DOE Results

All the average values, standard deviation (SD), and coefficient of variance (COV) of the melt temperature, shot weight, and relative viscosity for each process combination are collected while performing the designed experiment.

Effects on Melt Temperature

The effects on melt temperature showed a signal to noise ratio of 25.0. A ratio greater than 4 is desirable. Table 3 reports the significant effects of the process parameters on melt temperature. Back pressure is represented by A, screw speed by B, barrel temperature by C, and D stands for soak time.

“+” represents an increase in melt temperature with increasing setting of the parameter. “-” represents a decrease in melt temperature. The values of the effect show the amount of increase or decrease starting from the mean value. For example, the melt temperature increased by 20.5°C with an increase of parameter C (barrel temperature) from the midpoint (300°C) to the high level of the setting (320°C). As another example, the melt temperature increased by 1.25°C with an increase of parameter A (back pressure) from 0.1725 to 0.345 MPa. The results of the DOE with melt temperature as a response showed that barrel temperature had the largest effect. The effect of barrel temperature on melt temperature is visualized in Figure 4.

The effects of the other parameters and the interactions of the parameters on melt temperature are very small compared to that of barrel temperature. In addition, the soak time, D, had no significant influence on melt temperature. This may be due to the fact, that most thermal energy comes into the system by viscous dissipation. The melt temperature is already at a high value after plastication and no further significant conductive heating of the plasticated shot happens during the soak time.

Effect on Shots Weight

The effects on shot weight showed a signal to noise ratio of 14.5. A ratio greater than 4 is desirable. For shot weight, the smallest signal to noise ratio was determined. The low value

Table 3
Significant factors on melt temperature as a response

Factors	Value [°C]
A	+1.25
C	+20.5
BC	-1.67
CD	-1.25
ACD	+1.21
ABCD	-1.13
Melt temperature mean	330.2°C

Table 4
Significant factors on shot weight as a response

Factors	Value [g]
A	+17.19
B	+1.73
C	-4.25
AC	+3.28
CD	-2.49
ABC	+2.04
ACD	+1.89
Shot weight mean	74.37 g

could have been caused by some difficulties in catching all ejected melt. Table 4 reports the significant effects of the process parameters on shot weight.

As shown, back pressure, A, had the largest significant effect on shot weight. For the high back pressure level, a shot weighed 17.19 g more than at the medium setting. Shot weight is a direct measure of melt density, because each shot had the same volume in the barrel. Thus, a high shot weight means a high melt density, small volume of gas and little foaming. An increase in screw speed, B, also led to an increasing shot weight whereas an increasing barrel temperature, C, led to a decrease in shot weight. Soak time, D, had no significant influence on the shot weight.

Effects on Relative Viscosity

The effects on relative viscosity showed a signal to noise ratio of 28.0. A ratio greater than 4 is desirable. Table 5 reports the significant effects of the process parameters on relative viscosity.

Barrel temperature C had the largest effect on relative viscosity. The reason is, that with increasing barrel temperature, the melt temperature increased. An increase in melt

Table 5
Significant factors on relative viscosity as a response

Factors	Value [MPa-s]
A	+11.49
B	+4.72
C	-26.05
AC	-9.47
BC	-3.93
CD	-2.54
ABC	-2.05
ABD	-3.24
ABCD	+2.62
Relative viscosity mean	113.25 [MPa-s]

temperature leads to a decrease in melt viscosity at constant pressure and shear rate as was given for the trials using a constant ejection speed.

The second largest effect on the relative viscosity was back pressure, A. An increase in back pressure led to an increase in relative viscosity. The explanation can be found by recalling the effect of back pressure on shot weight. An increase in back pressure led to a higher melt density due to a lower volume of free gas. Without high shear and mixing resulting from high back pressure and screw speed, a two phase system of gas and polymer melt forms. The gas bubbles expand inside the barrel and nozzle as melt is ejected due to the pressure gradient that causes flow and thus they accelerate the total melt mass and lower the relative viscosity. Again, soak time D had no direct significant effect. The soak time neither influenced melt temperature nor the formation of a gas phase. As explained above, melt temperature and the existence of a gas phase both had an influence on the relative viscosity.

Purging of Polyamide-6,6

Polyamide-6,6 as primary resin was purged from the machine using PurgeX™ 3056 Plus in combination with the Neutrex and alternate procedure and using polyamide-6,6 only. Figure 5 shows the normalized mass fraction C/C_0 of manganese in the samples versus the shot number of the trial for the three different purging trials of polyamide-6,6.

The results of the neutron activation showed that using the Neutrex procedure took 19 shots to remove the primary resin. Applying the alternate procedure took 21 shots. To remove the primary resin with neat polyamide, 26 shots were necessary. A possible explanation for the difficulties in purging polyamide-6,6 with the purging compound, can be found by recalling the flow induced morphology of two- and multi-phase systems.

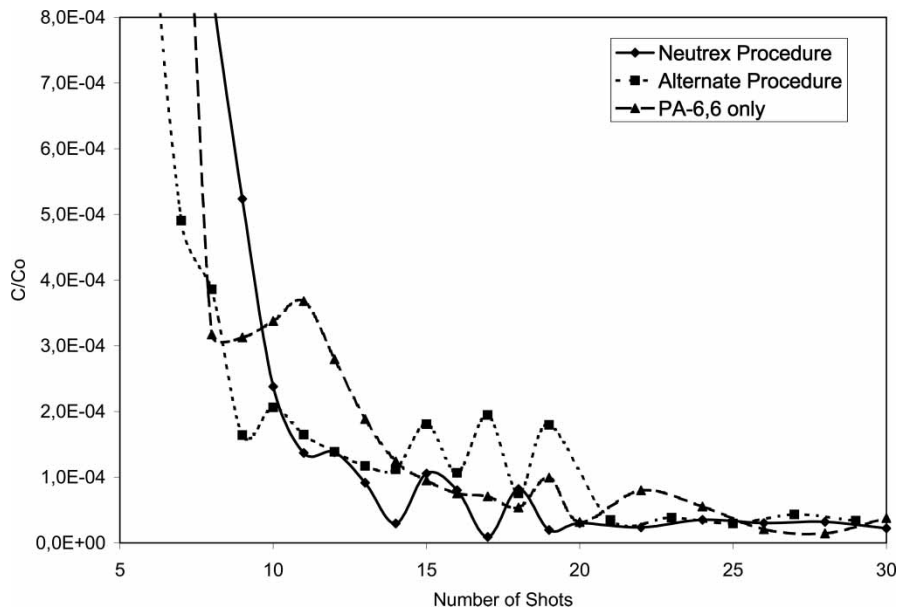


Figure 5. Plot of normalized manganese concentration vs. shot number for PA-6,6.

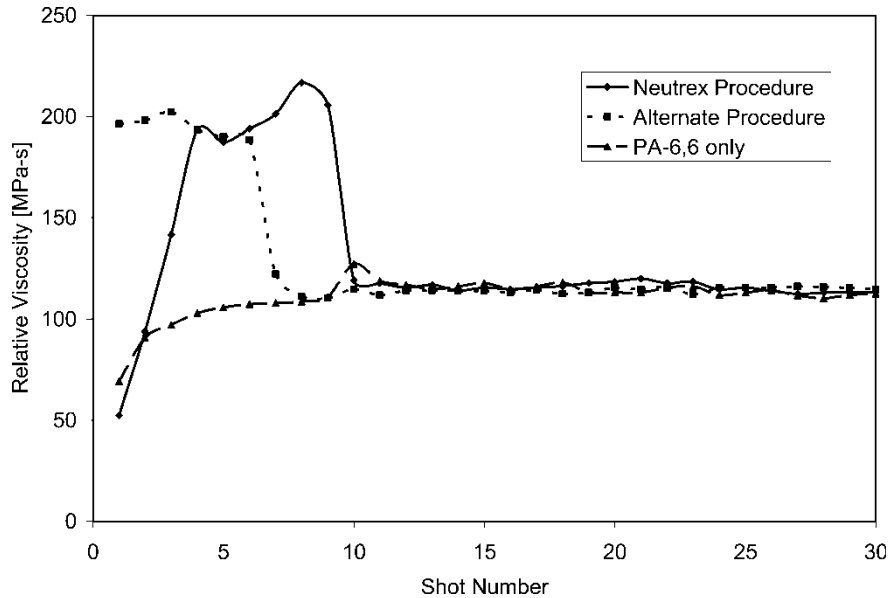


Figure 6. Relative viscosity vs. shot number as measured for all three purging procedures when purging polyamide-6,6.

The minor component of an immiscible blend accumulates in the center of the flow when it has a higher viscosity than the major component and vice versa. When purging PA-6,6, the minor component has a lower viscosity than the major component as can be seen in Figure 6, which shows the relative viscosity for the purging trials of PA-6,6.

As a result, the minor component which has to be discharged migrates to the high stress region of flow – the flow channel boundary – to lubricate the flow of the major, high viscosity component. Thus, the purging compound becomes lubricated by the primary resin instead of dragging out the primary resin. When purging PA-6,6, more shots to remove the primary resin were necessary using the alternate procedure vs. using the Neutrex procedure. A possible explanation may be the composition of each purge shot. Each purging compound shot contains a certain amount of undissolved gas depending on the value of back pressure and screw speed applied. The undissolved gas leads to a multi-phase flow morphology. The expansion of the gas bubbles increases the flow due to gas expansion greatly reducing the viscosity of all components. When a two-phase system of a minor component PA-6,6 and a major component of HDPE exists as in purging of PA-6,6 with HDPE, the low viscosity PA-6,6 migrates to the wall where it becomes difficult to remove. However, when the purging system contains a second minor phase with a lower viscosity than PA-6,6, as for example, undissolved gas bubbles, the gas bubbles “lubricate” the flow and the PA-6,6 is more readily removed. The three first shots of the alternate procedure are to built-up using back pressure and screw speed settings that lead to only few undissolved gas bubbles in the system. The Neutrex procedure uses back pressure and screw speed settings that lead to a higher amount of undissolved gas bubbles in the system. As a result, using the Neutrex procedure takes fewer shots to remove the PA-6,6 primary resin from the barrel and nozzle. Applying back pressure during plastication can lead to early solid bed break-up and difficulties in primary resin removal. When purging a low viscosity

melt like that of polyamide-6,6 back pressure also leads to an increase in leakage flow over the top of the screw flights and back mixing. The dependence of output, Q , on viscosity and back pressure was also observed in extrusion and can be expressed by:

$$Q = A * N - ((B * \Delta P) / \mu)$$

Where A and B are system constants. N is the screw speed, ΔP is the back pressure in the system, and μ is the apparent viscosity. When a low melt viscosity exists and the back pressure is set to a certain value, the second term of the equation becomes significantly and lowers the output. This means, that with a low melt viscosity and a certain value of back pressure as applied when purging polyamide with the alternate procedure the residence time increases.

Conclusions

As DOE results were reported, the melt temperature was mainly influenced by barrel temperature. An increase in shear heating by increasing the back pressure and screw speed also led to an increase in barrel temperature. Most thermal energy came into the system during shot build-up, so that soak time had no significant influence on melt temperature. Melt density and relative viscosity were strongly influenced by gas liberated from the foaming agent of the purge compound. DSC measurements showed that the onset temperature of foaming agent decomposition and gas liberation was about 70°C. As a result, gas is liberated in the feed zone of the plasticating unit. The amount of gas dissolved in the melt can be controlled by the setting of back pressure and screw speed as the DOE results showed. Both parameters influence how rapidly the base polymer of the purging compound melts and is able to dissolve the liberated gas. With high shear, as caused by high back pressure and screw speed, a sufficient amount of melt could be built-up to dissolve the gas. Lower settings of these parameters led to the formation of undissolved gas bubbles and gas escaping out of the feed throat. As the melt progresses forward during plastication, the melt pressure increases due to the restriction of the check valve. This causes some or all of the gas to dissolve. However, if the accumulated melt shot has gas that separates from the melt, this gas will generate pressure that pushes the screw backwards. This causes further gas formation and leads to an incomplete plasticated shot where melt has been displaced by the gas volume. Thus, the formation and expansion of undissolved gas bubbles caused the screw to move backwards. As a result, plastication time decreased and less melt accumulated in front of the screw-tip, leading to a low shot weight. The undissolved bubbles also led to a decrease in viscosity during ejection, as can be explained by the flow morphology of two-phase systems and the principle of energy minimization. The lower viscosity component – the gas bubbles – migrate to the high stress region of flow – the barrel wall – expand as melt ejection starts. During purging, the gas expands due to the pressure gradient that generates the flow. The expanding gas accelerates the melt causing a further reduction in viscosity and a rapid violent purge.

The flow morphology also influences the efficiency of purging polyamide-6,6 from the barrel and nozzle as the results of the performed purging trials showed. In general, it was difficult to remove PA-6,6. The reason for this may be that PA-6,6 has a lower melt viscosity than the purging compound. Polyamide-6,6 is immiscible with the purging compound and forms a two-phase system. When purging PA-6,6, the lower viscosity minor phase migrates to the high shear stress region of flow to lubricate the

flow of the purging compound. Thus, PA-6,6 was more difficult to remove from the machine. However, when purging a low viscosity material such as polyamide-6,6 from the barrel and nozzle, it is advantageous to use high undissolved-gas-content parameter settings as the results showed. The undissolved gas has the lowest viscosity in the three phase system and thus dominates the role of flow “lubrication”. This role will be played by the PA-6,6 if no gas phase exists, resulting in a difficult to remove primary resin.

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