Analysis of process parameters related to the single screw extrusion of recycled polypropylene blends by using design of experiments

Tingrui Fu[1], Barry Haworth[1] and Leno Mascia[1]

Abstract

The process dynamics of single-screw extrusion on mixtures of polypropylene (PP) and recycled PP were studied using a statistical, design of experiments (DoE) approach. For a conventional screw design, barrel temperature, screw speed and two vastly different melt viscosity PP mixtures were selected as the independent factors, whilst melt pressure, mass output, screw torque and temperature rise at the die due to shear heating, were the dependent responses. A central composite design (CCD) in the framework of response surface methodology (RSM) was constructed, and an analysis of variance (ANOVA) was carried out to determine the significance of the response surface models. The resulting statistical and response surface predictions have demonstrated that the low viscosity component concentration in the blend is a dominating factor on melt pressure and screw torque, apart from the expected effect of screw speed on output. Viscous heating is affected only by screw speed and recycled PP concentration. Furthermore, the predictions have identified a wider process operating window with increased low viscosity component concentration. The data confirms that statistical tools make quantitative predictions for the effects of experimental process variables, in accordance with the expected qualitative trends towards process optimisation, providing scope towards its application in scaled-up industrial processes.

Keywords

Single screw extrusion, process dynamics, recycled polypropylene (PP), design of experiments.

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Introduction

Single screw extrusion is by far the most widely used processing method in the polymer industry and is the basis for manufacturing an extensive product group (film, sheet, pipe, profile, coatings) for applications in many industrial sectors.[1-4] A polymer's unique characteristics can also be exploited through solid state processing (molecular orientation) using ram extrusion, although Mascia[5] reported that the products that can be manufactured by this method is very limited. Wilczyński[6] developed a predictive model to describe how an extruder-die system operates for a conventional single-screw process. This model enables researchers to predict mass flow rate, pressure and temperature profiles along the screw channel and in the die, solid bed profile in the feed zone, power consumption and also to obtain estimates for mixing degree and temperature fluctuations. Later, the process model was extended to include single screw extruders with non-conventional screw designs, determining that extruder output is independent of screw speed during starve-fed processes and that the melting mechanism is also completely different, relative to flood-fed conditions.[7] The authors then developed the first composite model for starve-fed single screw extrusion processes, which involves metering, melting and solid conveying.[8,9]

Derezinski[10] analysed the heat transfer in single screw extrusion to determine the melt temperature resulting from the combined effect of adiabatic heating generated by the screw and conduction heating by the barrel. He showed that the screw temperature can differ considerably from the melt temperature, particularly near the melting section inlet. He also predicted that the screw temperature never exceeds the melt temperature over the entire screw length. Abeykoon et al.[11] proposed a non-linear model to predict the die melt temperature profile and demonstrated that substantially reducing melt temperature variations are possible. In subsequent studies, Deng et al.[12] introduced new real-time energy monitoring methods to investigate how process settings affect energy efficiency and melt consistency. Developed from the initial studies reported by Kelly et al.[13], Vera-Sorroche et al.[14,15] measured thermal homogeneity and energy efficiency in extrusion processes, which relate to extruder screw geometry, screw rotation speed and polymer rheological properties. This approach also considered thermal conduction, convection and viscous dissipation effects over a range of process conditions, using dimensionless groups to quantify these effects. Lozano et al.[16] reported conditions that promote the beta crystalline phase in extruded PP and thereafter Navarro-Pardo et al.[17] investigated shear effects on beta-phase formation in isotactic PP, using different breaker plates at the extruder exit and by varying screw speed. Haworth and Ratnayake[18] measured many PP compound rheological properties, including the plasticising effect of low molecular weight polar additives,[19] the mechanism for which was subsequently proposed by Ratnayake et al.[20]

Design of Experiments (DoE) has proved to be a powerful statistical technique when dealing with multi-factor systems. Montgomery[21] defines this analytical technique as a test or series of tests in which purposeful changes are made to selected input variables of a process so that corresponding changes in the output responses may be observed, identified and then used in a predictive mode. Manufacturing processes relating to polymer-based products have been investigated using DoE methodologies; examples include blow moulding (Tahboub and Rawabdeh),[22] electrospinning of nanofibers (Coles et al.[23], Tsimpliaraki et al.[24]), mixing and blending processes for polyolefin compounds (Ramos et al.[25], Teixeira et al.[26]) and polymer-based composites (Costa et al.[27], Jang and Lim[28], Rocha et al.[29]). Other reported applications of DoE in process research have included extruded pharmaceutical products (Désiré et al.[30]) and UV-curable coatings (Kim et al.[31]). Relatively few studies have been conducted, on the other hand, to analyse single screw extrusion of polymers using a DoE approach. In their studies on low density polyethylene (LDPE) / thermoplastic elastomer blends, Borgaonkar and Ramani[32] employed a simple statistical design of experiments to optimise and characterise single screw extrusion; they have shown that the barrel temperature profiles and screw rotation speed are the most influential variables on melt temperature, extruder pressure, torque and machine output. However, since advanced DoE software packages were not widely available at that time, mathematical regression models and 3D response surfaces were not generated in this study. Vignol et al.[33] proposed simplified models for single screw extrusion operations, in which a set of extrusion simulations was carried out according to a fractional factorial design of experiments. In this study, the DoE analysis was based on the simulated data from the simplified models obtained by ‘Flow 2000’ software simulation without detailed emphasis on single screw extrusion. Wagner *et al.* [34] have reported graphical illustrations of a statistical analysis (DOE approach) carried out on extruding High Impact Polystyrene (HIPS), using widely different compression ratio to investigate the interaction of processing conditions. By recording the drive motor current they showed that a high compression screw may not allow the process to reach steady state conditions over the 10 minute intervals used for the measurements. More recently, Wagner & Cantor[35] used a similar DoE experimental strategy to determine that PP melt temperature in single screw extrusion can be predicted using a second order model, as a function of screw rotation speed and barrel temperature in the feed zone.

In the present study a commercial DoE software package is used to examine laboratory-scale single screw extrusion dynamics and determine the extrusion performance characteristics of a low melt flow index (MFI) polypropylene (PP) and a high MFI recycled PP blends from 0 to 100%. For immiscible polymer blends there can be difficulties arising from phase inversion at an unpredictable composition range, which can also be affected by operating conditions. For the case of miscible blends, Burch and Scott[36] showed that decreasing the viscosity ratio (minor/major component) delays the time for a mixer to reach steady state conditions and, therefore, for the blend to acquire a homogeneous structure. These authors also showed that for blends of the same composition the zero shear viscosity can be fitted to a law of mixtures based on the weight fraction of each component, which can be related to the weight average molecular weight of the individual component above the critical conditions for entanglement[37], i.e.

$$\eta_0 = K[W_{low}Mw_{low} + W_{high}Mw_{high}]^{3.4} \qquad (1)$$

The two polymers in this study are miscible and, therefore, according to equation 1 the resulting viscosity changes monotonically with both composition ratio and operating conditions, such as temperature and shear rate. This is aided by the very low interfacial energy between the two polymers, which prevents forming dispersed droplets and irregular agglomeration during evolution of a homogeneous melt.

The selected independent experimental factors for this work were:

- barrel temperature
- rotational screw speed
- recycled polymer concentration from 0 to 100%.

The dependent response parameters include:

- melt pressure
- temperature rise due to shear heating
- mass output
- screw torque.

A central composite design (CCD) was constructed to develop second order response surface models. The significance of the individual factors and two-factor interactions was determined by the analysis of variance (ANOVA) method, through which the generalised empirical model can be refined by removing insignificant terms. Overall, the investigation aimed to deliver statistical models and corresponding response surfaces from real-time experimental measurements, which would allow determining the manufacturing implications of recycled polypropylene mixtures with large differences in MFI values.

Experimental

Materials

Two polypropylenes (PP):

- Recycled compound (A850) (MFI = 9.5 dg min^{-1}, solid-state density unspecified, melt density = 754 kg m^{-3} at 230 °C) supplied by Regain Polymers Ltd.
- General extrusion homopolymer grade (531P) (MFI = 0.3 (2.16 kg load at 230 °C), density = 905 kg m^{-3} (solid-state) and 750 kg m^{-3} (melt-state) supplied by Sabic (UK) Ltd.

were selected to obtain blends with large viscosity variation across the composition range. Each material's melt flow index was checked according to ISO1133. There were no further rheological measurements since the melt flow characteristics of miscible blends can be predicted with approximate extrapolations from the individual material MFRs. For instance, it is known that the decrease in molecular weight of polypropylene taking place in service and through recycling operations reduces the shear thinning effect on viscosity. The resulting change in rheological behaviour, however, is not expected to have a significant effect on the interpretation of the data related to screw speed, which is the only relevant related process variable.

Experimental design - Selection of factor ranges and responses (single screw extrusion)

Table 1 outlines, in addition to composition ratio, other factors known to affect the optimisation of single screw extrusion operations[12-15] (screw speed; barrel temperature) and were varied as listed.

Table 1. The selected factor ranges and levels

	Factor names	Factor ranges	Low (-)	High (+)
A	Barrel heating temperature (℃)	180 - 240	192.2	227.8
B	Screw speed (rpm)	20 - 120	40.3	99.7
C	Content of Recycled PP (%)	0 - 100	20.3	79.7

The dependent responses to the selected independent factors include:

- Melt pressure, owing to the effect on energy consumption and die design
- Temperature difference between the die and the actual polymer melt temperature at the die exit (measured by a deep-set thermocouple), due to shear heating
- Extruder output as the factor determining the production capability. Given the close similarity in melt-state density of the constituent polymers, mass output was used in the analysis as it would not be affected by the composition ratio.
- Screw torque, as the factor determining the mechanical energy for heat generation and power consumption.

The temperature increment between the three zones along the barrel was set at 10°C, while the metering zone temperature was taken as the value at the section nearest to the die, with the two preceding sections stepped down by 10 °C. For example, a 180°C barrel temperature represents three zone temperatures, respectively 160, 170 and 180°C. The other factors were set around the general operating conditions, as outlined in Table 1.

Constructing the design

Within a typical design of experiments approach, a central composite design (CCD) is widely used to analyse a second order response surface. Generally, 2^k runs, 2k axial runs, and at least one centre point are required by a CCD, for an investigation based upon k factors. Figure 1 shows the central composite design when k is equal to 2. It is clear that each numeric factor is varied over 5 levels: ± alpha (axial points), ± 1 (factorial points) and the centre point (0,0).

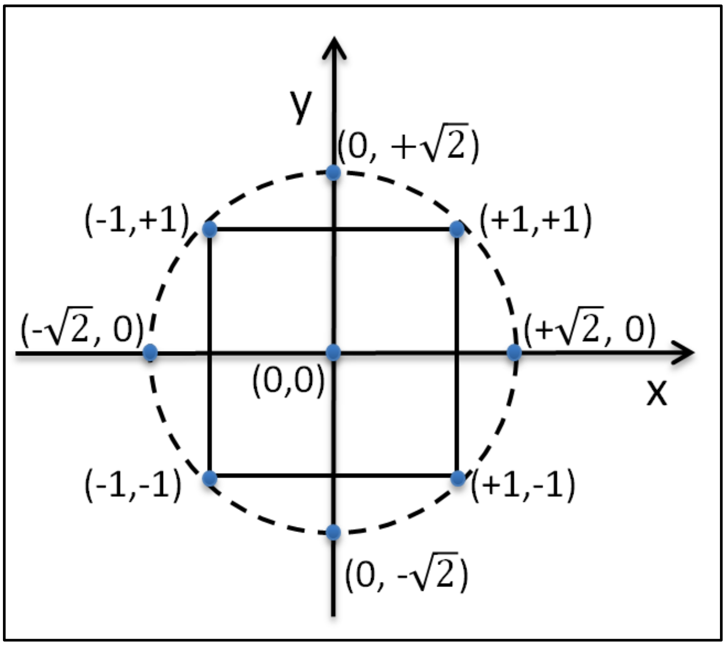

Figure 1. Central composite design for k = 2.

The sparsity of effects principle states that the direct effects and two-factor interactions usually dominate in a system whilst the higher order interactions are negligible. For this study, a CCD in 3 factors was used to fit a second order surface response model. Design Expert® software, version 8.0.7.1 (Stat-Ease, Minneapolis, USA) was used to build the response surface design. As the axial points are more extreme than the factorial points (see Figure 1), the factor ranges were then entered into the software as the 'alpha' (axial) points. Otherwise, the screw speed and the recycled PP concentration would have negative values at axial points if the factor ranges were entered as factorial points.

Table 2 shows the generated design matrix, which contains 8 factorial runs, 6 axial runs and 6 centre runs. A full second order regression model was selected for each response to start with. The mathematical model is shown as follows:

$$y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + \varepsilon \quad (2)$$

Where

Y	=	the response
X_1, X_2, X_3	=	the independent factors or variables
β_1, β_2, β_3, etc.	=	the regression coefficients
ϵ	=	a random error term.

In the equation, linear terms of the form $\beta_i X_i$ are the main / direct effects, two-factor interactions are in the form of $\beta_{ij} X_i X_j$, and quadratic terms of the form $\beta_{ii} x_i^2$ allow for curvature in the effect of a variable on the overall response. The estimate of β_0 is the grand average of all observations and the estimates of the other coefficients β_i are one-half the effect estimate for the corresponding factor (Montgomery[21]). The Design Expert® software uses symbols A, B and C to represent the variables.

Table 2. Design matrix generated by Design Expert® (A = barrel temperature, B = screw speed, C = A850 Recycled PP blend concentration)

Std Order	Run	A (^oC)	B (rpm)	C (%)	Coded A	Coded B	Coded C
1	15	192.2	40.3	20.3	-1	-1	-1
2	9	227.8	40.3	20.3	1	-1	-1
3	18	192.2	99.7	20.3	-1	1	-1
4	14	227.8	99.7	20.3	1	1	-1
5	16	192.2	40.3	79.7	-1	-1	1
6	5	227.8	40.3	79.7	1	-1	1
7	7	192.2	99.7	79.7	-1	1	1
8	4	227.8	99.7	79.7	1	1	1
9	8	180.0	70.0	50.0	-1.68	0	0
10	13	240.0	70.0	50.0	1.68	0	0
11	20	210.0	20.0	50.0	0	-1.68	0
12	6	210.0	120.0	50.0	0	1.68	0
13	2	210.0	70.0	0.0	0	0	-1.68
14	11	210.0	70.0	100.0	0	0	1.68
15	3	210.0	70.0	50.0	0	0	0
16	19	210.0	70.0	50.0	0	0	0
17	17	210.0	70.0	50.0	0	0	0
18	10	210.0	70.0	50.0	0	0	0
19	12	210.0	70.0	50.0	0	0	0
20	1	210.0	70.0	50.0	0	0	0

Analysis of variance (ANOVA)

Analysis of variance (ANOVA) was performed by the software to determine the model significance. F-ratios and p-values were calculated and compared. The calculation methodology cited by Montgomery[21] is briefly introduced here. Assume a single factor with *a* different levels or treatments has *n* observations for each treatment. Total sum of squares (SS_T), treatment sum of squares ($SS_{Treatments}$) and error sum of squares (SS_E) can be calculated as follows:

$$SS_T = \sum_{i=1}^{a}\sum_{j=1}^{n}\left(y_{ij} - \bar{y}\right)^2 = n\sum_{i=1}^{a}(\bar{y}_{i\cdot} - \bar{y})^2 + \sum_{i=1}^{a}\sum_{j=1}^{n}\left(y_{ij} - \bar{y}_{i\cdot}\right)^2 = SS_{Treatments} + SS_E \quad (3)$$

where

y_{ij}	=	j^{th} observation taken under treatment i,
$\bar{y}_{i\cdot}$	=	the average of the observations under the i^{th} treatment
$\bar{y}$	=	the grand average.

The mean square (MS) and F_0 can be calculated by the following equations:

$$MS = SS/D_f \quad (4)$$

$$F_0 = \frac{\frac{SS_{Treatments}}{a-1}}{\frac{SS_E}{[a(n-1)]}} = \frac{MS_{Treatments}}{MS_E} \quad (5)$$

where

D_f	=	number of degrees of freedom that corresponds to the sum of squares in equation (4).

The ratio F_0 has an F distribution with (a-1) and a(n-1) degrees of freedom. If the F_0 value computed is greater than the critical F value related to a certain significance level (α), that is $F_0 > F_{\alpha, a-1, a(n-1)}$, then the corresponding effect is considered as statistically significant. A p-value is an alternative approach, in which the p-value is equal to the probability above (F_0) in the $F_{\alpha, a-1, a(n-1)}$ distribution:

$$p - value = P[F_{a-1,a(n-1)} > F_0] \quad (6)$$

The significance level (α) is usually set as 0.05. With F_0 or p-value, it is possible to evaluate whether the variables are significant or the terms in Equation (2) are necessary. In this study, the full second order regression model for each response was simplified by removing the negligible terms with p-values greater than 0.10. The terms with p-values lower than 0.05 were considered as important and kept in the models. Residual analysis was carried out to confirm the adequacy of the model used.

Equipment and procedure

The extrusion equipment was Haake Rheomex 252p single screw extruder (Thermo Fisher Scientific Inc.) with screw diameter, D = 19.05mm and length L = 25D, designed as a standard single thread screw with compression ratio 4:1. A rectangular-section sheet die (width 25mm and a 1mm slit height) was used. According to the design matrix, PP mixtures were prepared by tumble blending prior to extrusion. The barrel zone temperatures, die temperature, and screw speed were set as described above, consistent with the design matrix. The extruder was continuously flood-fed and when the extrusion conditions were changed, sufficient stabilization time was given before steady-state measurements were made. Experiments were carried out following the randomised run order given by the design matrix. Melt pressure (ΔP) at the end of the metering zone and screw torque (M) data were monitored continuously on the extruder and analysed within the software. Some slight fluctuations were evident, so that the nearest whole numbers were taken as the resulting values. The extruded products (for 1-minute time increments) were collected and weighed to calculate the mass output ($\dot{m}$). An infrared thermometer was used to measure the melt temperature at the die exit and to determine the temperature difference (ΔT) between the polymer melt and the die.

Process optimisation

After the process data were entered into Design Expert® software, predictive models for the responses were determined and the related process optimisation was evaluated automatically after the significance criteria were set. These criteria were simply ‘high output, low pressure’ conditions. 240 bar and 71 bar were the highest and lowest melt pressures obtained in the experiment, while 68.5 and 12.3 g min^{-1} were the highest and lowest mass output reached. To optimise the process towards optimum conditions in this experiment, it was assumed that the melt pressure should not exceed 120 bar, while mass output should not drop below 50 g min^{-1}. For improved temperature control, the maximum temperature difference between the die and the melt was set to 10°C, as indicated earlier. Finally overlay plots were generated based on the above criteria.

Results and discussion

Table 3 summarizes the experimental results and Table 4 shows the ANOVA table for melt pressure, as a typical example of the data obtained. Table 5 shows the p-values of the model terms for the response variables.

Table 3. Melt pressure (ΔP), mass output ($\dot{m}$), screw torque (M) and melt temperature rise at the die temperature (ΔT). (A = barrel temperature, B = screw speed, C = A850 Recycled PP blend concentration)

Std Order	Run	Coded A	Coded B	Coded C	ΔP (bar)	$\dot{m}$ (g min^{-1})	M (Nm)	ΔT (°C)
1	15	-1	-1	-1	208.0	48.5	45.0	12.8
2	9	1	-1	-1	146.0	24.2	25.0	12.2
3	18	-1	1	-1	240.0	56.8	46.0	17.8
4	14	1	1	-1	190.0	57.7	35.0	17.2
5	16	-1	-1	1	90.0	24.0	18.0	3.8
6	5	1	-1	1	71.0	16.2	11.0	3.2
7	7	-1	1	1	123.0	48.1	28.0	8.8
8	4	1	1	1	100.0	63.4	17.0	8.2
9	8	-1.68	0	0	165.0	48.6	44.0	11.0
10	13	1.68	0	0	117.0	44.2	20.0	12.0
11	20	0	-1.68	0	93.0	12.3	18.0	5.0
12	6	0	1.68	0	165.0	68.5	30.0	18.0
13	2	0	0	-1.68	237.0	41.1	65.0	15.0
14	11	0	0	1.68	80.0	41.8	13.0	9.0
15	3	0	0	0	133.0	42.8	25.0	12.0
16	19	0	0	0	137.0	42.6	24.0	10.0
17	17	0	0	0	160.0	39.0	25.0	10.0
18	10	0	0	0	141.0	42.6	24.0	11.0
19	12	0	0	0	138.0	41.9	24.0	12.0
20	1	0	0	0	141.0	41.8	25.0	10.0

Table 4. ANOVA Table for melt pressure. A = barrel temperature, B = screw speed, C = A850 recycled PP blend concentration. D_f represents the number of degrees of freedom - corresponding to the sum of squares in equation (3).

Source	Sum of Squares	Df	Mean Square	F-Value	p-value
Model	42803.808	9	4755.979	69.335	< 0.0001
A	4034.335	1	4034.335	58.815	< 0.0001
B	4915.272	1	4915.272	71.657	< 0.0001
C	32287.895	1	32287.895	470.709	< 0.0001
AB	8.000	1	8.000	0.117	0.7398
AC	612.500	1	612.500	8.929	0.0136
BC	24.500	1	24.500	0.357	0.5634
A^2	2.513	1	2.513	0.037	0.8520
B^2	210.850	1	210.850	3.074	0.1101
C^2	628.667	1	628.667	9.165	0.0127
Residual	685.942	10	68.594		

Table 5. ANOVA table of p-values (A = barrel temperature, B = screw speed, C = A850 recycled PP blend concentration)

Source	Melt pressure	Mass output	Screw Torque	Temperature difference
A	< 0.0001	0.0822	0.0002	0.8946
B	< 0.0001	< 0.0001	0.0150	0.0002
C	< 0.0001	0.0176	< 0.0001	0.0001
AB	0.7398	0.0004	0.6932	1.0000
AC	0.0136	0.0074	0.3158	1.0000
BC	0.5634	0.0095	0.6932	1.0000
A^2	0.8520	0.1030	0.1450	0.9693
B^2	0.1101	0.5070	0.3964	0.9693
C^2	0.0127	0.8001	0.0038	0.7778

Melt pressure (ΔP)

For the recorded melt pressure data, A, B, C, AC and C^2 were considered as significant terms according to their p-values and thus the second order regression model was reduced by removing the remaining terms. With the computed coefficients, the final predictive equation for melt pressure with coded factors becomes:

$$\Delta P = 139.03 - 17.19A + 18.97B - 48.62C + 8.75AC + 6.91C^2 \qquad (7)$$

This equation confirms that the respective effects of the independent variables are consistent with theoretical predictions. For instance, increasing the melt temperature in the extruder metering zone (factor A) and the high MFI concentration component of the PP blend (factor C) each reduces melt viscosity and therefore, reduces pressure across the die. Since melt temperature also controls shear viscosity, the interactive term AC is significant. Furthermore, one notes the large effect of factor C in equation 7. Figure 2 shows a response surface predicted by this equation. The response surface shows that melt pressure is reduced with increased barrel temperature and the recycled PP content. The magnitude of the change in melt pressure, with respect to variables A and C, is consistent with the interpretation above.

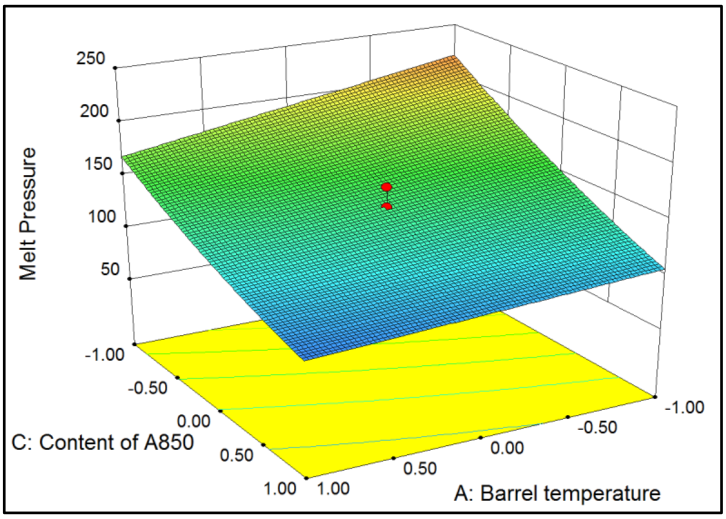

Figure 2. Melt pressure response surface versus PP composition ratio, factor C (A850 Content) and barrel temperature, factor A. (B is set at the neutral point, i.e. zero-level).

As theory predicts, higher screw speed (B) increases volumetric output and induces a higher pressure even though melt viscosity would be expected to decrease by shear-thinning effects. It should be noted, however, that the added high MFI component would reduce this effect. Under any given set of process conditions, melt viscosity of the PP compounds is determined by the recycled PP composition ratio which, in turn, will reduce the sensitivity of melt viscosity to changes in barrel temperature and, consequently, its effect will be borne out on the overall response. As a result, ANOVA showed that the AC interaction factor was significant. However, while interpreting interactive effects can be quite complex the second order term (C^2) is significant and characterizes the response surface curvature.

As mentioned previously the estimates of the coefficients $\hat{\beta}_i$ are one-half the effect predicted for the corresponding factor. Therefore, the first order term of C is the most influential on the response, which implies that melt viscosity dominates the developed pressure, hence energy consumption, in single screw extrusion processes.

Extruder mass output ($\dot{m}$)

For extruder mass output, equation 8 is the reduced regression model with coded factors.

$$\dot{m} = 42.31 - 1.7A + 15.19B - 2.5C + 6.03AB + 3.86AC + 3.69BC \quad (8)$$

Note that although melt transport theory in extrusion processes relates volumetric output (Q) to the process variables, this has no significant effect on the mass output ($\dot{m}$) predictions. Equation 8 directly relates to volume output if desired, due to limited compressibility effects. Furthermore, the modified model implies that over the factor ranges studied, non-linear effects are not identified from the experimental results. The coefficients suggest that screw speed (B) is the most significant effect on mass output, consistent with Borgaonkar et al.[32] and with predictions from the melt pumping analysis in single screw extrusion, consisting of laminar shear flow due to drag forces induced by screw rotation[2-4].

Figure 3 is a typical Output versus Pressure (Q/P) diagram derived from basic theoretical considerations, which illustrates the effects of rotational screw speed ($N_B > N_A$) consistent with the dominating effect of this variable predicted in Equation (8). This figure also implies that at a given screw speed there will be only a relatively small change in the operation viscosity, as reported elsewhere[1]. This also explains the small coefficients for the barrel temperature effect and PP blend ratio (term C in Equation 8), confirming that they have limited influence on mass output, despite their direct effect on melt viscosity. Therefore, output may increase or decrease as viscosity changes depending on the way the respective coefficients affect the extruder and die characteristics.

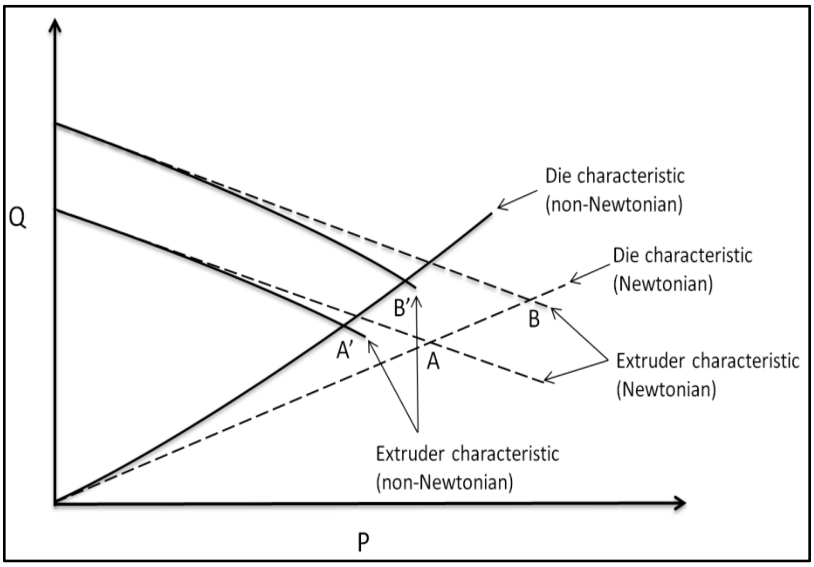

Figure 3. Schematic depiction of the output of a single screw extruder (Q) as a function of head pressure (P), defining respectively the screw and die characteristics, and their intersection as the operating condition. Solid lines represent the solution of the basic equation for pseudoplastic rheological behaviour and dotted lines for Newtonian flow.

Figures 4 and 5 show two ANOVA response surfaces represented by Equation (8). They reveal the complex nature of extrusion dynamics in predicting that all two-factor interactions are significant terms. Since each main factor can have a large effect on melt viscosity it is likely that a change in one factor can influence the effect of another factor on mass output simply through their effect on viscosity. It is clear also that increasing barrel temperature and low viscosity polymer blend component can significantly reduce melt pressure, albeit with a marginal predicted loss in extruder output. Since high barrel temperature increases thermal energy consumption the option of utilising low-viscosity polymers could be a viable alternative.

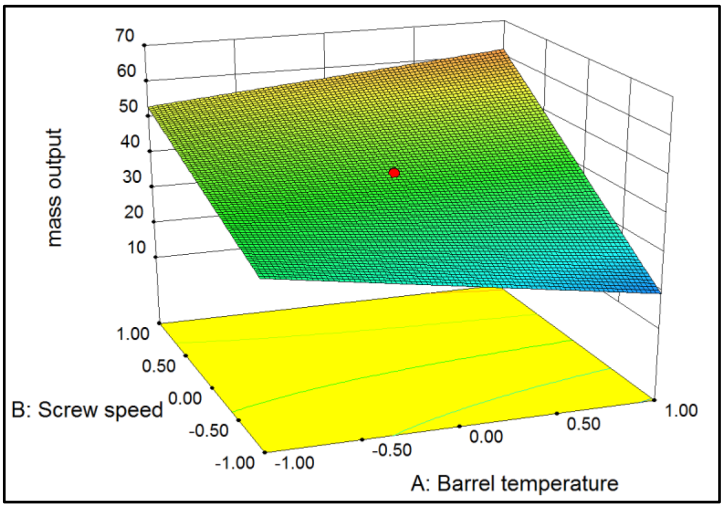

Figure 4. Mass output response surface versus barrel temperature (factor A) and screw speed (factor B), (C is set at the neutral point, i.e. zero-level).

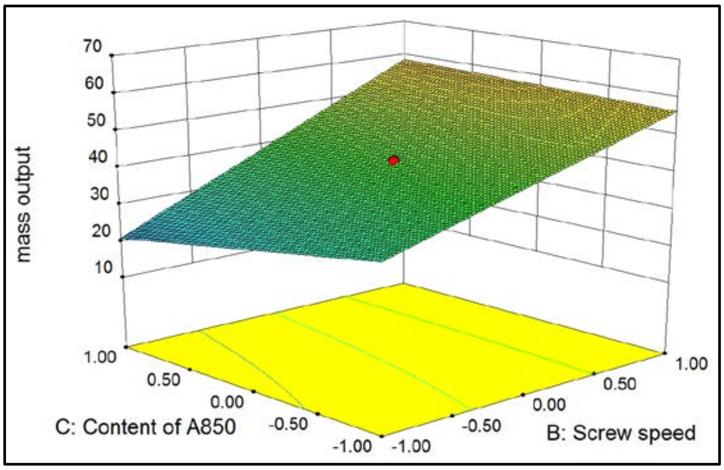

Figure 5. Mass output response surface versus screw speed (factor B) and composition ratio (A850 Content, factor C). (A is set at the neutral point, i.e. zero-level).

Screw torque (M)

The screw rotation torque relates to the shear forces generated along the extruder screw-barrel assembly and is the electrical to mechanical energy transformation that complies with the set process variables. In this study the predicted model obtained for screw torque (M) is:

$$M = 25.22 - 6.54A + 3.45B - 12.04C + 4C^2 \quad (9)$$

This equation predicts similar qualitative relationships to Equation 7 (melt pressure, ΔP) without the AC interaction term. This similarity is due to the melt pressure and screw torque dependence on shear viscosity.

Screw torque mainly relates to the metering zone conditions where the melting process is complete and power requirements are dominated by pumping the melt through the die via screw rotation. Increased low viscosity blend component (factor C) concentration has a similar effect on screw torque and includes a second order term in Equation 9. Increased screw speed also increases screw torque and machine output, despite the lower melt viscosity due to shear heating. Figure 6 shows one response surface predicted from Equation 9. Figure 6 indicates that the lowest screw torque is obtained when the barrel temperature and the low viscosity blend component are at their highest levels, which is due to lower shear viscosity. Since screw torque in single screw processes determines the required mechanical energy and electrical power requirements the analysis confirms that using low viscosity polymers reduces motor power.

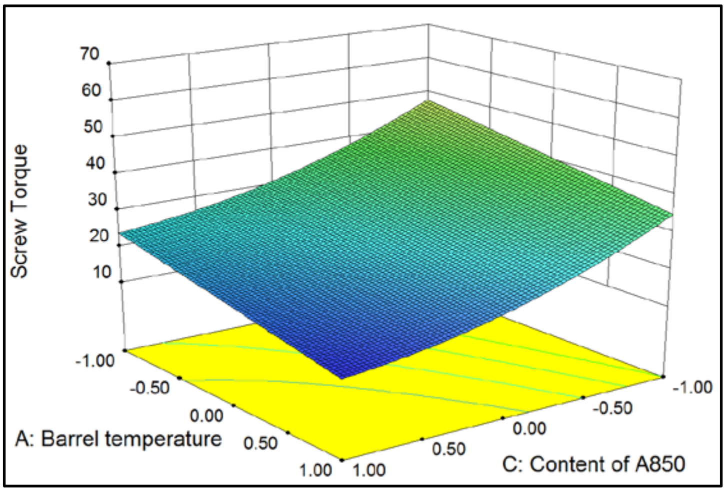

Figure 6. Screw torque response surface versus barrel temperature (factor A) and composition ratio (Content of A850) (factor C). B is set at the neutral point, i.e. zero-level).

Temperature increase at the die (ΔT)

With the aid of a p-value approach, the reduced regression model with coded factors for temperature difference (ΔT) between the barrel temperature (factor A) and the extruded melt, is as follows:

$$\Delta T = 10.95 + 3.07B - 3.37C \quad (10)$$

This response surface model is very simple, consisting only of first order terms for screw speed (B) and recycled PP concentration (C), so that the initial full second order model has been significantly reduced to a simple linear model. This result implies that only factors B and C were significant with a linearly balanced counteracting response on the temperature rise, as shown in Figure 7.

For simple shear flow, viscous heat dissipation per unit volume (φ_S) can be estimated by the following equation.[38]

$$\phi_S = \eta.(\dot{\gamma})^2 \quad (11)$$

Using a simple power law model for the dependence of viscosity (η) on shear rate ($\dot{\gamma}$) for polymer melts, where *k* and *n* are material constants:

$$\eta = k\dot{\gamma}^{n-1} \quad (12)$$

So that substitution gives:

$$\phi_S = k.(\dot{\gamma})^{n+1} \quad (13)$$

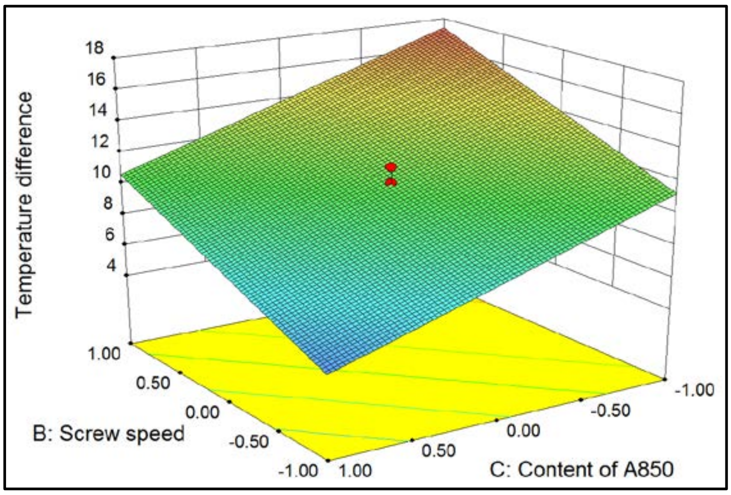

Figure 7. Die temperature rise response surface versus screw speed (factor B) and composition ratio (Content of A850) (factor C). A is the neutral point, i.e. zero-level).

Coefficients *k* and *n* are also temperature-sensitive, whilst shear rate $\dot{\gamma}$ is proportional to screw speed. Since the PP blend composition determines indices *k* and *n*, Equation 13 states that screw speed (B) and recycled PP concentration (C) are the two factors that determine shear heating, regardless of the barrel temperature (factor A), thereby confirming that the simple relationship expressed by Equation (10) is valid. Note that the barrel temperature effect was too small to be identified by ANOVA as it was lost in the noise or experimental error. Since as a first approximation, power law index (*n)* can be considered to be temperature independent, the barrel temperature can only be expected to affect the consistency index *k* and, therefore, is less influential than the recycled PP concentration.

The analysis shows that the temperature increment (ΔT) at the die is also significant, and therefore would have to be taken into account in some cases where either the polymer or the additives may be prone to temperature rises, as it is obviously the case when chemical blowing agents are used to produce cellular sheet products, for example. With the aid of a ANOVA analysis the barrel temperature can be set at a required lower level and allow the melt temperature to rise to the precise value required to initiate the blowing agent decomposition.

Process Optimisation

Figures 8 and 9 show overlay plots generated by the software for process optimisation. The yellow areas in the graphs represent the processing conditions ('operating windows') that meet the defined criteria, which for the system examined are melt pressure 120 bar (maximum), mass output not less than 50 g min^{-1} and temperature rise at the die no higher than 10°C. These Figures show the operating conditions where recycled PP concentration is 70% and 90% respectively. The two axes chosen were screw speed and barrel temperature in order to emphasise the influence of melt viscosity. The plots reveal that when viscosity is reduced by increasing the high MFI recycled component concentration in the blend, the processing operating window becomes wider, thus providing scope for a less stringent control of the composition and variability of the recycled components for industrial operations.

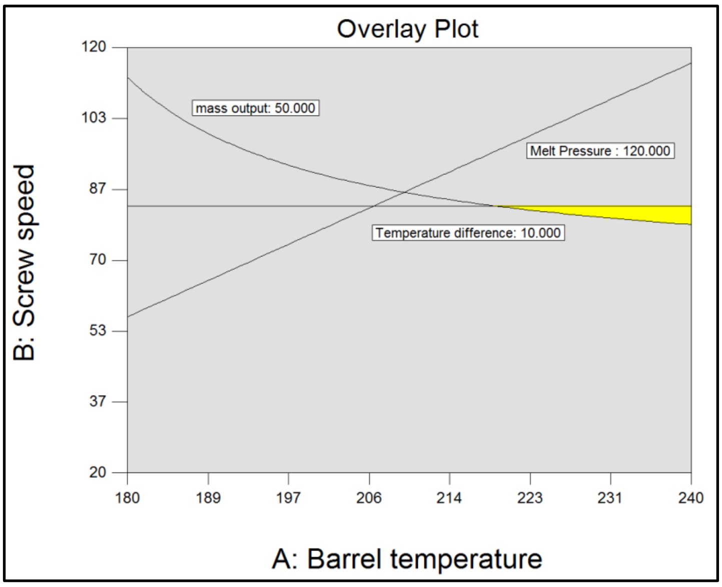

Figure 8. Overlay plot for process optimisation for composition ratio (factor C), A850 recycled PP = 70% (screw speed in RPM; barrel temperature in ^{o}C).

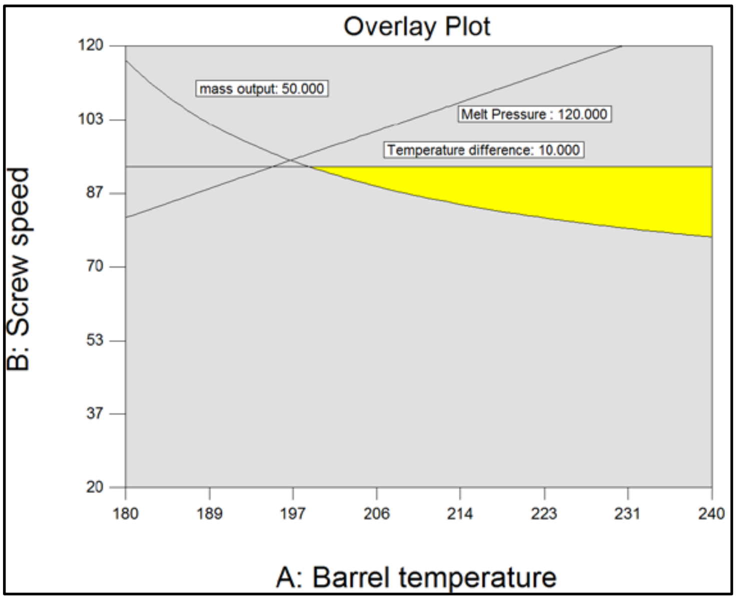

Figure 9. Overlay plot for process optimisation for composition ratio (factor C), A850 recycled PP = 90% (screw speed in RPM; barrel temperature in ^{o}C).

Summary and Conclusions

A DoE approach was used to investigate single screw extrusion dynamics and a central composite design (CCD) in the framework of response surface methodology (RSM) was selected. The variables ('factors') studied were barrel temperature, rotational screw speed and the low viscosity recycled component concentration in a PP blend system. The measured responses for the steady-state process operation included melt pressure, mass output, screw torque and the temperature rise at the die. The level of significance of the response surface models was determined by analysis of variance method (ANOVA). A high level of accuracy and consistency was achieved throughout, so that the resultant empirical models and predicted response surfaces would identify and quantify the effect of the respective variables on the selected responses. Within the experimental ranges of the three chosen factors, the recycled PP concentration and, therefore, the feedstock melt viscosity were found to have the highest effect on melt pressure and screw torque. Screw speed was found to be the predominant factor for the extruder output rate. Overall, the respective effects of the examined variables on mass output were confounded by two-factor interactions, which were found to be statistically significant. The simple linear regression model obtained for die temperature rise implies that screw speed and recycled PP content are the most significant factors, having similar but opposite effects. The study has provided quantitative data for single screw extrusion and has demonstrated the value of an experimental DoE approach for predicting the range of possible processing conditions for manufacturing operations.

Conflicts of interest

None declared.

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