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Use of experimental design to investigate processing conditions and *K* value effects in poly(vinyl chloride) window profile extrusion

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Statistical experimental design has been used on a laboratory scale, twin screw extruder to investigate how processing conditions and polymer *K* value affect both the extrusion process and important extrudate properties: *viz.* Charpy impact strength, colour, gloss, and degree of gelation. It is demonstrated how this approach can be used to define the optimum processing window. Although in production situations it may be impractical or too expensive to vary large numbers of variables over a wide processing range, it is possible to use the experimental design approach without initiating unwieldy experimental programmes by using Evolutionary Operation. This is illustrated with reference to extrusion line trials. PRC/1566

© 1999 IoM Communications Ltd. The authors are with European Vinyls Corporation (UK) Ltd, PO Box 8, The Heath, Runcorn, Cheshire WA7 4QD, UK. This paper was presented at PVC 99 – From strength to strength, held in Brighton, UK on 20–22 April 1999.

INTRODUCTION

The current trend in the poly(vinyl chloride) (PVC) window sector is towards ever increasing rates of output. In the 1980s twin screw extruders were typically run with line speeds of 1.0–2.0 m min⁻¹. In the early 1990s line speeds had risen to 2.0–4.0 m min⁻¹, corresponding to output rates of 150–350 kg h⁻¹. Recent developments make it possible to achieve line speeds^{1,2} of 5.0 m min⁻¹. With such increases in output, it is essential to understand the influence of processing conditions and polymer characteristics on the properties of the profile produced.³

The properties of extruded PVC are governed by the heat and shear history imposed on the material during processing. The primary particles of PVC break down to form a homogeneous matrix – the so-called gelation or fusion process.⁴ The level of gelation is a critical factor in determining mechanical properties. It seems that tensile strength properties increase with increasing gelation level until reaching a plateau, whereas impact properties reach a maximum and then decline as gelation level is further increased.⁵

Some earlier studies on the effect of processing conditions on profile properties have also used statistical experimental design.^{6,7} This is an alternative approach to the classical experimental method of changing one variable at a time. This approach allows many variables to be changed simultaneously, reduces the amount of experimentation required, provides information about interactions between variables, and enables prediction of an optimum set of operating conditions.

In this paper it is shown how statistical experimental design can be used to examine the way processing variables and polymer *K* value affect the properties of extruded PVC. The first set of experiments were carried out on a laboratory scale extruder over a wide range of processing conditions to produce a wide range of properties. From the results it is possible to predict the optimum processing window.

In the second set of experiments it is shown how the principles of experimental design can be used for process improvement during a normal production run. This is a process known as Evolutionary Operation or EVOP.⁸

LABORATORY SCALE EXTRUSION TRIALS

Experimental method

The processing variables chosen for the laboratory scale extrusion trials were as follows: screw speed in the range 15–55 rev min⁻¹; barrel zone 1 temperature in the range 165–195°C; barrel zone 2 temperature in the range 165–195°C; and screw temperature in the range 140–160°C. It was also decided to investigate the effects of polymer *K* value and apparent density; the latter via addition of a silica antistatic agent.⁹ The *K* value was varied between 66 and 68, and the level of antistatic agent between 0 and 0.3 parts per hundred parts of polymer (phr).

The experimental design used is given in Table 1. This was generated using a commercially available software package, known as ECHIP (ECHIP inc., Hockessin, DE, USA). Some midpoint values were included for the processing variables, whereas the formulation variables were only included at two levels.

The formulation used for these trials was a standard Pb stabilised window recipe, containing 7.5 phr of acrylic impact modifier. It was necessary to prepare four different blends to run the trials. The apparent density and time to gelation (on a Brabender torque rheometer at constant volume) of the four blends are shown in Table 2. Although the antistatic agent was added ostensibly to increase the apparent density of the blends, it was found to have no effect on the blend made with *K*-66 polymer. The polymer *K* value did, however, have a significant effect on the time to gelation. As expected, *K*-66 blends gelled faster than those based on *K*-68.

The extrusion trials were run on a Krauss-Maffei KMD 2-25 KKL twin screw extruder. The adapter

Table 1 Experimental design for laboratory scale extrusion trials

Trial no.	Screw speed, rev min ⁻¹	Zone 1 temp., °C	Zone 2 temp., °C	Screw temp., °C	K value	Antistatic agent, phr
1	15	165	165	140	68	0.3
2	55	165	165	140	68	0
3	55	165	165	140	66	0.3
4	15	195	165	140	68	0
5	15	195	165	140	66	0.3
6	55	195	165	140	66	0
7	55	195	165	140	68	0.3
8	55	165	195	140	68	0.3
9	15	195	195	140	68	0.3
10	55	195	195	140	68	0
11	55	195	195	140	66	0.3
12	15	165	165	160	66	0
13	55	165	165	160	68	0.3
14	55	195	165	160	66	0.3
15	15	165	195	160	66	0.3
16	55	165	195	160	66	0
17	55	195	195	160	68	0.3
18	15	165	195	160	68	0
19	15	195	195	160	66	0
20	15	165	195	140	66	0
21	15	195	165	160	68	0.3
22	55	195	165	160	68	0
23	35	165	165	140	66	0.3
24	15	180	180	140	68	0
25	35	180	180	140	66	0
26	15	180	165	150	66	0.3
27	35	180	165	150	66	0
1	15	165	165	140	68	0.3
2	55	165	165	140	68	0
3	55	165	165	140	66	0.3
4	15	195	165	140	68	0
5	15	195	165	140	66	0.3

temperature was kept constant at 190°C and the die temperature at 200°C. The material was flood fed. The dosing screw speed and haul-off speed were adjusted as required when changes were made to the screw speed. The extrudate produced was a strip with a width of 30 mm and a thickness of 3 mm.

Results

The torque, melt pressure, and output generated during each trial were noted. In addition, the following properties of the extruded strips were measured: colour (b^*), gloss, Charpy impact strength, and degree of gelation by a capillary rheometer method. All these results are given in Table 3.

All the results were fed into the ECHIP programme and regression analysis was carried out to assess the effects of the input variables on each of the response variables. A summary of the regression analysis for each response is given in its 'effects table', as shown in Tables 4–9. The magnitude and sign of the effect of each variable (and the interactions between vari-

ables) on the response is given in the left hand column. The constant in each table is the average value of the response. The 'effects' are changes in the response as the input variable moves from its lowest level to its highest level. The asterisks indicate the statistical significance (sig.) of the corresponding effect – the greater the number of stars the more significant the effect. The results for each response variable are discussed separately below.

Torque

The effects table for the extruder torque is given in Table 4. It is seen that screw speed has an enormous effect on torque; increasing the screw speed from 15 to 55 rev min⁻¹ gives an increase in torque of 31.7%. This is presumably because at higher screw speed there is increased shear, which promotes gelation and hence increases torque.

Melt pressure

It can be seen from Table 5 that melt pressure decreases with increasing temperature of barrel zone 1, barrel zone 2, and screw oil, but increases with increasing screw speed, K value, and antistatic agent. However, the effect of screw speed is complex because there is a squared term in the model: melt pressure is found to increase with increasing screw speed up to ~40 rev min⁻¹ and thereafter starts to decrease as screw speed is further increased.

Colour (b^*)

Colour was measured using a Dr Lange microcolorimeter. The light source in this technique is a xenon lamp. Reflected light is split into three components

Table 2 Properties of blends used in laboratory scale extrusion trials

Blend no.	K value	Antistatic agent, phr	Apparent density (± 2), g L ⁻¹	Brabender time to gelation (± 0.1), min
A	68	0.3	644	2.67
B	68	0	648	2.73
C	66	0.3	655	2.28
D	66	0	644	2.36

Table 3 Results of laboratory scale extrusion trials

Trial no.	Torque, %	Melt pressure, bar	Output, m min ⁻¹	Colour b*	Gloss, %	Charpy impact strength, kJ m ⁻²	No. of ductile failures	Capillary rheometer result, MPa	Gelation, %
1	33	210	1.02	3.1	8.40	7.8	0	12.0	34
2	65	259	2.08	2.7	7.85	67.9	10	16.5	72
3	68	260	2.17	2.9	8.20	71.1	10	16.7	73
4	35	206	0.60	3.7	22.43	15.6	7	15.2	60
5	35	209	0.58	3.8	23.63	65.3	10	15.3	61
6	66	216	2.25	3.2	10.38	11.6	0	18.2	86
7	70	239	2.24	3.2	7.13	13.7	1	19.2	94
8	69	223	2.18	3.3	7.82	13.2	0	19.2	94
9	35	202	0.53	4.2	25.44	67.1	10	17.5	79
10	66	206	2.17	3.6	9.30	11.4	0	19.4	96
11	68	204	2.23	3.3	7.58	12.0	0	18.3	86
12	35	197	0.64	3.1	15.80	6.3	0	12.7	39
13	70	256	2.24	3.0	6.60	61.8	10	19.1	93
14	68	213	2.31	3.5	7.05	11.8	0	18.2	86
15	36	193	0.60	3.5	26.02	66.7	10	15.2	60
16	65	188	2.34	3.3	5.25	9.8	0	18.1	85
17	65	207	2.27	3.8	6.93	12.1	0	19.9	100
18	34	187	0.58	3.5	22.35	66.6	10	15.6	64
19	38	165	0.60	4.2	28.93	14.9	0	16.9	75
20	36	187	0.64	3.3	28.82	16.2	0	15.0	58
21	37	212	1.02	3.9	21.68	66.7	10	16.5	71
22	67	213	2.16	3.4	5.95	12.4	0	19.0	92
23	50	252	1.44	2.7	9.48	9.1	0	14.1	51
24	34	195	0.57	3.5	18.97	12.0	0	15.3	61
25	52	231	1.47	3.0	16.93	67.0	10	16.7	73
26	35	207	0.60	3.4	17.93	16.1	5	14.5	54
27	50	242	1.47	2.9	14.27	68.8	10	15.8	65
1	31	206	0.62	3.1	9.52	6.7	0	12.5	37
2	61	268	2.27	2.8	9.13	67.3	10	15.6	64
3	66	251	2.22	2.8	13.00	67.4	10	17.7	81
4	33	203	0.57	3.8	26.67	16.4	7	15.8	65
5	34	205	0.58	3.5	32.35	18.4	8	16.1	68

via an optical waveguide and routed to three standard measurement filters. In this case the colour measurement of interest was the b^* value, which describes the yellow to blue scale (+ to -). As seen from Table 6, a significant increase in b^* (yellowing) is brought

about by increasing the temperatures of zone 1, zone 2, and the screw oil, and to a lesser extent by switching from $K-66$ to $K-68$ polymer. The effect of increasing screw speed is to give a significant decrease in b^* .

Table 4 Effects table for torque (%)*

Effects	Sig.	Term
50.97		Constant
31.72	***	Screw speed
0.90		Zone 1 temp.
0.24		Zone 2 temp.
0.67		Screw temp.
-0.79		K value
1.02		Antistatic agent
1.50	*	Antistatic × screw speed
-1.38	*	Zone 2 × screw temp.

* See text for explanation of effects tables.

Table 5 Effects table for melt pressure (bar)

Effects	Sig.	Term
235.34		Constant
26	***	Screw speed
-12.63	***	Zone 1 temp.
-22.74	***	Zone 2 temp.
-9.43	**	Screw temp.
9.83	**	K value
9.54	**	Antistatic agent
-10.53	**	Screw speed × zone 1 temp.
-9.91	**	Screw speed × zone 2 temp.
-26.95	***	(Screw speed) ²

Table 6 Effects table for colour (b^*)

Effects	Sig.	Term
3.422		Constant
-0.353	***	Screw speed
0.556	***	Zone 1 temp.
0.366	***	Zone 2 temp.
0.206	***	Screw temp.
0.126	**	K value
0.082		Antistatic agent
-0.171	**	Screw speed × Zone 1 temp.

Table 7 Effects table for gloss (%)

Effects	Sig.	Term
15.04		Constant
-14.48	***	Screw speed
4.08	**	Zone 1 temp.
3.81	**	Zone 2 temp.
-1.92		Screw temp.
-2.72	*	K value
-0.75		Antistatic agent
-4.78	***	Screw speed × Zone 1 temp.
-4.26	**	Screw speed × Zone 2 temp.
-2.44	*	Zone 1 temp. × Zone 2 temp.
2.32	*	K value × Screw speed

Gloss

Gloss measurements were made with a microgloss reflectometer. Light was directed onto the samples at an angle of 60° and the reflected light measured. Reflectometer values lie in the range 0–100, where 100 corresponds to a black glass standard. Hence, the higher the value, the more reflective the surface. Table 7 shows that gloss is dominated by screw speed. Increasing the screw speed causes a big reduction in gloss. Changing polymer *K* value from 66 to 68 also gives a reduction in gloss, albeit small compared with the effect of screw speed. Gloss can be improved by increasing the temperatures of barrel zones 1 and 2.

Gelation

Gelation measurements were carried out using a Rosand capillary rheometer (CR). This method is based on the assumption that the level of gelation of PVC compound is proportional to the pressure required to extrude it through a very short die.¹⁰ Samples from the ECHIP trial were granulated and extruded on the CR through a 'zero length' die at 145°C and at the following shear rates: 10, 30, 50, 100, 200, and 500 s⁻¹. Melt pressure readings at 100 s⁻¹ were used to determine gelation levels and these readings are given in Table 3.

The level of gelation was calculated according to the relationship¹¹

$$\% \text{ Gelation} = \frac{P - P_{\min}}{P_{\max} - P_{\min}} \times 100 \quad \dots \quad (1)$$

The value of P_{\max} was taken as the maximum value of pressure P recorded, which is assumed to correspond to 100% gelation. The value of P_{\min} was obtained by milling samples at 165°C for various times and measuring melt pressure in the CR. The value extrapolated to zero time was taken as the pressure of the ungelled material. This value is 8 MPa.

Table 8 Effects table for gelation (%)

Effects	Sig.	Term
74.16		Constant
26.79	***	Screw speed
15.55	***	Zone 1 temp.
13.25	***	Zone 2 temp.
4.57	*	Screw temp.
3.73	*	<i>K</i> value
3.69		Antistatic agent
-7.58	***	Screw speed × Zone 1 temp.
-4.91	*	Screw speed × Zone 2 temp.
-5.52	*	Zone 1 temp. × Zone 2 temp.

Table 9 Effects table for output (m min⁻¹)

Effects	Sig.	Term
1.447		Constant
1.590	***	Screw speed
-0.007		Zone 1 temp.
-0.036		Zone 2 temp.
0.083		Screw temp.
0.008		<i>K</i> value
0.028		Antistatic agent
-0.075		<i>K</i> value × Screw speed
0.076		Screw speed × Zone 2 temp.
0.067		Zone 1 temp. × Screw temp.

Values of percentage gelation calculated from equation (1) are given in Table 3. Regression analysis was carried out on these data using the ECHIP software and the effects table is shown in Table 8. Not surprisingly, it was found that increasing the extruder screw speed and barrel zone temperatures has a dominant effect on increasing the level of gelation. Increasing the screw temperature is also influential, but to a lesser extent. It is also seen that increasing the polymer *K* value apparently has the effect of increasing the level of gelation. This is at first surprising because it was found that the blends based on *K*-66 gelled faster than those based on *K*-68. However, increasing the polymer *K* value increases the melt elasticity and this is reflected in an increase in the pressure measurements.¹² Hence the effect of *K* value is on melt elasticity and not on degree of gelation *per se*.

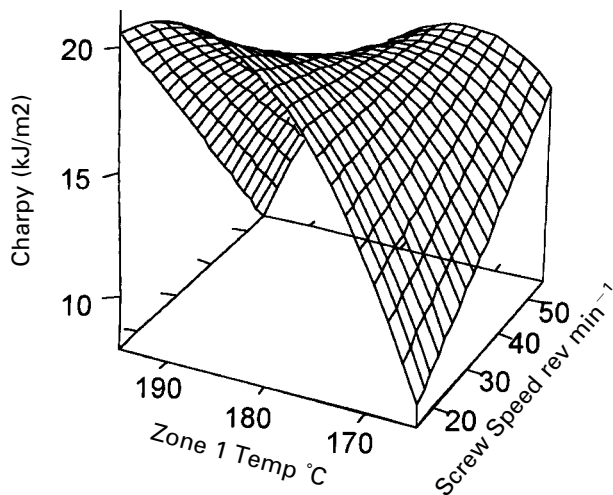
Charpy impact strength

Single notch Charpy tests were carried out according to BS 2782 Part 3 Method 359, as specified in BS 7413. For each experimental run, 10 Charpy measurements were carried out. The values given in Table 3 are an average of the 10 data points, except in cases where the failure mode was a mixture of ductile and brittle behaviour, in which case only the brittle values are considered. The number of ductile failures is also given in Table 3, showing that mixed failure modes were found in the following runs: 4, 4 (repeat), 5 (repeat), 7, and 26.

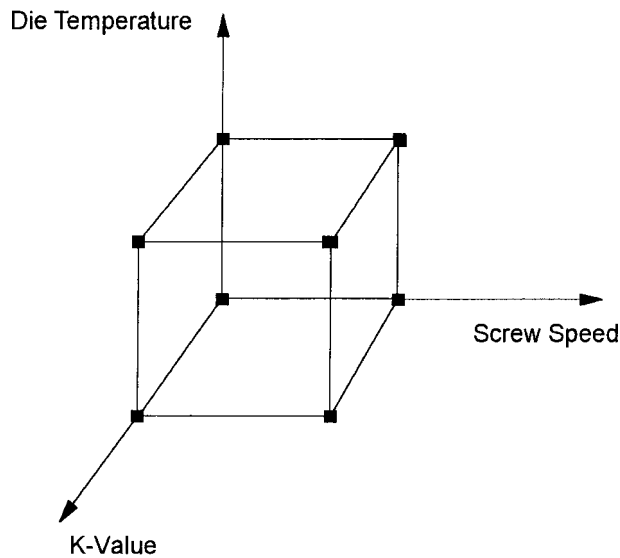
The occurrence of a mixture of ductile and brittle failure modes in the single notch Charpy data introduced a difficulty as far as the regression analysis was concerned: there is essentially a discontinuity in the data, which meant that it could not be processed using the ECHIP software. To remove the discontinuity and hence facilitate analysis, all the data points with 10 ductile failures were assigned values in the range $20 \pm 1 \text{ kJ m}^{-2}$. This value was obtained by linear extrapolation of the data with mixed failure modes. It was then possible to carry out regression analysis and model the Charpy behaviour.

The model gave reasonable agreement with the data. Poor Charpy results were obtained when the processing conditions were set at low screw speed with low barrel zone temperatures or at high screw speed with high temperatures. However, at low screw speed with high barrel zone temperatures and at high screw speed with low barrel zone temperatures good results were obtained. This is illustrated in the three-dimensional contour plot shown in Fig. 1. In terms of the model, there is a strong negative interaction between screw speed and both barrel zone temperatures. The effect of changing polymer *K* value from 66 to 68 was negligible.

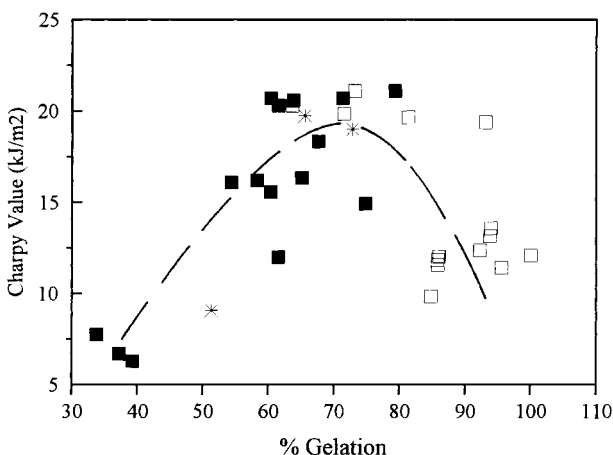
Charpy impact results are plotted as a function of gelation level in Fig. 2. All the data points with 10 ductile failures have been assigned values in the range $20 \pm 1 \text{ kJ m}^{-2}$, as discussed above. There is quite a lot of scatter in the data, but it is seen that the impact strength reaches a maximum at gelation levels of ~70% and then drops off. This effect is in agreement with published work.⁴⁻⁶ It is interesting to note from Fig. 2 that all the samples with high gelation levels and poor Charpy results were processed at high screw



1 Contour plot showing effect of screw speed and rear barrel zone temperature on Charpy impact strength



3 Illustration of design used in EVOP trials



2 Charpy impact strength as function of gelation level: (■) 15, (*) 35, and (□) 55 rev min⁻¹

speed, i.e. at high screw speed and temperature the material has been overprocessed.

Optimum processing conditions

The effects tables discussed above are a convenient way of expressing the results of the multiple regression analysis. For each response, multiple regression analysis generates the best fit polynomial equation to model the relationship between the response and the input variables

$$Y = a_0 + a_1x_1 + a_2x_2 + a_3x_3 + \dots + a_{12}x_1x_2 + a_{13}x_1x_3 + \dots + a_{11}x_1^2 + \dots \dots \dots (2)$$

In order to predict an optimum set of processing conditions, it is necessary to solve the equations for several responses simultaneously. This can be done using the ECHIP software.

Optimising the processing conditions will usually involve achieving the maximum output. The effects table for the output is given in Table 9 and, not unexpectedly, shows that screw speed is the only variable to have a statistically significant effect on output.

For the optimum processing conditions, it was assumed that it was required to maximise the extruder output, maximise the Charpy impact strength, maximise the gloss, and minimise the value of *b**. For this combination, it was calculated that the optimum processing conditions are as follows: screw speed = 55 rev min⁻¹, zone 1 temperature = zone 2 temperature = 165°C, screw temperature = 140°C, K value = 66, and antistatic agent = 0 phr. Hence the optimum processing window is at high screw speed and low temperature. These conditions give a predicted output of 2.23 m min⁻¹, a Charpy impact strength of 21 kJ m⁻² (i.e. ductile failures), a *b** value of 2.62, and a gloss of 10.6%.

If it were essential to have a higher gloss value but not necessary to maximise output, then maximising gloss and Charpy impact strength, and minimising *b** gives the following set of conditions: screw speed = 35 rev min⁻¹, zone 1 temperature = zone 2 temperature = 180°C, screw temperature = 140°C, K value = 66 and antistatic agent = 0 phr. The optimum processing conditions are now at intermediate screw speed and barrel zone temperatures. These conditions give a predicted output of 1.40 m min⁻¹, a Charpy impact strength of 20 kJ m⁻² (i.e. ductile failures) a *b** value of 3.17, and a gloss of 19.3%.

EVOP TRIALS

Experimental method

Evolutionary operation (EVOP) is a simple method of working systematically to understand the statistical significance of the effects, but which can be applied during a normal production run.⁸ Small changes are made in two or three variables and the effects on the production process are measured. The changes should be small enough not to result in product that is out of specification. It may be necessary to repeat the changes several times through an iterative cycle to improve the signal to noise ratio and hence obtain statistically significant results.

The principles of EVOP are illustrated in Fig. 3 with reference to trials carried out on a Krauss-Maffei

Table 10 EVOP trial – processing conditions and results

Trial no.	<i>K</i> value	Die temp., °C	Screw speed, rev min ⁻¹	Melt pressure, bar	Melt temp., °C	Torque, %	Output, kg h ⁻¹	Gloss, %	Colour <i>b</i> *	Charpy impact strength, kJ m ⁻²
1	68	195	18	390	191	71	85.8	38.0	4.5	14.27
2	68	195	24	401	197	74	114	36.2	4.5	15.05
3	68	200	24	398	195.5	72	117	37.5	4.3	13.99
4	68	200	18	383	191	71	85.8	45.8	4.7	15.85
1	68	195	18	386	189	70	84.7	37.8	4.6	15.13
2	68	195	24	402	198	73	114	32.4	4.5	15.61
5	66	195	18	383	190	70	85.7	38.7	4.4	13.93
6	66	195	24	388	194	67	112.8	37.6	4.1	14.09
7	66	200	24	389	194	68	115.4	41.6	4.1	14.61
8	66	200	18	380	189	69	84.5	42.4	4.3	12.60

50KK extrusion line. During these trials it was decided to make changes in the screw speed and die temperature. Polymer *K* value was used as an additional variable and this generated a 2³ factorial design, as illustrated in Fig. 3. This gave rise to eight different trials and two repeats were also included (Table 10).

Screw speed was varied between 18 and 24 rev min⁻¹, die temperature between 195 and 200°C, and *K* value between 66 and 68. The other processing temperatures were kept constant: barrel zones 1–4 were set at 170, 170, 160, and 165°C, respectively; screw temperature was set at 115°C; and the adapter temperature at 175°C. The material was flood fed. The dosing screw speed and haul-off speed were adjusted as required when changes were made to the screw speed. The extrudate produced was a window profile section.

Results

For each experimental run the melt pressure, melt temperature, torque, and output were recorded. These results are shown in Table 10. In addition the colour (*b**), gloss, and Charpy impact strength were measured on each profile (Table 10).

As for the previous trials, it was possible to use the ECHIP software to carry out multiple regression analysis to estimate the effects of the input variables (and their interactions) on the response variables. The effects tables are shown in Tables 11–16.

Table 11 Effects for melt pressure (Bar)

Effects	Sig.	Term
388.93		Constant
7.86	**	<i>K</i> value
-2.86		Die temp.
10.50	***	Screw speed
3.50	*	<i>K</i> value × Screw speed

Table 12 Effects for melt temperature (°C)

Effects	Sig.	Term
192.63		Constant
1.75		<i>K</i> value
-0.50		Die temp.
5.50	***	Screw speed
1.00		<i>K</i> value × Screw speed

Machine parameters

Increasing screw speed by 6 rev min⁻¹ was found to increase melt pressure by 10.5 bar and melt temperature by 5.5°C, but to have no effect on torque. In fact melt pressure and melt temperature were found to be the two factors limiting the speed of operation of the extruder. The die temperature did not have a statistically significant effect on these parameters.

However, *K* value did affect the machine parameters. Increasing *K* value from 66 to 68 gave an average increase in melt pressure of 8 bar and an average increase in torque of 3%. In addition there is a significant interaction between *K* value and screw

Table 13 Effects for torque (%)

Effects	Sig.	Term
70.14		Constant
3.29	**	<i>K</i> value
-0.29		Die temp.
0.17		Screw speed
2.17	*	<i>K</i> value × Screw speed

Table 14 Effects for colour (*b**)

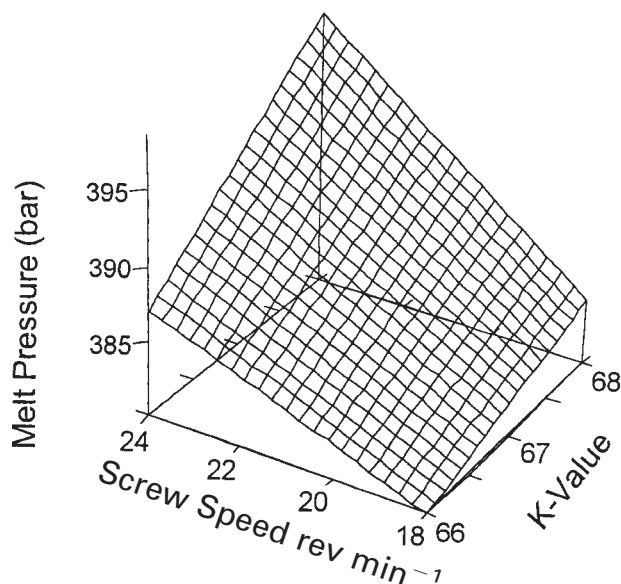
Effects	Sig.	Term
4.368		Constant
0.286	***	<i>K</i> value
-0.036		Die temp.
-0.208	*	Screw speed
0.042		<i>K</i> value × Screw speed

Table 15 Effects for gloss (%)

Effects	Sig.	Term
39.41		Constant
-1.32		<i>K</i> value
4.82	**	Die temp.
-3.06	*	Screw speed
-2.11		<i>K</i> value × Screw speed

Table 16 Effects for Charpy impact strength (kJ m⁻²)

Effects	Sig.	Term
14.38		Constant
1.14		<i>K</i> value
-0.23		Die temp.
0.31		Screw speed



4 Plot of melt pressure as function of screw speed and K value

speed, which means that increases in melt pressure and torque at higher K value are exacerbated at higher screw speed. This is particularly important for melt pressure because the maximum recommended is 400 bar, which has already been reached in trial 2. The relationship between screw speed, K value, and melt pressure is shown in the three-dimensional contour plot of Fig. 4.

Profile properties

It is seen from Table 14 that increasing screw speed by 6 rev min⁻¹ gave a reduction in b^* (yellowing), whereas the effect of changing polymer K value from 66 to 68 was to increase b^* . These results are in agreement with those found in the laboratory scale trials. Gloss was significantly increased by a 5°C increase in die temperature, whereas increasing screw speed by 6 rev min⁻¹ gave a reduction in gloss (Table 15).

The Charpy results obtained in these trials were all brittle failures – although all passed the standard specified in BS 7413. None of the variables (screw speed, die temperature, or K value) was found to have a statistically significant effect on the Charpy results (Table 16).

Process improvement

As described above, from the regression analysis it is possible to predict a set of optimised processing conditions. In this case it was required to maximise output, gloss, and Charpy impact strength, while minimising melt pressure, melt temperature, and b^* . For this combination of properties it was calculated that the optimum conditions are as follows: screw speed = 24 rev min⁻¹, die zone temperature = 200°C, K value = 66. These conditions can be used as a starting point for further EVOP trials.

CONCLUSIONS

Laboratory scale trials

Statistically designed experiments were carried out to investigate the effects of processing conditions and

polymer K value on the extrusion process and the properties of the extrudate. Over the wide processing window studied, it was found that the processing conditions had a much greater effect than the polymer type. Potential benefits of a K-66 over a K-68 polymer were faster gelation, improved gloss, and reduced b^* , without any statistically significant difference in impact strength.

Good Charpy results were obtained at low screw speed and high barrel zone temperatures, and at high screw speed and low barrel zone temperatures. However, at low screw speed and low temperature the material was poorly gelled, whereas at high screw speed and high temperature the material was over-gelled. It was found that Charpy impact strength reached a maximum at gelation levels of about 70% and then dropped away.

Optimum processing conditions were calculated to maximise extruder output, Charpy impact strength, and gloss, and to minimise the value of b^* . The optimum conditions were high screw speed with low barrel zone and screw temperatures, and with polymer of K value 66.

Evolutionary operation trials

Evolutionary operation (EVOP) is a simple process in which small changes are made in a production process. These changes should be small enough not to result in product that is out of specification. Changes are produced in a structured way using a simple factorial design and repeated until the results are statistically significant. It is then possible to shift the production settings in the direction of the improvement and start a new series of runs.

In the EVOP trials carried out on the Krauss-Maffei 50KK extrusion line, screw speed was varied by 6 rev min⁻¹, die temperature by 5°C, and K value by 2 units. In order to run at the higher screw speed, there were benefits in increasing the die temperature to offset the reduction in gloss. There were also benefits in reducing polymer K value from 68 to 66: the lower K value resulted in lower melt pressure (particularly at the higher screw speed) and a reduction in b^* .

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