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Citation: THOMAS, N.L. and HARVEY, R.J., 1999. Use of experimental design to investigate processing conditions and K value effects in poly(vinyl chloride) window profile extrusion. Plastics, Rubber and Composites, 28 (4), pp. 157-164

Additional Information:

• This article was published in the journal, Plastics, Rubber and Composites [© Maney Publishing] and is available at: http://maney.co.uk/index.php/journals/prc/

Metadata Record: https://dspace.lboro.ac.uk/2134/4511

Version: Published

Publisher: © Maney Publishing

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

N. L. Thomas and R. J. Harvey

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 Vinlys 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 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

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Results

The 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.

Table 2 Properties of blends used in laboratory scale extrusion trials

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<th>Blend no.</th>
<th>K value</th>
<th>Antistatic agent, phr</th>
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Figure 1. Schematic diagram of the extruder system. The PVC compound is fed into the hopper and then conveyed into the extruder through the feeder. The extruder consists of a screw in a barrel with cooling zones and a die head. The die head and the cooling zones are connected by optical waveguides to allow measurement of the thermal properties along the extruder. The extrudate is cooled and then cut into pellets for further processing.

Table 3 Results of laboratory scale extrusion trials

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<th>Trial no.</th>
<th>Torque, %</th>
<th>Melt pressure, bar</th>
<th>Output, m min⁻¹</th>
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<th>Gloss, %</th>
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<td>0·67</td>
<td></td>
<td>Screw temp.</td>
</tr>
<tr>
<td>−0·79</td>
<td></td>
<td>K value</td>
</tr>
<tr>
<td>1·02</td>
<td></td>
<td>Antistatic agent</td>
</tr>
<tr>
<td>1·50</td>
<td>*</td>
<td>Antistatic × screw speed</td>
</tr>
<tr>
<td>−1·36</td>
<td>*</td>
<td>Zone 2 × screw temp.</td>
</tr>
</tbody>
</table>

* See text for explanation of effects tables.

Table 5 Effects table for melt pressure (bar)

<table>
<thead>
<tr>
<th>Effects</th>
<th>Sig.</th>
<th>Term</th>
</tr>
</thead>
<tbody>
<tr>
<td>235·34</td>
<td></td>
<td></td>
</tr>
<tr>
<td>26</td>
<td>***</td>
<td>Screw speed</td>
</tr>
<tr>
<td>−12·63</td>
<td>***</td>
<td>Zone 1 temp.</td>
</tr>
<tr>
<td>−22·74</td>
<td>***</td>
<td>Zone 2 temp.</td>
</tr>
<tr>
<td>−9·43</td>
<td>**</td>
<td>Screw temp.</td>
</tr>
<tr>
<td>9·83</td>
<td>**</td>
<td>K value</td>
</tr>
<tr>
<td>9·54</td>
<td></td>
<td>Antistatic agent</td>
</tr>
<tr>
<td>−10·53</td>
<td>**</td>
<td>Screw speed × zone 1 temp.</td>
</tr>
<tr>
<td>−9·91</td>
<td>**</td>
<td>Screw speed × zone 2 temp.</td>
</tr>
<tr>
<td>−26·95</td>
<td>***</td>
<td>Screw speed</td>
</tr>
</tbody>
</table>

Table 6 Effects table for colour (\( \text{b}^* \))

<table>
<thead>
<tr>
<th>Effects</th>
<th>Sig.</th>
<th>Term</th>
</tr>
</thead>
<tbody>
<tr>
<td>3·422</td>
<td></td>
<td>Constant</td>
</tr>
<tr>
<td>−0·353</td>
<td>***</td>
<td>Screw speed</td>
</tr>
<tr>
<td>0·556</td>
<td>***</td>
<td>Zone 1 temp.</td>
</tr>
<tr>
<td>0·366</td>
<td>***</td>
<td>Zone 2 temp.</td>
</tr>
<tr>
<td>0·206</td>
<td></td>
<td>Screw temp.</td>
</tr>
<tr>
<td>0·126</td>
<td>**</td>
<td>K value</td>
</tr>
<tr>
<td>0·082</td>
<td></td>
<td>Antistatic agent</td>
</tr>
<tr>
<td>−0·171</td>
<td>**</td>
<td>Screw speed × zone 1 temp.</td>
</tr>
</tbody>
</table>

Table 7 Effects table for gloss (%)

<table>
<thead>
<tr>
<th>Effects</th>
<th>Sig.</th>
<th>Term</th>
</tr>
</thead>
<tbody>
<tr>
<td>15·04</td>
<td></td>
<td>Constant</td>
</tr>
<tr>
<td>−14·48</td>
<td>***</td>
<td>Screw speed</td>
</tr>
<tr>
<td>4·08</td>
<td></td>
<td>Zone 1 temp.</td>
</tr>
<tr>
<td>3·81</td>
<td>**</td>
<td>Zone 2 temp.</td>
</tr>
<tr>
<td>−1·92</td>
<td></td>
<td>Screw temp.</td>
</tr>
<tr>
<td>−2·72</td>
<td>*</td>
<td>K value</td>
</tr>
<tr>
<td>−0·75</td>
<td></td>
<td>Antistatic agent</td>
</tr>
<tr>
<td>−4·76</td>
<td>***</td>
<td>Screw speed × Zone 1 temp.</td>
</tr>
<tr>
<td>−4·26</td>
<td>**</td>
<td>Screw speed × Zone 2 temp.</td>
</tr>
<tr>
<td>−2·44</td>
<td></td>
<td>Zone 1 temp. × Zone 2 temp.</td>
</tr>
<tr>
<td>2·32</td>
<td></td>
<td>K value × Screw speed</td>
</tr>
</tbody>
</table>
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. 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 relationship
\[
\% \text{ Gelation} = \frac{P - P_{\text{min}}}{P_{\text{max}} - P_{\text{min}}} \times 100
\]  

The value of \(P_{\text{max}}\) was taken as the maximum value of pressure \(P\) recorded, which is assumed to correspond to 100% gelation. The value of \(P_{\text{min}}\) 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>
<thead>
<tr>
<th>Effects</th>
<th>Sig.</th>
<th>Term</th>
</tr>
</thead>
<tbody>
<tr>
<td>74:16</td>
<td></td>
<td>Constant</td>
</tr>
<tr>
<td>26:79</td>
<td>***</td>
<td>Screw speed</td>
</tr>
<tr>
<td>15:55</td>
<td>***</td>
<td>Zone 1 temp.</td>
</tr>
<tr>
<td>13:25</td>
<td>***</td>
<td>Zone 2 temp.</td>
</tr>
<tr>
<td>4:57</td>
<td>*</td>
<td>Screw temp.</td>
</tr>
<tr>
<td>3:73</td>
<td>*</td>
<td>K value</td>
</tr>
<tr>
<td>3:69</td>
<td></td>
<td>Antistatic agent</td>
</tr>
<tr>
<td>7:58</td>
<td>***</td>
<td>Screw speed × Zone 1 temp.</td>
</tr>
<tr>
<td>4:91</td>
<td>*</td>
<td>Screw speed × Zone 2 temp.</td>
</tr>
<tr>
<td>5:52</td>
<td>*</td>
<td>Zone 1 temp. × Zone 2 temp.</td>
</tr>
</tbody>
</table>

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

<table>
<thead>
<tr>
<th>Effects</th>
<th>Sig.</th>
<th>Term</th>
</tr>
</thead>
<tbody>
<tr>
<td>1:447</td>
<td></td>
<td>Constant</td>
</tr>
<tr>
<td>1:590</td>
<td>***</td>
<td>Screw speed</td>
</tr>
<tr>
<td>0:007</td>
<td></td>
<td>Zone 1 temp.</td>
</tr>
<tr>
<td>0:036</td>
<td></td>
<td>Zone 2 temp.</td>
</tr>
<tr>
<td>0:083</td>
<td></td>
<td>Screw temp.</td>
</tr>
<tr>
<td>0:008</td>
<td></td>
<td>K value</td>
</tr>
<tr>
<td>0:028</td>
<td></td>
<td>Antistatic agent</td>
</tr>
<tr>
<td>0:075</td>
<td></td>
<td>K value × Screw speed</td>
</tr>
<tr>
<td>0:076</td>
<td></td>
<td>Screw speed × Zone 2 temp.</td>
</tr>
<tr>
<td>0:067</td>
<td></td>
<td>Zone 1 temp. × Screw temp.</td>
</tr>
</tbody>
</table>

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. Hence the effect of K value is on melt elasticity and not on degree of gelation per se.

Charpy impact strength
Single notched 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 notched 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 ± 1 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 ± 1 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. 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
For the optimum processing conditions, it was assumed that it was required to maximise the extruder output, 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 $= 165$ °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 $b^*$ level: ($\times$) 15, ($\bullet$) 35, and ($\square$) 55 rev min$^{-1}$ gives the following set of conditions: screw speed $= 35$ rev min$^{-1}$, zone 1 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. 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

<table>
<thead>
<tr>
<th>Trial no.</th>
<th>K value</th>
<th>Die temp., °C</th>
<th>Screw speed, rev min⁻¹</th>
<th>Melt pressure, bar</th>
<th>Melt temp., °C</th>
<th>Torque, %</th>
<th>Output, kg h⁻¹</th>
<th>Gloss, %</th>
<th>Colour strength, b⁺</th>
<th>Charpy impact strength, kJ m⁻²</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>68</td>
<td>195</td>
<td>18</td>
<td>390</td>
<td>187</td>
<td>71</td>
<td>85.8</td>
<td>38.0</td>
<td>4.5</td>
<td>14.27</td>
</tr>
<tr>
<td>2</td>
<td>68</td>
<td>195</td>
<td>24</td>
<td>401</td>
<td>197</td>
<td>74</td>
<td>114</td>
<td>36.2</td>
<td>4.5</td>
<td>15.05</td>
</tr>
<tr>
<td>3</td>
<td>68</td>
<td>200</td>
<td>24</td>
<td>398</td>
<td>195.5</td>
<td>72</td>
<td>117</td>
<td>37.5</td>
<td>4.3</td>
<td>15.99</td>
</tr>
<tr>
<td>4</td>
<td>68</td>
<td>200</td>
<td>18</td>
<td>383</td>
<td>191</td>
<td>71</td>
<td>85.8</td>
<td>45.8</td>
<td>4.7</td>
<td>15.95</td>
</tr>
<tr>
<td>5</td>
<td>68</td>
<td>195</td>
<td>18</td>
<td>366</td>
<td>189</td>
<td>70</td>
<td>84.7</td>
<td>37.8</td>
<td>4.6</td>
<td>15.13</td>
</tr>
<tr>
<td>6</td>
<td>68</td>
<td>195</td>
<td>24</td>
<td>402</td>
<td>198</td>
<td>73</td>
<td>114</td>
<td>32.4</td>
<td>4.5</td>
<td>15.61</td>
</tr>
<tr>
<td>7</td>
<td>66</td>
<td>195</td>
<td>18</td>
<td>383</td>
<td>190</td>
<td>70</td>
<td>85.7</td>
<td>38.7</td>
<td>4.4</td>
<td>13.93</td>
</tr>
<tr>
<td>8</td>
<td>66</td>
<td>195</td>
<td>24</td>
<td>388</td>
<td>194</td>
<td>67</td>
<td>112.8</td>
<td>37.6</td>
<td>4.1</td>
<td>14.09</td>
</tr>
<tr>
<td>9</td>
<td>66</td>
<td>200</td>
<td>24</td>
<td>389</td>
<td>194</td>
<td>68</td>
<td>115.4</td>
<td>41.6</td>
<td>4.1</td>
<td>14.61</td>
</tr>
</tbody>
</table>

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 $2^3$ 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)

<table>
<thead>
<tr>
<th>Effects</th>
<th>Sig.</th>
<th>Term</th>
</tr>
</thead>
<tbody>
<tr>
<td>388·93</td>
<td>**</td>
<td>Constant</td>
</tr>
<tr>
<td>7·86</td>
<td></td>
<td>K value</td>
</tr>
<tr>
<td>−2·95</td>
<td></td>
<td>Die temp.</td>
</tr>
<tr>
<td>10·50</td>
<td>***</td>
<td>Screw speed</td>
</tr>
<tr>
<td>3·50</td>
<td>*</td>
<td>K value × Screw speed</td>
</tr>
</tbody>
</table>

Table 12  Effects for melt temperature (°C)

<table>
<thead>
<tr>
<th>Effects</th>
<th>Sig.</th>
<th>Term</th>
</tr>
</thead>
<tbody>
<tr>
<td>192·63</td>
<td></td>
<td>Constant</td>
</tr>
<tr>
<td>1·75</td>
<td></td>
<td>K value</td>
</tr>
<tr>
<td>−0·50</td>
<td></td>
<td>Die temp.</td>
</tr>
<tr>
<td>5·50</td>
<td>***</td>
<td>Screw speed</td>
</tr>
<tr>
<td>1·00</td>
<td></td>
<td>K value × Screw speed</td>
</tr>
</tbody>
</table>

Table 13  Effects for torque (%)

<table>
<thead>
<tr>
<th>Effects</th>
<th>Sig.</th>
<th>Term</th>
</tr>
</thead>
<tbody>
<tr>
<td>70·14</td>
<td>**</td>
<td>Constant</td>
</tr>
<tr>
<td>3·29</td>
<td></td>
<td>K value</td>
</tr>
<tr>
<td>−0·29</td>
<td></td>
<td>Die temp.</td>
</tr>
<tr>
<td>0·17</td>
<td></td>
<td>Screw speed</td>
</tr>
<tr>
<td>2·17</td>
<td>*</td>
<td>K value × Screw speed</td>
</tr>
</tbody>
</table>

Table 14  Effects for colour (b⁺)

<table>
<thead>
<tr>
<th>Effects</th>
<th>Sig.</th>
<th>Term</th>
</tr>
</thead>
<tbody>
<tr>
<td>4·368</td>
<td></td>
<td>Constant</td>
</tr>
<tr>
<td>0·286</td>
<td>***</td>
<td>K value</td>
</tr>
<tr>
<td>−0·036</td>
<td></td>
<td>Die temp.</td>
</tr>
<tr>
<td>−0·208</td>
<td></td>
<td>Screw speed</td>
</tr>
<tr>
<td>0·042</td>
<td></td>
<td>K value × Screw speed</td>
</tr>
</tbody>
</table>

Table 15  Effects for gloss (%)

<table>
<thead>
<tr>
<th>Effects</th>
<th>Sig.</th>
<th>Term</th>
</tr>
</thead>
<tbody>
<tr>
<td>39·41</td>
<td></td>
<td>Constant</td>
</tr>
<tr>
<td>−1·32</td>
<td></td>
<td>K value</td>
</tr>
<tr>
<td>4·82</td>
<td>**</td>
<td>Die temp.</td>
</tr>
<tr>
<td>−3·08</td>
<td></td>
<td>Screw speed</td>
</tr>
<tr>
<td>−2·11</td>
<td></td>
<td>K value × Screw speed</td>
</tr>
</tbody>
</table>

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

<table>
<thead>
<tr>
<th>Effects</th>
<th>Sig.</th>
<th>Term</th>
</tr>
</thead>
<tbody>
<tr>
<td>14·38</td>
<td></td>
<td>Constant</td>
</tr>
<tr>
<td>1·14</td>
<td></td>
<td>K value</td>
</tr>
<tr>
<td>−0·23</td>
<td></td>
<td>Die temp.</td>
</tr>
<tr>
<td>−0·31</td>
<td></td>
<td>Screw speed</td>
</tr>
</tbody>
</table>

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 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 speed.
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 \(b^*\).

ACKNOWLEDGEMENTS
The authors gratefully acknowledge the assistance of Marino Tognon of EVC (Italia) in carrying out trials on the Krauss-Maffei 50KK extruder at Porto Marghera.

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Plastics, Rubber and Composites 1999 Vol. 28 No. 4