A Practical Approach to Process Optimization Using Design of Experiments

Adam Dreiblatt
Director, Process Technology
CPM Century Extrusion

dreiblatta@centuryextrusion.com
(231) 421-6896
Design of experiments (DOE)

It is not the purpose of this presentation to provide a thorough understanding of statistical Design Of Experiments (DOE)...this would take several days...

This presentation will outline some “practical” approaches of using one DOE method to quickly determine if improved operating parameters and/or screw designs can be identified.
Example DOE results – optimization of dispersion using barrel temperature, screw speed, feed rate and polymer melt index (MI) as variables. Largest influence is screw speed.
For this DOE, a test procedure was developed to ‘quantify’ the compound dispersion quality using numerical values – dispersion value represents number of undispersed particles.
Example DOE results – optimization of dispersion using barrel temperature, screw speed, feed rate and polymer melt index (MI) as variables. Programs calculate the interactions between variables.
DOE – process optimization

Regression Analysis: DISPERSION vs TEMPERATURE, SCREW SPEED, FEED RATE, MI

The regression equation is

\[
\text{DISPERSION} = 60.4 - 0.0063 \times \text{TEMPERATURE} - 0.530 \times \text{SCREW SPEED} + 0.537 \times \text{FEED RATE} - 0.083 \times \text{MELT INDEX}
\]

<table>
<thead>
<tr>
<th>Predictor</th>
<th>Coef</th>
<th>SE Coef</th>
<th>T</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constant</td>
<td>60.37</td>
<td>16.22</td>
<td>3.72</td>
<td>0.003</td>
</tr>
<tr>
<td>TEMPERATURE</td>
<td>-0.00625</td>
<td>0.06085</td>
<td>-0.10</td>
<td>0.920</td>
</tr>
<tr>
<td>SCREW SP</td>
<td>-0.53000</td>
<td>0.04868</td>
<td>-10.89</td>
<td>0.000</td>
</tr>
<tr>
<td>FEED RATE</td>
<td>0.5375</td>
<td>0.1217</td>
<td>4.42</td>
<td>0.001</td>
</tr>
<tr>
<td>MELT IND</td>
<td>-0.0833</td>
<td>0.2704</td>
<td>-0.31</td>
<td>0.763</td>
</tr>
</tbody>
</table>

\( S = 4.868 \) \hspace{1cm} \text{R-Sq} = 92.0\% \hspace{1cm} \text{R-Sq(adj)} = 89.3\%

Analysis of Variance

<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>SS</th>
<th>MS</th>
<th>F</th>
<th>P</th>
</tr>
</thead>
<tbody>
<tr>
<td>Regression</td>
<td>4</td>
<td>3273.75</td>
<td>818.44</td>
<td>34.54</td>
<td>0.000</td>
</tr>
<tr>
<td>Residual Error</td>
<td>12</td>
<td>284.37</td>
<td>23.70</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>16</td>
<td>3558.12</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Source</th>
<th>DF</th>
<th>Seq SS</th>
</tr>
</thead>
<tbody>
<tr>
<td>TEMPERAT</td>
<td>1</td>
<td>0.25</td>
</tr>
<tr>
<td>SCREW SP</td>
<td>1</td>
<td>2809.00</td>
</tr>
<tr>
<td>FEED RATE</td>
<td>1</td>
<td>462.25</td>
</tr>
<tr>
<td>MELT IND</td>
<td>1</td>
<td>2.25</td>
</tr>
</tbody>
</table>

Unusual Observations

<table>
<thead>
<tr>
<th>Obs</th>
<th>TEMPERAT</th>
<th>DISPERSION</th>
<th>Fit</th>
<th>SE Fit</th>
<th>Residual</th>
<th>St Resid</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>-20.0</td>
<td>17.00</td>
<td>26.96</td>
<td>2.71</td>
<td>-9.96</td>
<td>-2.46R</td>
</tr>
<tr>
<td>5</td>
<td>-20.0</td>
<td>47.00</td>
<td>37.71</td>
<td>2.71</td>
<td>9.29</td>
<td>2.29R</td>
</tr>
</tbody>
</table>

R denotes an observation with a large standardized residual

DOE regression analysis can determine operating setpoints for these four parameters to produce a specified dispersion quality.
Statistical software programs are available to design experiments and analyze results. These programs are expensive and require advanced knowledge of statistics.

Such programs are required for more than three variables.

If we can limit the number of variables to three, we can design the experiments and analyze the results without needing such software. Three variables can be visualized in three dimensions.
Full factorial DOE

3 Variable “Cubes” (Full Factorial)

Number of factors: 3  
Number of levels: 2  
Number of trials: \((2)^3 = 8\)

Each corner of the cube represents one of the (8) trials

Three variables can be ‘visualized’ in three dimensions with a “cube plot” – eliminating the need for software to design experiments and analyze results.
Correct statistical procedure for executing these tests requires multiple duplicate tests and randomization to minimize errors. Operating under these conditions would require a large number of tests and would also consume a lot of time and resources (e.g. raw materials) – for example, using barrel temperature as a variable would mean running at low temp conditions, then high temp conditions, then low temp conditions, etc...taking a lot of production time...
Design of experiments - blocking

The term “blocking” is used to group tests together with a common variable – for example, barrel temperature. In this case, all low temperature conditions are run in a sequence, followed by all high temperature conditions.

This procedure introduces some error, but substantially reduces the time to execute the experimental design.

Eliminating replicate tests also introduces some error, but also reduces the time required.
Design of Experiments (DOE)

Full Factorial: 3 Factors, 2 levels

Experimental Plan*:

<table>
<thead>
<tr>
<th>Run. No.</th>
<th>Screw Speed</th>
<th>Feed Rate</th>
<th>Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>(Low)</td>
<td>(High)</td>
<td>(Low)</td>
</tr>
<tr>
<td>2</td>
<td>(High)</td>
<td>(High)</td>
<td>(Low)</td>
</tr>
<tr>
<td>3</td>
<td>(High)</td>
<td>(Low)</td>
<td>(Low)</td>
</tr>
<tr>
<td>4</td>
<td>(Low)</td>
<td>(Low)</td>
<td>(Low)</td>
</tr>
<tr>
<td>5</td>
<td>(Low)</td>
<td>(Low)</td>
<td>(High)</td>
</tr>
<tr>
<td>6</td>
<td>(High)</td>
<td>(Low)</td>
<td>(High)</td>
</tr>
<tr>
<td>7</td>
<td>(High)</td>
<td>(High)</td>
<td>(High)</td>
</tr>
<tr>
<td>8</td>
<td>(Low)</td>
<td>(High)</td>
<td>(High)</td>
</tr>
</tbody>
</table>

*Block (Temperature) and non-randomized to minimize run time
Design of experiments - variables

We want to determine the operating conditions to produce acceptable quality compound at highest productivity.

The most significant variables are (in order of significance):

- Screw design
- Screw speed
- Feed rate
- Feeding position (e.g. premix versus downstream feeding)
- Barrel temperature
- Die pressure
Design of experiments - variables

Testing all of these variables at two levels would require \(2^6 = 64\) trials...and take a long time to complete.

If we limit the test variables to the three most significant operating parameters (versus configuration parameters) – then we can imagine running eight \(2^3\) trials:

- Screw speed
- Feed rate
- Barrel temperature
Design of experiments - approach

It takes approximately 15 minutes for the extruder to stabilize once the operating conditions are set...for each barrel temperature setting, changing screw speed or feed rate should take no more than 15 minutes for each test condition including collecting sample.

- Four tests takes one hour
- Stop machine, change barrel temperature settings
- Repeat four tests (one hour)

Total time required to execute DOE ≈ 2 hours
Design of experiments - limitations

Where do we start?

It is CRITICAL that ALL test conditions be executed in order to get the value out of the DOE (also critical if using statistical software)!

Consider those conditions that would prevent one test condition from being run:

- Torque (amps) too high
- Pressure too high
- Auxiliary equipment limitation (feeder, pelletizer, etc.)
Design of experiments – worst case

**Test condition #1**: low screw speed, high feed rate, low barrel temperature

- Identify ‘worst-case’ where machine can operate (“screening test”)
- All other conditions OK (need to check feeder, pelletizer at low rate)
- Then determine range for each variable (+/-)
Design of experiments - range

What range (+/-) do we use for each variable?

We need to see a large enough difference in processing conditions... ...but not too much!

Screw speed: recommend 15-25% of rpm value
Feed rate: recommend 25-50% of feed rate value
Temperature: recommend 20 to 25°C difference

It is possible that some conditions will produce “bad” product; setting the range for each variable too small (to avoid making bad product) will defeat the purpose of the DOE...
DOE – analyzing results

Analyze samples, enter property data for each test condition on cube plot
- Can enter actual property data (impact, Yellow Index, tensile, etc.)
- Can also rank samples in order of best quality, 1 to 8
DOE – analyzing results

EXAMPLE
- All samples on right side of cube are better than all samples on left side

Higher screw speed is better than lower screw speed!
DOE – analyzing results

EXAMPLE
- Top edge on right side of cube is better than lower edge

Higher temperature is better than lower temperature *at high rpm*
Analyzing results – higher screw speed

If samples on right side are all better than samples on left side –
- run (four) more tests at higher screw speed
- if one edge is better than other, only need two tests
- if max rpm, change screw design to increase shear
Analyzing results – higher specific energy

If the front edge on right side of cube is better than back edge

- These conditions create the highest specific energy
- *This is good indication to change screw design (higher shear)*
Analyzing results – lower screw speed

If samples on left side are all better than samples on right side –
- run (four) more tests at lower screw speed
- If one edge is better than other, only need two tests
- if max % torque (amps), change screw design to reduce shear
Analyzing results – lower specific energy

If the back edge on left side of cube is better than front edge

- These two conditions create the lowest specific energy
- *This is good indication to change screw design (reduced shear)*
Analyzing results – lower feed rate

- If samples on front side are all better than samples on back side –
  - run (four) more tests at lower feed rate
  - If one edge is better than other, only need two tests
  - change screw design to increase shear
Analyzing results – higher feed rate

If samples on back side are all better than samples on front side –
- run (four) more tests at higher feed rate
- If one edge is better than other, only need two more tests
If samples on top are all better than samples on bottom
- run (four) more tests at higher barrel temperature
- If one edge better than other, only need two more tests
Analyzing results – lower temperature

If samples on bottom are all better than samples on top
- run (four) more tests at lower barrel temperature
- If one edge better than other, only need two more tests
Summary – design of experiments

A “practical” approach to DOE can be developed and executed in a production environment with minimum resources.

✓ This method is recommended for developing the operating conditions for new products.

The results from this type of DOE will lead to the operating conditions (and screw designs) that produce the highest compound quality at the highest productivity.
THANK YOU!