Process Characterization Using Response Surface Methodology

A Senior Project Presented to

The Faculty of the Statistics Department

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In Partial Fulfillment of the Requirements for the Degree

Bachelor of Science, Statistics

By Katherine A Eng

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# Table of Contents

Introduction ........................................................................................................................................... 2  

Background ........................................................................................................................................ 2  

Experimentation .................................................................................................................................. 4  
  Experimental Design ....................................................................................................................... 4  
  Data Collection .............................................................................................................................. 7  

Results ............................................................................................................................................... 8  

Recommendations ............................................................................................................................. 9  

Further Study .................................................................................................................................... 9  

References ......................................................................................................................................... 9  

Acknowledgements ........................................................................................................................... 9
Introduction
A Santa Barbara county engineering firm proposed a collaboration with the Cal Poly Statistics Department to investigate the sources of variability in a certain measurement process, understand normal operability characteristics of the machine, reduce variability in machine measurements, establish process monitoring and control for the system, and verify utility of the proposed process control through designed experimentation. The proposed process control will be reviewed for potential integration into the operating specification system, and the two students (one statistics, and one materials engineering major) involved in the project have a unique opportunity to gain valuable experience applying their statistical and engineering knowledge towards a real world problem prior to graduation. This project is on-going and may be available for future Cal Poly student involvement, as further experimentation is necessary to devise, implement, and verify statistical process control measures. Participation in such a collaboration requires complete discretion from all Cal Poly contributing members, as all involved parties are under nondisclosure agreements. Proprietary information may not be revealed or released to anyone not directly involved in the project.

Background
All measurement systems have intrinsic variability. Measurement variability is often characterized into two different sources:

1. **Repeatability**, also called precision variability or equipment variability, is a measure of dispersion of measurement results when all measurements are made under the same conditions (e.g., same appraiser, same equipment, same production environment, same time period).
2. **Reproducibility** is a measure of dispersion of measurement results when the measurement conditions change (e.g., different appraisers, different machines, or different measurement conditions).

The measurement process studied was found to have both reproducibility and repeatability variability in all three measurement outcomes (coded y1, y2, and y3). Three years’ worth of testing data have been meticulously recorded by the engineering firm for all tests conducted on a single material type manufactured by one vendor, and variance component analysis on data collected on that specimen type indicate large portions of observed variability are due to day-to-day effects and test-to-test effects nested within day. Further analysis into the historical data shows that trends exist in each of the three measurements. Measurements on y1 (Figure 1) and y2 (Figure 2) are regularly overestimated compared to the vendor claims (horizontal line), and measurements on y3 (Figure 3) are regularly underestimated compared to vendor claims. In addition, there are mean shifts in measurement readings from the first specimen measured to the second specimen measured, and so on. Therefore, measurement readings do not appear to be independent of one another.
Figure 1 – Mean and range plot illustrating positive bias of y1 measurements compared to vendor claims.

Figure 2 – Mean and range plot illustrating positive bias of y2 measurements compared to vendor claims.
Experimentation
There are known sources of variability attributable to these erratic measurements; however, no studies have been conducted to empirically quantify the effects of these sources of variability on the measurement outcomes. The factors chosen to be investigated in this experiment are designed to address how controllable parameters influence measurement outcomes.

Experimental Design
The approved experimental design investigated three factors:

1. Sample holder type
2. Rest time
3. Air flow rate into the machine / air pressure

Sample holder type is a binary categorical variable; rest time is the duration of machine inactivity between sample tests; and air flow rate is set by an air pressure gradient across a flowmeter. Two levels of holder type (A and B), five levels of rest time, and five levels of air flow would be tested. Coded levels of these two quantitative factors are shown below in Figure 4.

<table>
<thead>
<tr>
<th>Variable</th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rest Time</td>
<td>$-\sqrt{2} = -1.414$, $-1$, $0$, $1$, $\sqrt{2} = 1.414$</td>
</tr>
<tr>
<td>Pressure</td>
<td>$-\sqrt{2} = -1.414$, $-1$, $0$, $1$, $\sqrt{2} = 1.414$</td>
</tr>
</tbody>
</table>

Figure 4 – Coded experimental factors and levels.
A central composite design (CCD) is an efficient design that can effectively and confidently predict measurement outcomes at most rest time and pressure combinations within the design space (Figure 5). From the data collected in a CCD, heat release measurements can be predicted using two quantitative variables in a regression-like fashion using response surface methodology. Every quantitative treatment combination that is incorporated into a central composite design is run one time except for the (0,0) treatment combination (the center runs). Apart from the number of center runs, this is an unreplicated design. By replicating the center run treatment combination, internal estimates of error can be estimated for the model. The number of center runs that should be conducted in an experiment will be discussed later.

There are three kinds of design points used in a central composite design: 1) the center runs; 2) factorial runs; and 3) axial runs. The factorial points are made up of the (±1, ±1) treatment combinations. When only factorial runs are conducted, only linear effects and interaction terms can be estimated. With the addition of center runs, curvature can be estimated in the response surface model. With the addition of axial points, the points coded 0 for one factor and ±√2 in the other factor, second order models can be constructed (Figure 6). The magnitude and sign of these second order coefficients determine the shape of the predicted response surface (Figure 7).

Central composite designs have several desirable properties, including rotatability, sphericity, and stability. When a design is rotatable, the variance of the predicted values, or the prediction variance, depends only on a treatment combination’s distance away from the center of the design space in a given experiment. The prediction variance at any treatment combination is the product of the MSE and the treatment combination’s leverage value (Figure 8). Therefore, all treatment combinations that are the same distance away from the center have the same prediction variance (Figure 9). To achieve rotatability, the axial distance should be √k = √2, where k=2 factors for this CCD. Since this design region is spherical (the distance from each of the experimental factor combinations from the center are the
same, \( \sqrt{2} \)), there is low prediction variance within that spherical region. To make these prediction variances stable throughout the entire design region, an appropriate number of center runs, should be conducted. When there are too few center runs (e.g., 2 center runs), the prediction variance is not uniform throughout the design region, but when there are a sufficient number of center runs (e.g., 4 center runs), there is fairly uniform prediction variance (Figure 10).

To accommodate a categorical binary variable (e.g., holder type), a central composite design is run for each holder type, so that two different response surfaces are fit for each of the three measurement outcomes.

\[
E(y) = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{12} x_1 x_2
\]

Figure 6 – A second order model

![Second order model graphs](image)

Figure 7 – Second order response surface shapes (Walker)

\[
s^2 \{ \hat{y} \} = \text{MSE} \left[ \frac{1}{n} + \frac{(\hat{X} - \bar{X})^2}{\sum (X - \bar{X})^2} \right]
\]

Figure 8 – The prediction variance formula (Walker)
Data Collection

It was also discovered on the day of data collection, 25 April 2013, that the measurement apparatus was not capable of running the two higher levels of air pressure. Unfortunately, testing had already begun when this limitation was discovered. A CCD could not be run, since the factor-levels used are chosen precisely in order to obtain the design properties listed in the previous section. Data were collected using new pressure – rest time treatment combinations chosen on site (Figure 11).
Results
The amount of variability in the sample holder level A data is lower than the sample holder level B data, and statistically significant response surface models were constructed for y1 (Figure 12) and y2 (Figure 13) when using sample holders at level A. The y1 model is a second order model, with significant quadratic effects on both rest time and pressure and a significant interaction effect. The y2 model was a first order model, where only pressure has a significant linear effect. No significant results were found for the y3 measurement, or with any measurement outcomes using level B sample holders.

Figure 12 – Second order response surface for y1, using sample holders at level A
Figure 13 – First order response surface for y2, using sample holders at level B
**Recommendations**

Based on these response surface models, the Cal Poly team made the following recommendations to the engineering firm: 1) use the identified factor-level settings that would optimize \( y_1 \) and \( y_2 \) measurements to match vendor supplied data; and 2) use sample holders A to reduce measurement variability in \( y_1 \) and \( y_2 \) measurements. Implementing these recommendations will decrease variability and diminish biases in measurement outcomes.

**Further Study**

Additional experimentation needs to be conducted to observe day-to-day variability in measurement outcomes. Exploring higher levels of air pressure would also be warranted, since the machine was not capable of running high air pressure levels at the time of experimentation.

**References**


**Acknowledgements**

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