# CHANGEOVER TIME FOR SINGLE AND TWIN-SCREW EXTRUDERS

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## Abstract

Changing extrusion formulations causes downtime and wasted materials before a new steady state is achieved. Accurate determination of changeover times minimizes these negative impacts. In this work, changeover times are compared for single and twin-screw extruders by online Raman spectroscopy. Our results show that twin-screw extruders usually purge more quickly than single-screw extruders, attributed to their self-wiping design.

## Introduction

The purpose of this work is to determine factors that influence downtime when changing extrusion formulations. Minimizing this downtime has the potential to increase production and reduce waste. In particular, this work aims to quantify changeover times for a single-screw extruder (SSE), and compare these times to those found previously for a twin-screw extruder (TSE). Our hypothesis is that TSEs will changeover more quickly, as a result of their self-wiping design.

Due to the complex, transient nature of extrusion formulation changes, theory is inadequate for accurately predicting residence times and changeover times. Therefore, an experimental approach is usually implemented, in which a step change is imposed on the feed formulation and the discharge composition is monitored until a new steady state is reached.

Several papers have reviewed online methods for measuring exit composition in extruders [1-3]. Some of the most sensitive and robust techniques include spectroscopic methods [4]. For example, Alig et al. demonstrated online near-infrared (NIR), Raman, and ultrasonic spectroscopy to quantify antioxidant and carbon black concentrations in polymer extrudates [5]. Coates et al. also utilized online NIR, Raman, ultrasonic, and attenuated total reflectance infrared (ATR-IR) spectroscopy to monitor a variety of mixtures and reactions for molten polymers [6].

Gilmor et al. used a charge coupled device spectrometer to study changeover times between red and blue colored masterbatches in a SSE.[7] They found changeover times ranging from 3 – 20 minutes when transitioning between a white purge material and 95 / 5 blends of linear low density polyethylene (LLDPE) DOWLEX™ 2045 resin and colored masterbatch. Changeover time depended on the screw speed (throughput) and the type of masterbatch used. The authors attributed the masterbatch differences to viscosity and type of colorant, although viscosity correlations with changeover time were not established.

Changeover time has also been studied in TSEs [8]. Wang et al. used online Raman spectroscopy to measure the time to change between polyethylene (PE) and polystyrene (PS) on a 25 mm TSE. Of the four factors studied, screw speed and mixing zone location showed no significant effect on changeover time, whereas increasing the end/initial viscosity ratio and the throughput significantly decreased changeover time. Measured changeover times ranged from 1 – 3 min for rates between 4.5 and 9 kg/h. This study also validated the Raman quantitation of exit composition via offline ATR-FTIR.

This study uses the same Raman instrument to measure changeover times between PE and PS in a SSE, with the purpose of finding factors that reduce time and waste in material transitions.

## Materials and Methods

LLDPE DOWLEX™ 2247G resin (MFI = 2.3 dg / min / 190 °C / 2.16 kg, density = 0.917 g/cm$^3$) and LLDPE DOWLEX™ 2045G resin (MFI = 1.0 dg / min / 190 °C / 2.16 kg, density = 0.920 g/cm$^3$) were used to examine changeover time as a function of viscosity. PS STYRON (Trademark of Trinseo) 685 D resin (MFI = 1.5 g / 5 min / 200 °C / 5 kg, density = 1.05 g/cm$^3$) was alternately fed with the polyethylenes to produce strong, distinct Raman peaks.

A 25.4 mm diameter, 24:1 L:D single-screw extruder with a general PE screw was placed in series with a 1.28 cm$^3$/rev gear pump (Maag 22-6) and a die. The extruder speed was automatically controlled by the gear pump inlet pressure, set to a target of 1.72 MPa. All temperatures were set to 220 °C, except the first two extruder zones, which were 168 °C and 204 °C, respectively.

Changeover times were studied by changing the feed from 100% PE → 50/50 wt% PE/PS → 100% PS → 50/50 wt% PE/PS → 100% PE. This was done at two sets of flow rates (30 and 80 gear pump rpm) for two different LLDPEs. During changeovers, the feed level was allowed to decrease to the top of the screw flights. Then, the new formulation was added to the hopper and the start time was recorded. Blends were dry mixed in zippered bags before feeding. This differs from twin-screw feeding procedures, which rely on gravimetric feeders to control flow rate. The melt temperature, gear pump outlet pressure, and average extruder speed were recorded for each steady state.

A BWTek iRaman Pro with Kaiser optical probe was connected to a pressure gauge port at the extruder outlet to monitor composition (<2 s between spectra). More information on the instrument and optical probe may be found in Wang et al. [8]. Pure component spectra were obtained by averaging sample spectra over several minutes at steady state.

Raman signal was used to calculate percent PE and PS near the probe location (i.e., extruder exit). First, classical least squares (CLS) was used to fit sample spectra with a linear combination of PE and PS reference spectra within the 2750-3150 $cm^{-1}$ spectral region (Figure 1) [9-11]. Raw CLS signal strength is sensitive to sample transparency, which changes dramatically from the pure materials (transparent melt, strong signal) to the blends (opaque melt, weaker signal). Therefore, one additional corrective term is necessary to fix the 50/50 wt% blend steady state signal at 50% PS ($\alpha_{PS}/\alpha_{PE}$, where $\alpha$ is Raman response factor). Percent PS is calculated from CLS signal by Equation 1.

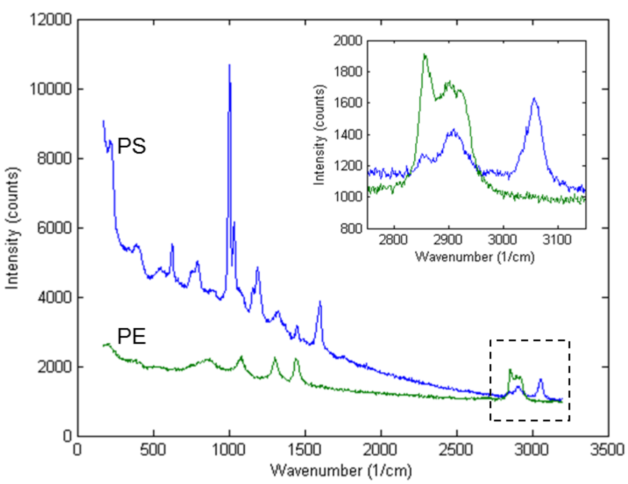


Figure 1. Raman spectra of pure PE and PS

$$\% \text{ PS} = \frac{(\text{PS CLS signal})}{(\text{PS CLS signal}) + (\text{PE CLS signal}) \times \frac{\alpha_{PS}}{\alpha_{PE}}} \quad (1)$$

Changeover start times were manually recorded in a text file, and read as input to an online MATLAB analysis program. The MATLAB program parses the Raman data between changeover start times, calculates percent PS for each time-point, normalizes percent PS and time, fits a curve to the data, and finally extracts a changeover time. This program runs online, to provide operators with feedback for when changes are complete.

Noise in the Raman signal makes it difficult to pick a changeover time value from raw data. Therefore, a Double Weibull curve was fit to the data for each changeover. It is clear that the standard deviation is larger for 50/50 blends than pure materials (opaque melt leads to a weaker signal), so bi-square weightings were given to the errors. The fitted curve is monotonic, so picking a time from the fitted curve is easy and more accurate. The 99% changeover time (99% COT) is defined in Equation 2, where percent PS initial and final values are averages of the first or last 10 data points.

$$\% \text{ PS}(99\% \text{ COT}) = [\% \text{ PS}(\text{final}) - \% \text{ PS}(\text{initial})] \times 0.99 + \% \text{ PS}(\text{initial}) \quad (2)$$

Figure 2 shows a sample output of CLS raw response and percent PS versus time. The black vertical dashed lines indicate a change in feed formulation, and the black vertical solid lines indicate a 99% COT. The red solid line indicates the Double Weibull curve fit. Three fitted changeover time curves showed significant deviation from the raw data, due to a large number of points after the changeover was complete, coupled with drift in the raw data signal at steady state. These three curves were fit using only 400 data points, which resulted in more reasonable fits and changeover times.

Viscosity ratio was calculated as a function of temperature, shear rate, and composition. Pure component shear viscosities were obtained at 200 °C, 220 °C, and 240 °C by parallel plate rheology (Figure 3). Cross model and Arrhenius fits were used to extrapolate viscosity over a range of shear rates and temperatures (Equation 3), where $\eta_0$ is zero shear viscosity, $\dot{\gamma}$ is shear rate, $E_A$ is a fitted activation energy, $R$ is the Ideal Gas constant, $K$ is a Cross model material constant, $m$ is the power law exponent, and $T_0$ is the reference temperature. $T$ was the extrudate hand-held melt temperature for each run.

$$\eta(\dot{\gamma}, T) = \frac{a_T \, \eta_0}{1 + (K \, a_T \, \eta_0 \, \dot{\gamma})^{1-m}}$$

$$a_T = \exp\left[\frac{E_A}{R}\left(\frac{1}{T} - \frac{1}{T_0}\right)\right] \quad (3)$$

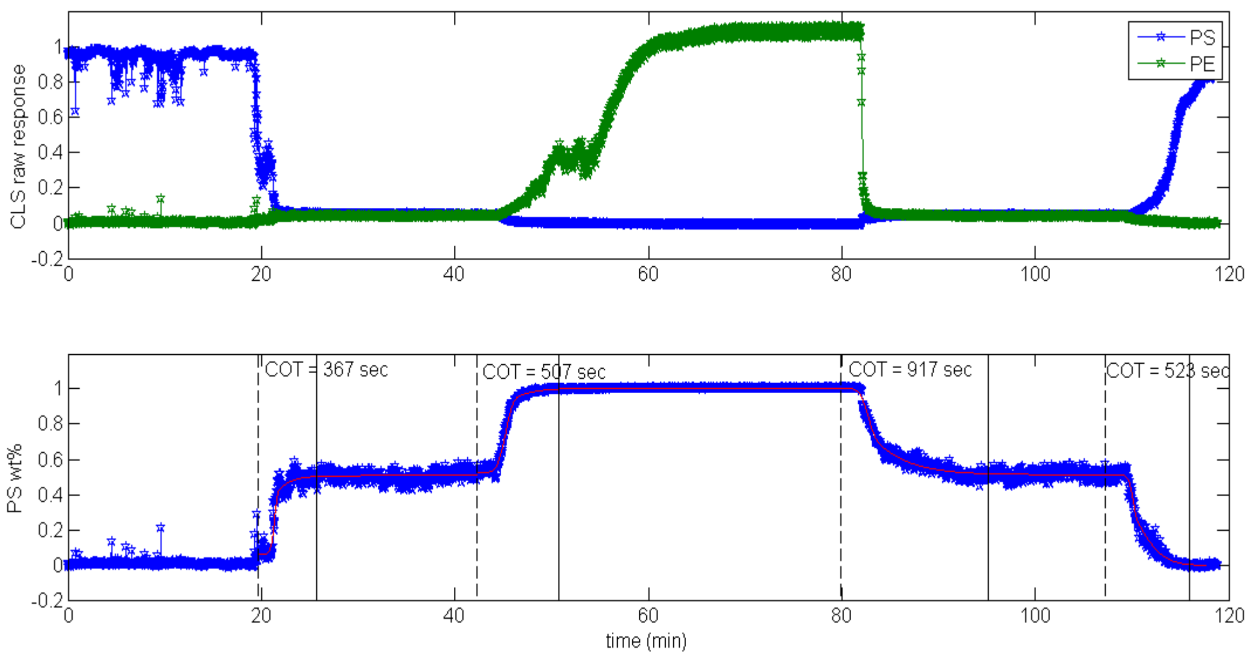


Figure 2. Sample changeover time results for the first five entries in the Appendix; (top) Raw signal response versus time. (bottom) Percent PS versus time.

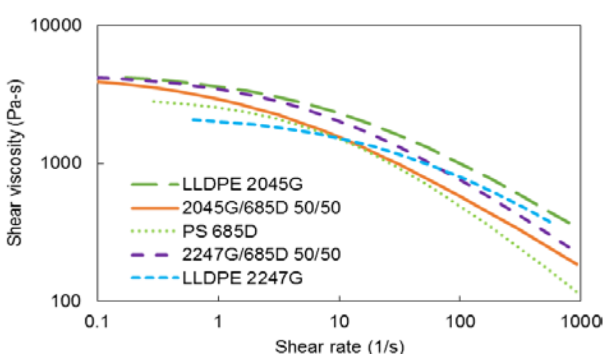


Figure 3. Shear viscosity Cross model fits of pure materials and blends at 240 °C.

The average shear rate in the extruder is estimated as the metering section shear rate (Equation 4), where $D_c$ is the screw core diameter, $N$ is the extruder speed (rpm), and $H$ is the distance from the screw root to the barrel [12]. For this particular SSE, $D_C \approx 22.8$ mm and $H \approx 1.27$ mm. Finally, viscosity ratio is calculated as the steady state viscosity of the final material divided by the steady state viscosity of the initial material.

$$\dot{\gamma} = \frac{\pi D_C N}{H} \qquad (4)$$

There are many errors associated with these viscosity estimations. The values are extrapolated outside the temperature range of experimental rheology data (200 – 240 °C). This is also true for the shear rates, which are estimated to be much higher than any measured in rheology. Both of these parameters are not constant, and vary spatially in the extruder. However, the estimated shear viscosity ratios are expected to correlate with true viscosity ratios in extrusion, and therefore are still included as a factor for changeover time.

## Results and Discussion

Changeover times for the SSE experiments are summarized in the Appendix, along with the relevant processing parameters. The measured melt temperatures show significant shear heating (up to 74 °C), similar to previous TSE trials.[8] The mass flow rates are not constant due to density differences between PE and PS, but were generally around 1.8 and 4.5 kg/h for the two given gear pump speeds (30 and 80 rpm, respectively). As expected, the gear pump outlet pressure varies directly with viscosity. Changeover times ranged from 2.5 to 15 minutes, generally longer than those found for a 25 mm TSE.

Changeover times were analyzed in the Fit Model platform of SAS JMP, with factors viscosity ratio and gear pump rpm (results shown in the Appendix, Figure A). ANOVA shows that there is at least one significant factor at a 95% confidence level ($p < 0.0001$). Effects tests show that both viscosity ratio and gear pump speed are significant factors ($p = 0.0024$ and $p < 0.0001$, respectively). Other measured factors, such as melt temperature, were either not significant or highly collinear with the two significant factors. Higher order terms were not appropriate for this screening, and independent cross terms were not significant. The final prediction expression shows that changeover time increases with decreasing flow rate and increasing viscosity ratio.

Flow rate is a strong effect, indicated by the large prefactor in the model prediction and the low *p*-value in the effects test. It is somewhat surprising that the viscosity term shows the opposite effect as the analogous TSE experiments. Several factors that may have affected this result are the different softening/melting temperature profiles of PE and PS, pellet sizes and shapes, and melt densities. Also, as stated before, there is a large degree of uncertainty in the viscosity calculations.

There are several differences between the SSE and TSE experiments, which must be highlighted. First, the residence volume of the 25 mm TSE is approximately 390 $cm^3$ (assuming 50% fill fraction), compared to 220 $cm^3$ for the SSE. This would favor the SSE for shorter changeover times. Secondly, the TSE has gravimetric feed control whereas the SSE is flood fed, but this is ameliorated by using gear pump speeds that roughly matched the 4.5 kg/h settings on the TSE feeders. Third, a different analysis method was used for determining changeover time for these two experiments. For the TSE, the data was fit to a Sigmoidal curve and changeover times were defined when a composition change of less than 0.1% occurred between time points. The SSE changeovers from this study were fit using both equations and criteria (Sigmoidal / 0.1% change between points, Double Weibull / 99% of concentration change), and there was about 22 s difference on average. This is not enough to affect the main conclusions of our work (that SSEs change over slower than TSEs). Fourth, the control system on the SSE is a feedback loop, where the extruder speed is controlled to keep the gear pump inlet pressure constant. This control loop may have caused the system to take more time to equilibrate, but a steady inlet pressure was achieved within a minute, so this is not considered to be a large factor. Lastly, the SSE had more discharge pressure than the TSE (due to the die and gear pump), which has an unknown effect on changeover time.

If a comparison is to be made between TSE and SSE changeover time, it should be at similar mass throughput with the same materials, since both are significant factors for changeover time in SSEs and TSEs. This occurred for four changeovers at 4.5 kg/h, between 685D and 2045G resins, compared in Figure 4. From this data, the twin-screw extruder appears to change over more rapidly.

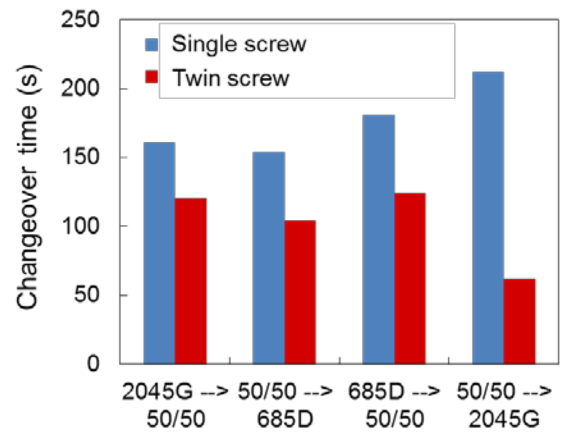

Figure 4. Comparison of changeover time for single-screw and twin-screw extruders at approximately 4.5 kg/h throughput.

## Conclusions

Changeover times for a SSE were estimated by online Raman, analyzed in MATLAB and JMP. Increasing throughput and decreasing viscosity ratio decreased changeover time.

Changeover times ranged from 2.5 – 15 min, and were generally longer than those found in a 25 mm TSE. This confirmed our hypothesis; that TSEs change formulation more quickly, attributed to their self-wiping capability.

## Acknowledgements

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## References

1. L. Saerens, C. Vervaet, J. P. Remon, T. De Beer, *Journal of Pharmacy and Pharmacology*, **66**, 180 (2014).
2. A. Poulesquen, B. Vergnes, P. Cassagnau, A. Michel, O. S. Carneiro, J. A. Covas, *Polymer Engineering & Science*, **43**, 1849 (2003).
3. M. D. Wetzel, C. K. Shih, U. Sundararaj, *ANTEC Papers*, **3**, 3707 (1997).
4. D. Fischer, K. Sahre, M. Abdelrhim, B. Voit, V. B. Sadhu, J. Pionteck, H. Komber, J. Hutschenreuter, *Comptes Rendus Chimie*, **9**, 1419 (2006).
5. I. Alig, D. Fischer, D. Lellinger, B. Steinhoff, *Macromolecular Symposia*, **230**, 51 (2005).
6. P. Coates, S. Barnes, M. Sibley, E. Brown, H. G. Edwards, I. Scowen, *Polymer*, **44**, 5937 (2003).

7. C. Gilmor, S. T. Balke, F. Calidonio, A. Rom-Roginski, *Polymer Engineering & Science*, **43**, 356 (2003).
8. J. Wang, C. Thurber, X. Chen, M. Read, N. Horstman, C. Pavlicek, J. Stanley, *SPE ANTEC Papers*, **1**, 863 (2016).
9. D. M. Haaland, R. G. Easterling, *Applied Spectroscopy*, **34**, 539 (1980).
10. D. M. Haaland, R. G. Easterling, D. A. Vopicka, *Applied Spectroscopy*, **39**, 73 (1985).
11. K. R. Beebe, R. J. Pell, M. B. Seasholtz, "Chemometrics: A practical guide", Wiley (1998).
12. G. A. Campbell, M. A. Spalding, "Analyzing and troubleshooting single-screw extruders", Cincinnati, OH, Hanser (2013).

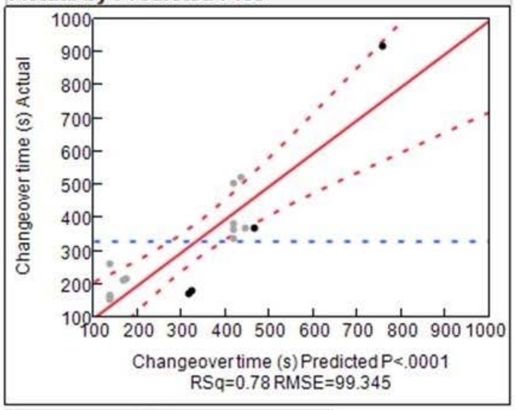
# Appendix

Table 1. Changeover time results

| Material | Gear pump (rpm) | Melt T (°C) | Mass flow (kg/h) | Gear pump outlet pressure (MPa) | Viscosity ratio (-) | Changeover time (s) |
|---|---|---|---|---|---|---|
| 2045G | 30 | 269 | 1.54 | 8.8 | | |
| 50/50 | 30 | 270 | 1.71 | 6.2 | 0.14 | 367 |
| 685D | 30 | 262 | 2.02 | 4.8 | 0.02 | 507 |
| 50/50 | 30 | 265 | 1.80 | 5.6 | 89 | 917 |
| 2045G | 30 | 277 | 1.55 | 8.2 | 4.4 | 523 |
| | | | | | | |
| 2045G | 80 | 294 | 3.79 | 12.3 | | |
| 50/50 | 80 | 284 | 4.47 | 7.93 | 0.12 | 161 |
| 685D | 80 | 275 | 5.15 | 6.76 | 0.02 | 154 |
| 50/50 | 80 | 284 | 4.59 | 7.65 | 49 | 181 |
| 2045G | 80 | 294 | 3.88 | 12.1 | 8.4 | 212 |
| | | | | | | |
| 2247G | 30 | 264 | 1.57 | 6.27 | | |
| 50/50 | 30 | 268 | 1.67 | 4.76 | 0.10 | 339 |
| 685D | 30 | 263 | 1.96 | 4.55 | 0.11 | 385 |
| 50/50 | 30 | 265 | 1.75 | 4.62 | 12 | 369 |
| 2247G | 30 | 279 | 1.60 | 5.79 | 7.3 | 372 |
| | | | | | | |
| 2247G | 80 | 289 | 4.01 | 8.96 | | |
| 50/50 | 80 | 281 | 4.13 | 6.48 | 0.09 | 261 |
| 685D | 80 | 280 | 5.16 | 6.55 | 0.02 | 166 |
| 50/50 | 80 | 281 | 4.27 | 6.62 | 47 | 171 |
| 2247G | 80 | 293 | 3.82 | 8.96 | 9.9 | 218 |

**Whole Model**

**Actual by Predicted Plot**




**Summary of Fit**

| | |
|---|---|
| RSquare | 0.781059 |
| RSquare Adj | 0.747375 |
| Root Mean Square Error | 99.34537 |
| Mean of Response | 331.4375 |
| Observations (or Sum Wgts) | 16 |

**Analysis of Variance**

| Source | DF | Sum of Squares | Mean Square | F Ratio |
|---|---|---|---|---|
| Model | 2 | 457714.39 | 228857 | 23.1883 |
| Error | 13 | 128303.54 | 9870 | Prob > F |
| C. Total | 15 | 586017.94 | | <.0001* |

**Effect Tests**

| Source | Nparm | DF | Sum of Squares | F Ratio | Prob > F |
|---|---|---|---|---|---|
| Viscosity ratio | 1 | 1 | 139900.33 | 14.1750 | 0.0024* |
| Gear pump speed (rpm | 1 | 1 | 319347.76 | 32.3570 | <.0001* |

**Prediction Expression**

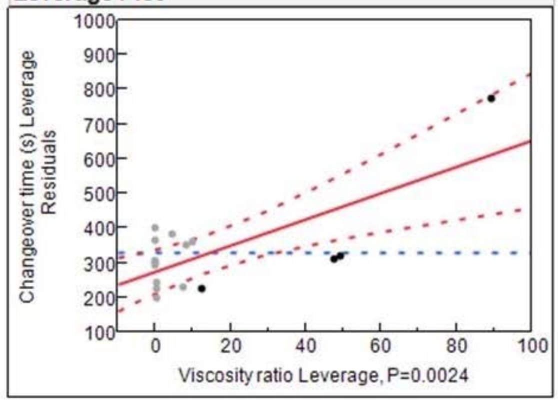
588.104426059433

+ 3.78437038453293 * Viscosity ratio

+ -5.6511237333843 * Gear pump speed (rpm)

**Viscosity ratio**

**Leverage Plot**




**Gear pump speed (rpm)**

**Leverage Plot**

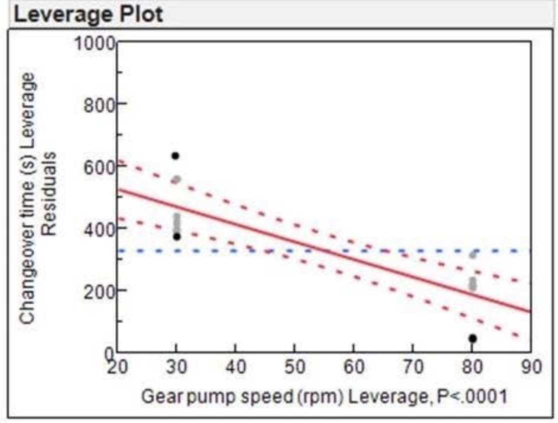



Figure A. Model fit for SSE changeover time, as a function of viscosity ratio and gear pump speed