

**DEVELOPMENT OF AN EXTRUDER BASED MELT INDEX SOFT SENSOR****Ian R. Alleyne¹, Sirish L. Shah¹, Uttandaraman Sundararaj¹, Brent West²**¹*Department of Chemical and Materials Engineering,
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Abstract: The control of polymer quality has become increasingly important as the production of polymer grades has increased in quantity and diversity. On a typical polymerization plant there is an extruder downstream of the reactor. This paper provides details on the use of the operating variables of such an extruder as an empirical sensor for the melt flow index, an important indicator of polymer quality. An empirical model was built in several stages. First a simple model was built which related the melt index with the extruder pressure. After many iterations a model which also included extruder speed and temperature compensation and a bias updating procedure was developed. The final bias updated model has been installed at the plant and detects a change in the melt index nine (9) minutes before the online instrument. The end goal is to use this soft sensor to build a data based plant model, and subsequently use this model to compute optimal grade transition trajectories in the plant. *Copyright © 2005 IFAC*

Keywords: Polymer Reactor control, Soft sensing, Trajectories, Polymerization, Predictive control, Optimal Trajectory, System Identification.

1. INTRODUCTION

The increased reliance on polymers with specifically tailored properties for different applications has been documented extensively (Alperowicz 2005). Previously the specifications for polymer grades and products were very relaxed. However, with the advent of much larger capacity plants; many smaller plants are moving from commodity to speciality polymers which have higher profit margins. These products however have tighter specifications. Thus variability which was acceptable before is no longer tolerable. This implies online properties of polymers need to be controlled more tightly. The more diverse, lower volume product requirements by speciality customers forces the product specification required from the plant to change regularly. Therefore reliable online polymer quality measurements are very critical.

This research was undertaken in collaboration between industry and academia. The plant in this study was the AT Plastics EVA high pressure polymerization plant in Edmonton, Canada. The main polymer quality variable on the plant was the melt index. This plant ran approximately twenty grades of polymer regularly in the 1 – 1000 gm/min. melt index range. The online reading for this melt index was determined by an online rheometer. This instrument gave an update every six (6) minutes and was subject

to transport lags. These two problems were not significant as there are system identification algorithms which can compensate for these issues. However, a more significant issue, because of the large range of melt index measurement required, was that the rheometer used several models and die sizes. Each die has a manufacturer's recommended pressure range for which the readings are very close to linear. However, once the instrument gets close to one of the limits, i.e. the polymer is too hard or soft for the particular die and measurement temperature, the readings become unreliable. This is acceptable if the plant is running a grade campaign which does not have a significant change in melt index. Therefore, the goal of modelling the grade transitions was severely hampered because the rheometer data during the grade transitions were plagued with issues such as the unit having to be switched off or becoming unreliable, as it needed die changes or gave inaccurate results because it was at the end of its linear range. It was clear that some new variable was required to give online polymer melt index values.

The measurement of polymer quality through the use of indirect variables (soft sensors) has been the subject of much research. Ohshima and Tanigaki 2000 gave a comprehensive review of property estimation methods published for different polymerization processes. The typical design of these

soft sensors involves building relationships between the process control variables, such as the reactor, pressure and temperature and the polymer property to be controlled. The method of building this relationship typically involves methods such as linear observers (Lines et al. 1993), extended Kalman Filters (Scali et al. 1997), nonlinear parameter estimation (Kiparissides et al. 1996), neural networks (Chan and Nascimento 1994) and partial least squares (Han et al. 2005). Here a different approach is taken; after the reactor there is an extruder, in which many variables are monitored. The identification of the relationship between these and the polymer properties is the main subject of this paper.

Watari et al. 2004 and Nagata et al. 2000 independently used measurements from an extruder estimating the properties of molten polyethylene. However, their methods required the installation of a fibre optic sensor at the end of the extruder to obtain NIR (near infra-red) readings. The method proposed here uses the raw data which is commonly measured and monitored on extruders to give an indication of the melt index of the polymer produced on the reactor.

1.1 Outline

The plant at which this project was undertaken is described next followed by a description of the procedure for development of the melt index soft sensor. This includes the method for selection and lag times of the extruder variables. More details on the modelling of the errors and the significant disturbances that were the cause of these errors are then given. Methods for reducing these errors are also detailed.

1.2 Process Description

The plant considered is an EVA polymerization plant. The plant has the ability to manufacture polymers over a wide range of melt indices and vinyl acetate (VA) content. The flow sheet for the plant is shown in Figure 1.

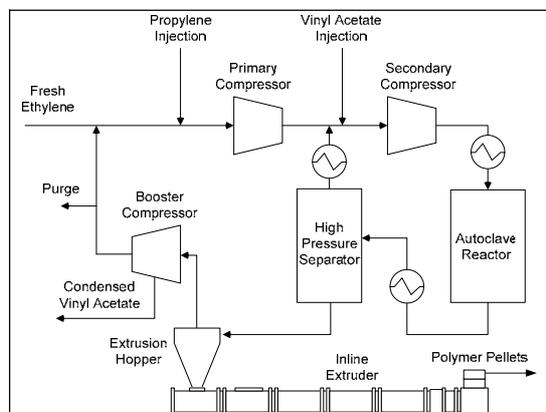


Figure 1 – Process Flowsheet

The monomers used are ethylene, vinyl acetate and propylene (used as a modifier). These are compressed to supercritical conditions and then mixed with peroxide-based initiators in a multi-zone autoclave

reactor. An exothermic polymerization reaction occurs in the autoclave, and about twenty percent of the monomer is polymerized in a single-pass. The autoclave product is cooled and then goes through two separation stages where the unconverted monomers are recycled. The polymer product is then compounded with additives in the extruder before it is pelletized. There can be a build-up of inert components and unwanted propylene in the recycle loop and a purge stream is used to control this. At the booster compressor the majority of the Vinyl Acetate (VA) condenses. This is collected, refined, and then reused with fresh VA. There are three main heat exchangers (coolers) shown. These are the feed gas coolers, product coolers and return gas coolers. These become fouled over time with polymer. When this occurs, the pressure drop across the cooler becomes significant and the process efficiency is compromised. This fouling over time causes a drift in the pressures at the extruder. An operator controlled “cooler cook” is usually performed to remove the fouling. This involves increasing the temperature of the contents of the heat exchanger to remove the polymer lining from the heat exchanger inner wall.

2. MELT INDEX SOFT SENSOR

The steps in building the soft sensor for the melt index are detailed here. It was observed that the plant operators relied on the extruder pressure to give them an indication of the melt index of the polymer whenever the online reading was offline.

2.1 The Extruder

A schematic of the extruder is shown in Figure 2. Several pressures and temperatures are monitored. The extruder screw is driven by a motor. The motor’s frequency is modulated (thus screw speed) to maintain a constant level in the extruder feed hopper. There is a mesh screen pack between the last two pressure sensors on the extruder. This mesh is very fine and becomes clogged with solids and gel particles over time. This causes the extruder variables, predominantly the upstream pressure, to change even if the flow rate and other conditions are constant. All the pressures at the extruder drift due to cooler fouling; while the pressures just before the screen pack have an even more significant drift because the screen pack becomes clogged.

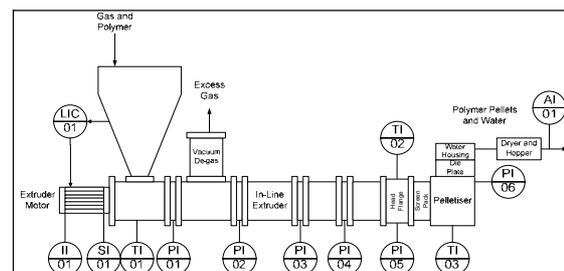


Figure 2– Extruder Schematic

After the extruder there is a pelletizer. The polymer pellets are dried, and then pass through a hopper

before a side stream is used to measure the melt index with the online rheometer.

The extruder is downstream of the reactor thus changes in the melt index of the polymer would occur first at the reactor and its effect felt at the extruder. Clearly, the online rheometer being down stream of the extruder detects changes in the melt index much later than the extruder would.

2.2 Variable correlation and lags

All of the variables shown were correlated with the polymer's melt index. It has been shown (McAuley and MacGregor 1991) that the melt index has significant log-linear relationship with plant process variables such as temperature and pressure. The same relationship was noticed in the data reported here and therefore log-transformed variables were used in the analysis. Table 1 shows a summary of the correlation of the extruder variables with the log of the melt index.

The variable used for fitting and validation of the soft sensor was the online rheometer. It was expected that these readings would be delayed. Thus a delay estimation algorithm and cross-validation via visual inspection were used to estimate the time delays between the variables. The lags between the extruder variables and the online rheometer are also shown in Table 1.

Table 1 Extruder variables correlation and lag with log of rheometer measured melt index

| Variable | Correlation | Lag (min.) |
|----------|-------------|------------|
| PI-01 | -0.742 | 13.92 |
| PI-02 | -0.799 | 13.50 |
| PI-03 | -0.881 | 12.08 |
| PI-04 | -0.712 | 10.08 |
| PI-05 | -0.917 | 10.00 |
| PI-06 | -0.981 | 9.92 |
| TI-01 | -0.887 | 7.83 |
| TI-02 | -0.705 | 5.33 |
| TI-03 | -0.947 | 4.91 |
| SI-01 | 0.922 | 10.25 |
| II-01 | -0.863 | 10.33 |

The correlation between the variables was found using standard correlation analysis.

The data set used to calculate the correlation comprised 16340 one minute samples from fifteen (15) different grades with melt indices ranging from 1.7 to 870 gm/min.

Figure 3 shows one of the plots used to determine the time delay between the signals based on visual inspection. The most significant deviations in the data (large peaks) were used to visually confirm the time delay.

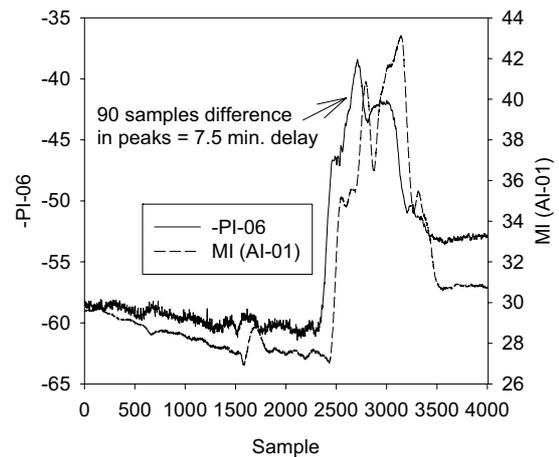


Figure 3 – Visual inspection plot for time delay

The delay estimation algorithm used was defined by (Moddemeijer 1988). It is a relatively old algorithm, it is available online and it was simple to use. The algorithm requires no a priori information about the signals; however, it assumes the signals to be stochastic and stationary. These assumptions are not fully true for the signals being considered here. As mentioned before, the extruder variables drift with time. However, over relatively short times, they can be considered stationary. This method involves splitting the two signals into a past and future vector. Then the capture of information between the concatenated past and future vectors is calculated. A function which continuously splits the data series into the past and future vectors is used to find which split gives the maximum common information. The capture of information is stored in a variable pair called the criterion. The maximum value of the criterion gives the number of sample times which correspond to the estimated time delay between the two signals.

Figure 4 shows the criterion plotted against sample intervals for two of the extruder variables (sample time = 5 seconds).

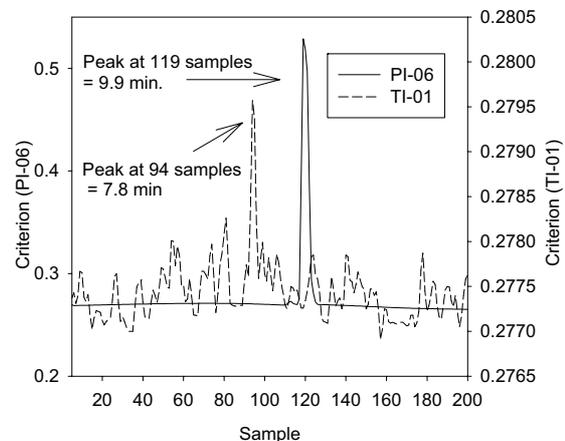


Figure 4 – Delay estimation algorithm for two extruder variables

2.3 Soft sensor models

The selection of plant variables which comprise the soft sensor was based on the correlation with the melt index, time delays and lack of disturbances. The main disturbances affecting the extruder variables are the screen pack fouling and cooler fouling. The typical disturbances caused by these are shown in Figure 5. This figure clearly shows that a screen pack change causes a significant change in the operating pressure for PI-05. A cooler cook event causes a change in the pressure and melt index, however the steady state melt index after the event is the same while the corresponding steady state pressures are not.

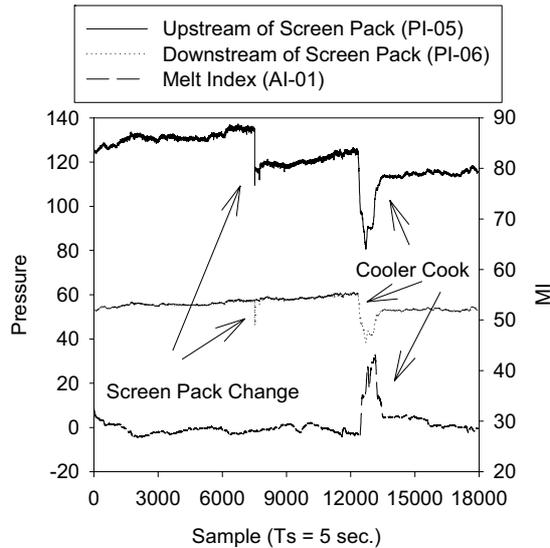


Figure 5 – Disturbances effecting extruder variables

The most important variables were chosen based on the physics and rheology of the extruder and the correlation. As described, the melt index range for products produced by this facility is very large. Initially, one simple model was developed which covered the entire product range. However, during events such as cooler cooks and grade changes, the melt index for each grade showed an exaggerated change. Thus, a speed and temperature modified model was developed.

The Basic Model. This model was built using least squares regression. It was based on Equation(1).

$$MI = f(P) \quad (1)$$

where

MI is the melt index.

P is the pressure at PI-06 (the variable with the highest correlation).

$$MI = \exp\left(a + b(P^\alpha)\right) \quad (2)$$

Equation(2) was implemented, where a , b and α were constants found using regression. This was found useful to give an idea of the relative behaviour of the melt index (increasing or decreasing and rate of change). However, the absolute value was found to contain errors; due to the speed of the extruder being controlled by the hopper level controller and the fouling of the coolers and screen pack.

S and T Compensated Model. This model was based on equation 2 but includes some more information about the physics of the extruder. Equation(3) shows the basis of the model.

$$MI_{T_r, S_r} = f\left(P_{T_r, S_r}\right) \quad (3)$$

where

MI_{T_r, S_r} is the melt index at a reference temperature,

T_r and reference speed S_r .

P_{T_r, S_r} is the pressure at a reference temperature, T_r

and reference speed S_r .

In this application the extruder can be viewed as a pseudo melt indexer. With a typical melt index instrument, melt index is measured by applying a fixed pressure to the polymer at a fixed temperature. For the melt index measurement, the polymer is forced through a die, and the weight of polymer which flows through the die in a fixed time interval is the melt index. With the extruder, the pressure applied to the polymer depends on the extruder speed and temperature. This gives the relationship shown in Equation(4).

$$MI_{T, S} = f\left(P_{T, S}\right) \quad (4)$$

where

$MI_{T, S}$ is the melt index at a operating temperature,

T and operating speed S .

$P_{T, S}$ is the pressure at a operating temperature, T

and operating speed S .

This equation can be modified to give Equation(5); which compensates for the change in temperature and speed.

$$MI_{T_r, S_r} = f\left(P_{T, S}\right) + f(T - T_r) + f(S - S_r) \quad (5)$$

Equation(5) reports a melt index similar to that measured by the rheometer.

The relationships internal to these functions are not exactly known, but based on the high correlations observed, a relationship was assumed. The relationship which included the speed compensation was of the form shown in Equation (6).

$$MI_{T, S_r} = \exp\left(a + b(P^\alpha)\right)\left(c + d(S)\right) \quad (6)$$

This was expressed in the form shown in equation(7).

$$MI_{T, S_r} = \exp\left(a + b(S) + c(SP^\beta) + d(P^\alpha)\right) \quad (7)$$

The relationship shown in equation(8) was found after the initial regression.

$$\beta = -\alpha \quad (8)$$

Thus equation(9).

$$MI_{T, S_r} = \exp\left(a + b(S) + c\left(\frac{S}{P^\alpha}\right) + d(P^\alpha)\right) \quad (9)$$

Where a, b, c, d and α are constants fit using least squares regression.

A relationship between the melt index and temperature (TI-03) was found by manipulation of the

variables and observing the correlation. The relationship found is shown in equation(10).

$$MI \propto \frac{1}{T^2} \quad (10)$$

Thus equation(9) was extended to equation(11) which included both speed and temperature compensation.

$$MI_{T,S} = \frac{\exp\left(a + b(S) + c\left(\frac{S}{P^\alpha}\right) + d(P^\alpha)\right)}{T^2} \quad (11)$$

The model with and without the temperature compensation are shown in Figure 6. It was noticed that without the temperature compensation there was a significant overshoot during the dynamic periods (such as cooler cook events and grade transitions) and the model was more susceptible to the fouling errors. The temperature compensation alleviated the majority of the overshoot and some of the offset due to fouling; this is as shown during a typical cooler cook in Figure 6.

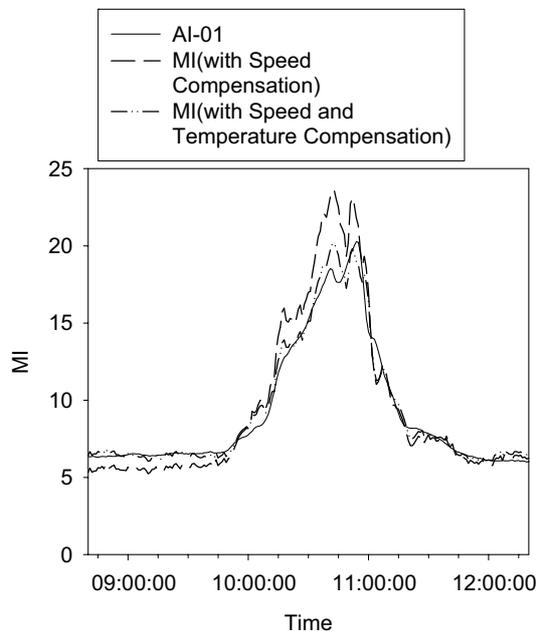


Figure 6 – Cooler Cook Event

The model was modified during the fitting process. This included lagging the independent variables to take advantage of the time delay information found previously. It was found that use of the lagged data did not give any significant gains. Upon implementation it was found using the most current data available for all independent variables gave a value for the melt index nine (9) minutes ahead of the online rheometer.

Figure 7 shows the validation data for a dynamic run. It can be seen the model captured the dynamics of the melt index change well.

Figure 8 shows the validation data for the model. It can be seen the model showed an excellent fit for the full range of product produced by the plant (these products were produced over a four month period).

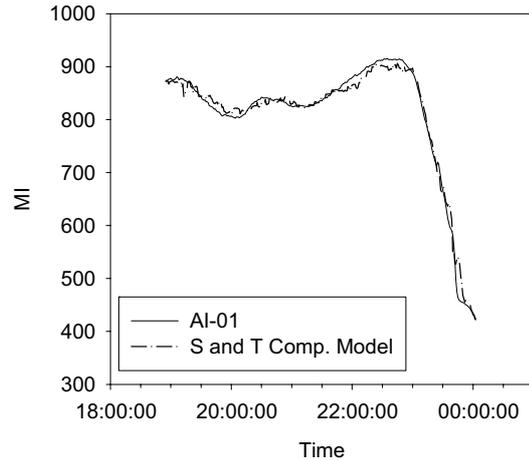


Figure 7 – S and T Compensated Model Dynamic Validation Plot

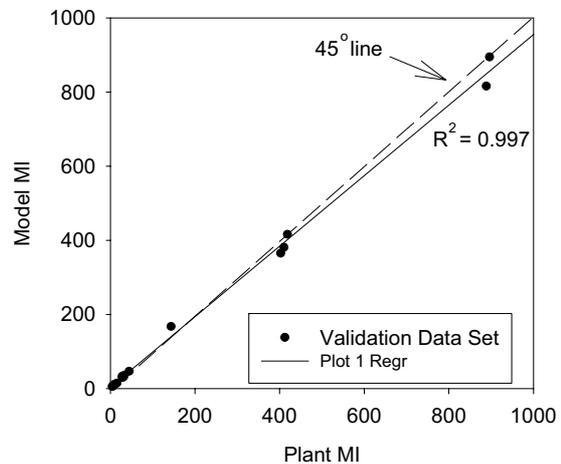


Figure 8 - S and T Compensated Model Validation Plot (several grades)

Bias Updating of the Model. The compensated model built was found to operate well for a certain period of time and at certain grades, then drifting occurred. This was attributed to the significant changes in the extruder operating conditions due to the fouling. In order to compensate for this characteristic of the process a bias updating scheme for the model was implemented. The bias was applied to the constant a in equation(11). Equation(12) shows how the new a_{Calc} was found.

$$a_{Calc} = \ln(MI_{Avg} \times T^2) - b(S) - c\left(\frac{S}{P^\alpha}\right) - d(P^\alpha) \quad (12)$$

The bias which is calculated in equation(13) was stored in the historian.

$$bias = a_{Calc} - a \quad (13)$$

Equation(14) shows how a_{Calc} was used to calculate the new melt index.

$$MI_{Bias} = \frac{\exp\left(a_{Calc} + b(S) + c\left(\frac{S}{P^\alpha}\right) + d(P^\alpha)\right)}{T^2} \quad (14)$$

Figure 9 shows the flow sheet of the procedure developed for implementing the bias updating scheme.

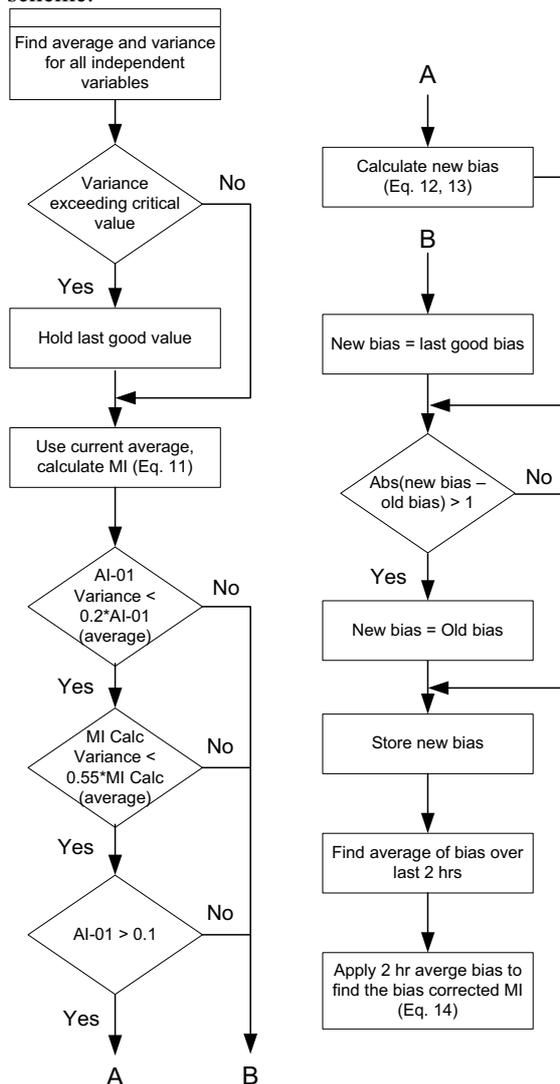


Figure 9 – Bias updating flowsheet (30 min. execution period)

3. CONCLUSION

A soft sensor model for the melt index of EVA copolymer was built and has been implemented at AT Plastics Inc (Edmonton, Canada). This soft sensor was built at different stages to handle different problems. The soft sensor used variables from the plant extruder (pressure, speed and temperature) to calculate the melt index. Biased nonlinear least squares regression was used to calculate the parameters in the soft sensor model. To compensate for drifts due to fouling, bias correction was added. It was also found that it would require some time for the parameters in the bias to update after the plant had gone through a grade transition or a cooler cook. The main goal of this soft sensor was to have a reliable reading for the melt index during the grade transitions to build a model of the process. The compensated model with bias updates fits this application well. The next step will be to use this soft sensor to model the plant and produce optimal grade

transitions and compare them with those produced by a first principles model.

4. ACKNOWLEDGEMENTS

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