MODELING FOR CONTROL OF REACTIVE EXTRUSION PROCESSES

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Abstract: A modeling and control framework for effective control of end-use product characteristics of reactive extrusion processes is proposed. We discuss for an example process the development of two important components of the modeling scheme: an identified model that relates manipulated inputs to process outputs and a first principles process model that relates the inputs to quality variables.

Keywords: multirate, multivariable system, nonlinearity, modeling, model-based control, identification

1. INTRODUCTION

Reactive extrusion processes have assumed significance in the polymer processing industry due to their wide-ranging applications in manufacturing neat polymers, polymer blends and, more recently, nanocomposites. The ever-tightening customer demands on product specs have necessitated comprehensive dynamics and control studies of these processes, which, until now, have mostly focused on the control of a single variable such as viscosity (Broadhead *et al.*, 1996).

The specifications are usually given for product properties such as tensile strength, melt index etc. that are rarely measured online and are obtained using measurements having variable time requirements (multirate measurements). For the purposes of effective control, it is necessary to separate these properties into product quality variables '*q*' (Melt index, viscosity, density, etc) and end-use physical characteristics '*w*' (Toughness, UV/chemical resistance, etc.). The process output variables '*y*' (Die pressure, melt temperature etc.) are measured online at a much faster rate than the product properties. In addition to the multirate nature of the system, effective control of the product properties is made difficult by the complex process mechanisms that result from the interactions between fluid mechanics, heat transfer, reaction kinetics and the extruder geometry. The ultimate objective of this work is to develop a framework for controlling product properties and assuring acceptable end use performance.

1.1 Modeling Scheme

The approach adopted for this challenging problem begins with an adequate mathematical representation of the relationships between variables across the entire processing chain. Such a representation will serve two crucial purposes: (i) provide estimates of the infrequently measured product properties at a much faster rate, and (ii) facilitate the development of a control system to meet the above mentioned objective.

Fig. 1 shows a schematic representation of the proposed modeling scheme, which consists of the following models: i) M_{uy} – a model relating the manipulated variables, u, to process output variables, y, (ii) M_{uq} – a process model relating the manipulated variables, u, to the internal product quality variables, $\hat{q}$, (iii) M_{qq} – a model relating internal quality variables, $\hat{q}$, to product quality variables, q,

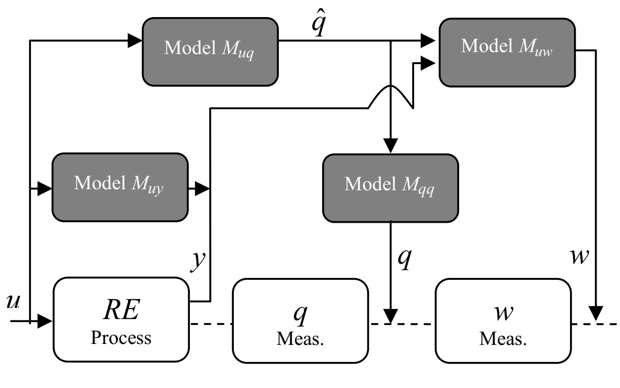

Fig. 1. Proposed modeling scheme

(iv) M_{qw} – relating internal quality variables, $\hat{q}$, to end use characteristics, and (v) M_{wz} (*not shown in Fig. 1*) – relating end-use physical characteristics, w, to product performance in end-use, z_w, a binary variable that represents acceptable performance as 1, and unacceptable performance as 0.

A modeling scheme that relates the different classes of variables sequentially, although more intuitive, is impractical in this case. This is because it is usually difficult to model the relationships between the process outputs and the measured product quality variables, since these variables based on the measurements, which are selected on practical grounds such as the availability of sensor locations on the extruder, and developing a mathematical relationship between these two classes of variables is not straightforward. To overcome this problem, an additional class of variables, called internal product quality variables '$\hat{q}$' (Composition, weight average molecular weight) that constitutes an indirect way to link 'y' with 'q', is introduced in the proposed modeling scheme.

1.2 Control Scheme

The control paradigm is predicated upon using the above network of models for two important tasks: (i) to translate the customer requirements on end-use performance to set points for process variables, and (ii) to make appropriate modifications (that is, to take control action) wherever appropriate along the manufacturing chain based on all available information. For this purpose a multivariable cascade control scheme (Fig. 2) is proposed, consisting of a fast model-based controller *C1* for the inner loop between the manipulated variables and the output variables and a slower (model based) controller *C2*, which will translate the end use performance objectives to set points for the output variables. In addition to these loops, there exists an innermost basic regulatory control loop, which ensures that the set point changes in the manipulated variables are efficiently tracked.

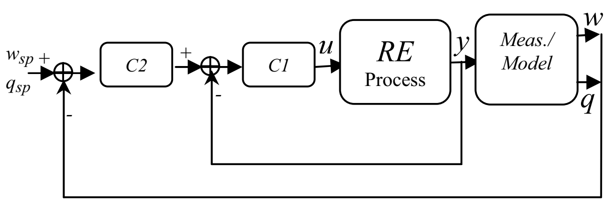

Fig. 2. Proposed control scheme. *C1* and *C2* are the inner loop and outer loop controllers respectively.

The objective of this paper is to discuss the development of two important components of the proposed modeling scheme: (i) model M_{uy} identified from input/output data, and (ii) model M_{uq} developed using first principles. The example system consists of the reaction of a functionalized ethylene co-polymer, "Elvaloy®" (Ethylene/n-Butyl Acrylate/Glycidal Methacrylate Terpolymer (E/BA/GMA)) with an acid co-polymer "Nucrel®" (Ethylene/Methacrylic Acid Copolymer (E/MAA)) in a Coperion W&P ZSK-30mm co-rotating, intermeshing twin screw extruder.

2. MODEL M_{uy}

Model M_{uy} represents the mathematical relationship between the manipulated inputs and the outputs. Due to the complexity of the interacting process mechanisms, a first-principles model is impractical for control at this level. A model identified from carefully obtained input/output data is a more practical alternative. A systematic procedure was employed to carry out the three major steps of system identification of this class of processes: (i) experimental test design and collection of input/output data, (ii) model structure and order selection and (iii) model validation.

2.1 Inputs and Outputs

The manipulated inputs (u) are the screw speed, E/MAA feed-rate, E/BA/GMA feed-rate and the barrel temperatures for the seven extruder zones. The changes in all the inputs are implemented manually. However, the barrel temperature regulatory controller loop dynamics are much slower than the process dynamics excluding the inner regulatory loops. Therefore, with the exception of a step change, any other dynamic change in the barrel temperature is impractical. The inner loops for other manipulated inputs are fast compared to the process; therefore, comparatively rapid changes can be implemented in these variables. The process outputs are E/MAA weight fraction in the melting zone, die pressure, exit melt temperature, and motor power. With the exception of the E/MAA weight fraction, which can be easily obtained from the feed-rates of the two polymers, all other outputs are measured.

2.2 Identification Tests

There are two components to the experimental test design: (i) preliminary tests (ii) final identification test. The preliminary tests were aimed at obtaining *à priori* knowledge needed for the design of the final identification test. These tests consisted of a series of step changes as well as simultaneous staircase changes in the manipulated inputs at two operating points: (A) a low melting-zone composition of E/BA/GMA (~ 1%) inducing a low extent of reaction and a relatively small change in the product viscosity as compared to the viscosity of the pure E/MAA feed, and (B) a high melting-zone composition of E/BA/GMA (~ 4 %) inducing a high extent of reaction and a significant change in the product viscosity. This selection of the operating points enabled study of the effect of the reaction on the process dynamics behavior. The key results of the preliminary test were: (i) the reaction has a strong nonlinear effect on the process dynamics, (ii) the system is ill-conditioned at the operating point A, and (iii) the process exhibits an approximately linear behavior in the vicinity of the two operating points.

Based on these results, the final identification test, aimed at developing a linear model at each operating point, was designed using generalized binary noise (GBN) signals (Tullenken, 1990). As proposed by Zhu (2001), GBN signals with a mean switching time equal to 1/3rd of the process settling time (~360 s) were used in the *open loop* identification experiments. In recognition of unmeasured disturbances and the high measurement noise levels (low signal to noise ratio), the duration of the test was approximately 15 times the process settling time. Three uncorrelated GBN signals were administered simultaneously in the screw speed (u_1), E/MAA feed-rate (u_2), and E/BA/GMA feed-rate (u_3) at each operating point (*Fig. 3*). These uncorrelated signals are suitable for the identification of a well-conditioned process as well as identifying the high gain direction of an ill-conditioned process.

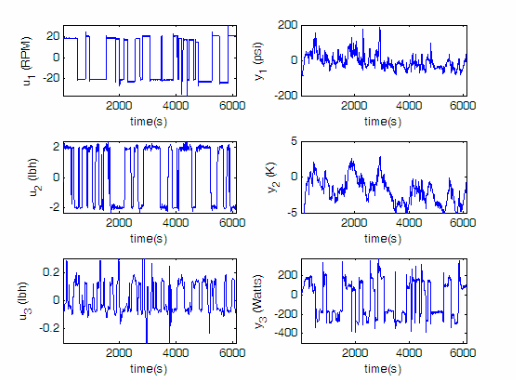

Fig. 3. GBN test data at operating point B: Inputs, left panels, outputs right panel

An additional test, based on the open loop design suggested by Zhu (2001), was used to identify the low gain direction of the process at the operating point A. The GBN signals, having correlated high amplitude periods combined with uncorrelated low amplitude periods, were administered to the inputs u_1 and u_2, while the input u_3 was unchanged.

2.3. Model Order and Structure

The purpose of model structure and order selection was to obtain a suitable linear model around each operating point. The candidate models structures included: (i) Multi Input Single Output (MISO) structure having a common model for each output, and (ii) Multi Input Multi Output (MIMO) structure having a common model for all outputs. The suitability of these model structures for representing the input/output data was tested for different parametric models (for e.g., Auto regressive Moving Average with eXogenous inputs [ARMAX], Box Jenkins [BJ] etc.). The parameter estimation was performed using the System Identification Toolbox of Matlab®. Out of these models, MISO Box Jenkins (Eq .1) model was found suitable for describing the dynamics in the input/output data.

The criteria used for selecting the model orders were: (i) percentage of output variation that is captured by the model, (ii) Akaike's final prediction error, and (iii) pole-zero diagrams to check for over parameterization. Table 1 presents the selected model orders for the MISO BJ model corresponding to each output.

Table 1 Identified BJ models at the two operating points

O/I	*MISO Model Orders* u_1 (n_a,n_b)	u_2 (n_a,n_b)	u_3 (n_a,n_b)	Noise (n_c,n_d)	*Delay* (n_{d1}, n_{d2}) u_1	u_2	u_3
			Operating Point A				
y_1	5,5	5,5	5,5	2,2	0,0	0,0	0,0
y_2	5,5	5,5	5,5	1,1	0,0	0,0	0,0
y_3	3,3	3,3	3,3	0,0	0,0	0,0	0,0
y_4	2,2	2,2	2,2	1,1	0,0	0,0	0,0
			Operating Point B				
y_1	5,5	5,5	5,5	3,3	1,1	1,1	1,1
y_2	4,4	4,4	4,4	1,1	0,0	0,0	0,0
y_3	3,3	3,3	3,3	1,1	0,0	0,0	0,0
y_4	2,2	2,2	2,2	1,1	0,0	0,0	0,0

$$y(t) = \frac{B(q)}{A(q)} u(t - n_{d1}) + \frac{C(q)}{D(q)} e(t - n_{d2}) \qquad (1)$$

where,
$$A(q) = 1 + a_1 q^{-1} + \ldots + a_n q^{-n_a}$$
$$B(q) = \quad b_1 q^{-1} + \ldots + b_n q^{-n_b}$$
$$C(q) = 1 + c_1 q^{-1} + \ldots + c_n q^{-n_c}$$
$$D(q) = 1 + d_1 q^{-1} + \ldots + d_n q^{-n_d}$$
$$and, \quad q^{-k} v(k) = v(t-k)$$

The overall model structure for M_{uy} is thus a collection of local linear models that are valid in each region of the input/output variable space defined by the E/BA/GMA weight fraction.

The validation data consists of a fraction of the final test(s) data that was not used for estimation, in addition to the preliminary test data. (Fig. 4).

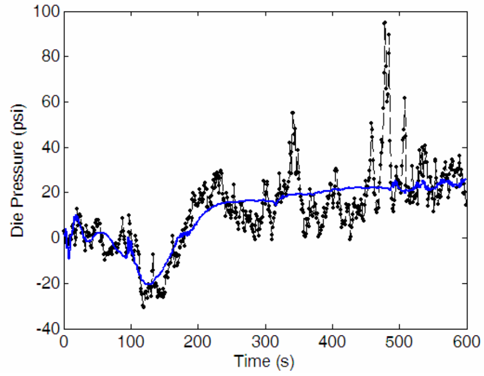

Fig. 4. Measured die pressure (dots) and the identified model predictions (solid line) for a step change in E/MAA feed-rate.

3. MODEL M_{uq}

Model M_{uq} represents the mathematical relationship between the manipulated inputs, u, and the internal quality variables, $\hat{q}$, a class of *unmeasured* variables motivated by the modeling scheme (Fig. 1). Therefore, an empirical model is not appropriate for this purpose; first-principles modeling, an alternative to the empirical models, is, therefore, an obvious choice. For validation, however, alternative quality variable measurements are essential. In this work, online viscosity measurements are used for validation.

3.1. Experimental Investigation to Obtain Fundamental Process Information

The development of such a first principles model, M_{uq}, is facilitated if the information regarding the key process mechanisms that influence the process dynamic behavior is available. The key process mechanisms may differ for dissimilar reaction systems and commonly made assumptions in the modeling of reactive extrusion processes, such as Newtonian fluid flow and isothermal conditions, may be inappropriate. Specifically, it is important to know the degree of interaction between the reaction kinetics, melt flow in the complex extruder geometry, heat transfer, and the melting of the polymers that must be captured in the first principles model. Since this information is not always available in the literature, an experimental process investigation becomes essential.

Three techniques were employed for the experimental investigation of the example process: (i) Pulse Technique: to probe the melting dynamics, (ii) Residence Time Distribution (RTD) experiments to probe the coupling between the reaction kinetics and fluid flow, and (iii) Step change experiments to study the transient process behavior. A summary of the main results of this investigation is presented below (for more details, see Garge *et. al*, 2005):

- Very little reaction occurs during the melting of the polymers.
- The melting zone location is weakly influenced by the operating conditions.
- The reaction rate is strongly influenced by the E/BA/GMA concentrations.
- The viscosity change due to the reaction does not significantly influence the residence time distribution, and in turn the flow of the polymer melt.
- The interaction between the heat transfer and the reaction kinetics is weak at the low E/BA/GMA concentration, but strong at the high E/BA/GMA concentration. Similarly, the dependence of the reaction on the flow is weak at the low E/BA/GMA concentration, while it appears to be somewhat stronger at high E/BA/GMA concentrations.

3.2. Model Components

As noted earlier, the model M_{uq} is needed for mathematically describing the relationships between the manipulated inputs, u, and the internal product quality variables, $\hat{q}$, such as the product composition, which are directly dependent on the reaction. Obviously, the process mechanisms that are related to or that affect the reaction need to be modeled appropriately, whereas the modeling of other mechanisms is not critical. The key aspects of the strategy for developing a transient process model are:

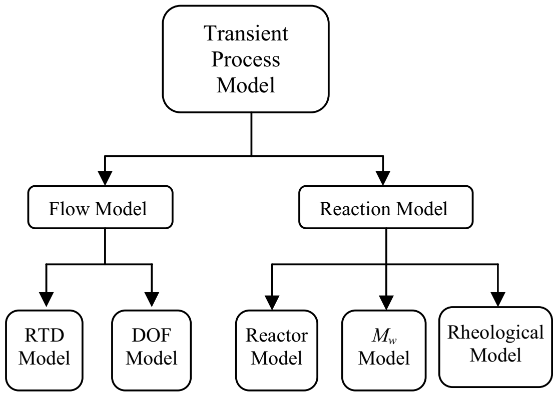

Fig. 5. Components of the transient process model

1] The experimental results suggesting that the melting does not significantly influence the reaction indicate that a simple melting model will suffice to predict the axial location where the melting process is almost complete. The location of the melting zone, however, is almost invariant for different operating conditions. Instead of developing a detailed melting model, it is proposed to demarcate the melting zone extent empirically for each operating condition and use this melting zone extent for all the other operating conditions.

2] The experimental results suggest that the flow of the polymer melt is weakly dependent on the reaction in the extruder and vice versa. Taking advantage of this fact a divided model structure consisting of distinct flow and reaction models is proposed, as illustrated in Fig. 5. Such a structure allows for the development of a simple flow model and a relatively complex reaction model.

3] The reaction kinetics are influenced by the heat transfer, at least at the high E/BA/GMA operating point (B). Therefore, these two mechanisms are coupled in the reaction model.

The details of the reaction model as well as the flow model are presented elsewhere (see Garge *et. al*, 2005). The salient features of the two are discussed below.

Flow Model*:* The flow model is split into two parts: (i) a RTD model and (ii) a 'Degree of Fill' (DOF) Model. Such a distinction, although not essential for developing a transient flow model, considerably simplifies the coupling between the flow and reaction models. The DOF model demarcates the *partially filled* and the *fully filled* regions. The RTD model predicts the mean residence time and time delay that is used, along with the degree of fill profile obtained from the DOF model, to calculate the average axial velocity in different regions of the extruder.

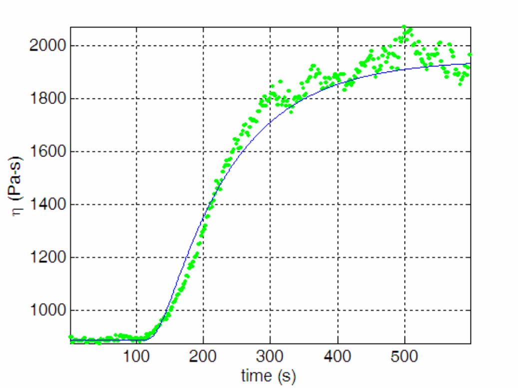

Fig. 6. Measured viscosity (dots) and the transient process model predictions (solid line) for a step change in E/BA/GMA feed-rate.

Reaction Model: The main objective of the reaction model is to quantify the effect of the reaction on the quality variables, **q**, specifically on the product composition, viscosity and the average molecular weight ($\overline{M_w}$). It can be divided into three components: (i) a reactor model to describe how the reaction occurs in the extruder. The one-dimensional axial dispersion model was found suitable for the example system. Non-isothermal terms were included to incorporate heat transfer effects (Eq. 2). (ii) a $\overline{M_w}$ model to represent the effect of the reaction on the average molecular weight, and (iii) a rheological model to represent the effect of the reaction on the viscosity.

Concentration for the species i (C_i):

$$\frac{\partial C_i}{\partial t} + \frac{\partial (v_x C_i)}{\partial x} = \frac{\partial}{\partial x}\left[D_L \frac{\partial C_i}{\partial x}\right] - R_{C_i} \qquad (2a)$$

Temperature (T):

$$\rho C_p \left[\frac{\partial T}{\partial t} + \frac{\partial (v_x T)}{\partial x}\right] = \frac{\partial}{\partial x}\left[\lambda \frac{\partial T}{\partial x}\right] + Q \qquad (2b)$$

In the above equations, v_x stands for average axial velocity, D_L is axial dispersion coefficient in the zone, R_{ci} is the reaction rate, ρ is the mean density of the polymer melt, C_p is its average specific heat, and Q represents the heat generation terms. The transient model, obtained by coupling the reaction and flow models, was validated with the viscosity measurements for step changes in the inputs, u (Fig. 6).

3. SUMMARY AND CONCLUSIONS

A modeling and control framework has been proposed for effective control of the product end-use properties of a reactive extrusion process. The modeling framework consists of a network of models that mathematically represent the relationships between the different classes of variables across the manufacturing chain. Due to practical reasons, the introduction of an additional class of unmeasured variables, called internal quality variables, is essential for this modeling scheme. The proposed multivariable cascade control scheme will then use this network of models, in addition to the all the available measurements, to guarantee acceptable end use performance.

The paper briefly describes two important components of the modeling scheme: (i) M_{uy} – a model relating the manipulated variables, u, to process output variables, y; (ii) M_{uq} – a process model relating the manipulated variables, u, to the internal product quality variables, $\hat{q}$. Based mainly upon the anticipated computational effort and applicability, it is suggested to use an empirical model obtained from carefully obtained input/output data for M_{uy}, and a first-principles model for M_{uq}. The development of these models was illustrated for an example process consisting of a reaction between two functionalized polymers in a co-rotating twin-screw extruder.

A systematic experimental approach for obtaining input/output data, consisting of a pre-test and a final test based on GBN signals, was employed for the identification of the model M_{uy}; a multi-model structure, consisting of a MISO BJ model for each output, was found suitable in this case. The first principles model, M_{uq}, was based on an experimental investigation carried out to probe the important physical mechanisms of the system. A divided model structure, consisting of a reaction model and a flow model, was developed and validated against the viscosity data for transient step changes in the manipulated inputs.

Our current effort is focussed on developing the models M_{qw} and M_{wz} in the modeling scheme of Fig. 1. The melting process strongly influences the product morphology, and thus plays an important role in determining some of the end-use product properties such as tensile strength. It is, therefore, important to incorporate this "melting effect" in the model M_{qw} that relates the quality variables to the end-use properties. For this purpose, we propose to use a two-stage procedure consisting of: (i) developing an empirical model that will provide system parameters related to the melting process, and (ii) quantitatively relating these parameters, along with appropriate quality variables, to the end-use properties (Garge *et. al*, 2006).

The primary purpose of the model M_{wz} is to relate customer feedback to the end-use properties, with customer feedback as a binary variable, z_w, that is '1' for acceptable product performance and '0' for unacceptable performance. Based on this representation, we propose to use a *binary logistic regression model* for representing the relationships between z_w and the end-use properties.

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