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Quality control of polymer production processes

Masahiro Ohshima*, Masataka Tanigaki

Material Processing Engineering Laboratory, Department of Chemical Engineering, Kyoto University, Yoshida Sakyo-ku, Kyoto 606-8501, Japan

Abstract

For the polymer production industries, the competitive edge will come from the technology that excels in controlling the polymer properties in a consistent way over the entire plant and in maximizing the production performance while keeping safety regulations. Based on the experience in applying advanced process control and scheduling schemes to industrial polyolefin polymerization plants, the state of the art in quality control systems for providing the polymer production plant with an enlarged capacity for product discrimination and flexibility is reviewed. On-line soft-sensing and optimal grade changeover control problems are the main focus of this paper. A quality control system for polymer production plants, which integrates optimal control with on-line sensing and scheduling techniques, is discussed making reference to an application of a prototype system to an industrial plant. © 2000 IFAC. Published by Elsevier Science Ltd. All rights reserved.

Keywords: Polymer production plant; Quality control; On-line soft sensor; Grade changeover operation; Blending operation

1. Introduction

Use of polymers has been growing steadily in many industrial fields, such as automobile, food, apparel, electronics, etc. In the US, a fivefold growth of plastics in two decades (1974–1994) was reported [1]. It was attained by exploring new and various plastic applications. Polypropylene (PP) is now used for almost all automobiles. Soft drink bottles made of PET (polyethylene terephthalate) have almost completely superseded glass bottles, and PE (polyethylene) plastic bags have replaced the paper bags at grocery stores.

Consumers are learning that many plastic products are made from the same polymer. For example, PP used for a core material of instrumentation panel of cars is also used for car batteries, indoor—outdoor carpeting, and polyolefin intimate apparel. For each use, specific properties of the polymer are needed. In order to meet these demands, the polymer industries are producing many different grades of high quality polymers.

In recent years, the pressure from customers for greater grade variation and product diversification has been growing while specification of polymer quality becomes increasingly severe. For the future polymer

E-mail address: oshima@cheme.kyoto-.ac.jp (M. Ohshima).

industries, the competitive edge will come from a technology that excels in tailoring polymer properties and in controlling production plants toward maximizing product quality as well as production performance and safety.

In this paper, the state of the art in quality control systems for polymer production processes is discussed, based on the experience of joint university—industry projects in applying advanced process control and scheduling schemes to industrial polyolefin polymerization plants.

A significant amount of research has been done in the area of control, monitoring, and modeling of polymerization reactors, and excellent reviews have also been given by several researchers [2–6]. Because of the authors' bias and space limitation, this paper could not cover every important issue addressed in the past. Online soft sensing and optimal grade changeover control are mainly focused on this paper.

One of the issues discussed throughout this paper is plant-wide total quality control with design of molecular structure at microscale to macroscale levels: The polymer production plant does not consist only of reactor(s). It consists of reactor(s), separator(s), dryer(s), extruder(s) and blending tank(s). Therefore, quality of the polymer is affected not only by reactors' operation conditions but also by extruding and blending operations. In addition, polymer properties are determined by

^{*} Corresponding author. Tel.: +81-75-753-5586; fax: +81-75-753-5588

low-order and high-order molecular structures of the polymer. Thus, to provide the production plant with an enlarged capacity for product discrimination and flexibility, the polymer properties should be controlled on each molecular structure level in a consistent way throughout processing history — from reactors to separation units, extruders, and to blending tanks. A scheme of quality control system for polymer production plants, which integrates optimal control with online sensing and scheduling techniques, is discussed making reference to an application of a prototype system to an industrial plant.

2. Polymer production plant

A highly simplified schematic diagram of a polymer production plant is illustrated in Fig. 1. In general, polymer production plants consist of a train of reactors in series, separation processes, extruders, and blending tanks. As Ogunnaike [7] described, neither a recycle loop of unreacted monomer nor blenders is required, making the operation very simple if environmental regulations, production cost and product quality are not considered. But, in reality, a train of reactors is used to produce layered products, and the separation units are installed to recycle the unreacted monomer in an attempt to meet various environmental regulations and to reduce the production cost. The blenders are also required to damp out product quality variability. Furthermore, to meet grade variation and product diversification without increasing equipment cost, it is not unusual that a plant produces as many as 10 grades or even more by changing its operating conditions. As a result, the plant becomes increasingly complex and the operation increasingly difficult.

Basic regulatory control of key process variables, such as temperature and flowrates is imperative, but this alone is not sufficient to produce various grades of high-quality polymer products at an economically attractive cost.

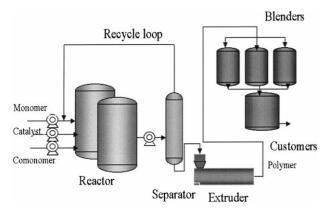


Fig. 1. Simplified schematic of polymer plant.

An overall view of a prospective control system for a polyolefin plant is illustrated in Fig. 2, soon after the specification of the polymer is given by the customers, polymer quality that the individual processing unit produces should be specified appropriately. Operating conditions are then determined for each unit to meet the specified quality. Taking plant stability into account, a production schedule is determined so as to meet the demand while keeping the inventory cost low. Following the determined schedule, safe and quick changeover operations are performed by a sophisticated control system.

3. Needs for quality modeling

Polymerization reaction engineering contributes greatly to modeling and understanding how the reactor operating conditions affect molecular properties, such as molecular weight distribution, composition distribution, and degree of chain branching, etc. [6]. Rheology plays an important role in bridging a gap between molecular properties and rheological properties, which are close to end-user quality.

Table 1 describes qualitative relationships between molecular properties and polymer properties as well as processability of the polymer [8]. As illustrated in Table 1 and Fig. 3, end-user properties depend strongly on the polymer low-order and high-order molecular structures as well as their distributed nature of variables (molecular weight distribution, etc.). For example, hardness of the polymer depends on polymer crystallinity that is determined by stereoregularity of polymer: a linear polymer with small side-groups is highly crystalline; a linear polymer with bulky but regular side-groups has low crystallinity; and a linear polymer with bulky and random side-groups is non-crystalline. Similarly, the morphological form of a HIPP polymer is often a key variable of end-user properties, and it depends on particle size distribution, polymer composition distribution and processing history in the extruder.

To determine the operating condition for each individual processing unit, it is indispensable to have deeper understandings about how the end user properties are affected by molecular structure, its distribution, and operating conditions of each processing units. The quantitative representation of the relationships is called the quality model. To construct a quality control system for polymerization plants, it is crucial to develop such quality models which can describe the relationships among end-user properties, polymer properties, their molecular structures, and processing history. Several attempts have been made to develop such models. However, there are many relationships yet to be described quantitatively.

Production Planning, Target Value **Optimal** Polymerization **Product Quality Product Blending** Changeover **Rate Control** Control **Planning** Operation Setpoint On-line Concentration Ratio Reaction Temp. Product property Control Control **Blending & Storing** Sensor Pressure **Materials Feed Rate** scheduling Control Control Process Reactor Level Polymerization Reactor Control Monitoring **Control System** Catalyst Polymerization Blending Separation | Extruder Processing Reactor Storing

Advanced Control System in Polyolefin Plant

Fig. 2. A structure of prospective control system.

Table 1
Relationship between molecular structure and properties of polyethylene [8]

	Molecular structure	Molecular weight $(M_{\rm w})$	Molecular weight distribution	Branching chemicals	Degree of branching	Degree of branching distribution	Long-chain branching (LCB)
Mechanical and chemical property	Transparency	О	0	0	0	О	
	Tensile strength	o	O	o	o	O	o
	Impact strength	o	O	o	o	O	o
	Rigidity				o	О	
	Heat resistance				o	О	
	Cold resistance	o	0	o	o	О	
	Chemical resistance	0	0	o	o	О	o
	Heat seal	o	o	o	o	o	
Processability	Bubble stability	o	o				o
	Draw-down	o	o				o
	Extrusion torque	o	0				o

4. On-line soft sensing

4.1. Basic structure of inferential system

Polymer properties, such as Melt Index and density, are difficult to measure on-line. At best they are available only infrequently. On the other hand, many other process variables, such as pressure, temperature, flowrates etc. are easily and frequently measured. Even though on-line hardware sensors have been advanced [9,10], the use of new sensors has largely been limited to laboratory reactors. Lack of on-line sensors for key polymer properties makes the quality control of polymer plant more difficult. It is, therefore, of interest to use the process variables to infer the critical polymer

properties. Several state estimation methods and software sensors have been proposed in the area of polymerization reactor control.

Table 2 shows a collection of papers published in the last 5 years on estimation and inference of polymer properties. For a survey of the papers published before 1994 refer to Embirucu et al. [11]. Table 2 is prepared by following their description. As evident from several papers listed in Table 2, the development of an on-line inferential system for polymer property is a very active research area of polymerization reactor control.

A schematic of inferential systems is illustrated in Fig. 4. Similar structures have been proposed by several researchers [12–14]. An overview of the inferential system for chemical processes was recently given by Doyle [14].

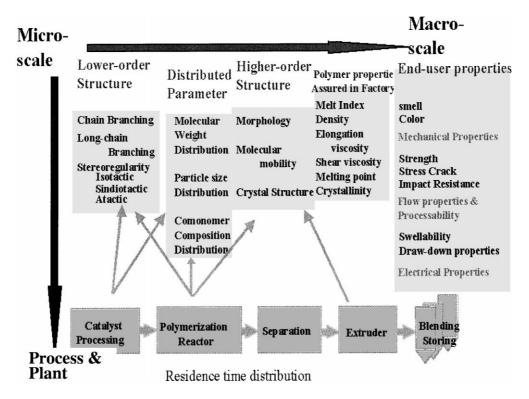


Fig. 3. Relationships among polymer properties and processing condition.

In the inferential system for polymer processes, a quality model, which can infer the polymer quality (Molecular weight, Conversion, Melt Index, Density, etc) from process variables such as temperature and concentrations in the reactor, is utilized together with a filtering scheme in order to estimate the quality on-line. When a new measurement of polymer quality becomes available off-line from the quality inspection laboratory, model adaptation is performed on the basis of the difference between the measurement and the predicted value given by the model. After each update, prediction of the quality is made by successive evaluation of the model with process variables measured on-line until the next measurement is available from the laboratory. Control actions are determined on the basis of the predicted values.

These inferential techniques are strongly dependent on quality models which can describe the relationship between polymer quality and secondary (correlated) variables. As listed in Table 2, the models used in inferential systems can be roughly categorized into three groups: (i) phenomenological models (i.e. mechanistic model) derived from first principles [15–20] (ii) empirical models derived from laboratory data and (iii) black box models obtained from operational data by applying neural nets [21,22] and/or statistical methods, such as multivariate and PLS analyses [23,24]. In the inference system for controlling polymer quality, the first kind of

models are often used together with the second and the third kind of models [25–33]. The following section will illustrate some examples of the models that the author experienced using at the polyethylene manufacturing plants.

4.2. Mechanistic vs empirical vs neural net models

McAuley and MacGregor [13] developed an inferential system for on-line estimation of Melt Index (MI) and density of Low Density Polyethylene (LLDPE) produced by a UNIPOL fluidized-bed reactor. Two models were used in their inferential system: (1) an instantaneous polymer property model which describes the relationship between process variables and the current polymer property; and (2) the cumulative property model which describes the relationship between the instantaneous and cumulative polymer properties in the reactor. These models were derived with the assumption that: polymerization reaction occurs very quickly compared with the residence time of the polymer in the reactor; all the polymer generated in a short period is similar in structure; the structure of the polymer is determined by the process operating condition of the moment. Their instantaneous MI model was given by Eq. (1), which was derived by incorporating the reaction kinetics models with the empirical relationship between MI and average molecular weight:

Table 2 A collection of papers on property estimation 1994–1997

Paper no.	MON^a	Kinetics ^b	Reac-T ^c	Reac-M ^d	Researche	Work	Poly-Tf	FMV^g	Estimate ^h	$IMV^{i} \\$	Tech ^j	$Model^k$
[15]	STY	FR	BATC	EML	A	SIM	НОМ	RC	PS	PS	EKF	ME
[16]	MMA	FR	BATC	SOL	A	EXP	HOM	$X/T/\rho$	T/RC/HT	$M_{ m w}/X$	EKF	ME
[17]			CSTR	SOL	IND	EXP	TER	RC	$PR/RC/M_{ m w}$	$PR/C/\eta$	EKF	ME
[18]	MMA	FR	CSTR	SOL	A	SIM	HOM	T/RC	$M_{ m w}/RC/T/X$	MWD	EKF	ME
[19]	aOlefin	ZN	FB	GAS	A	SIM	CO	$\eta/T/RC$	$M_{ m w}/C/ ho$	ρ	OB	ME
[20]	MMA	FR	CSTR	SOL	A	SIM	HOM	$T/\rho/X$	$T/X/M_{ m w}$		EKF	ME
[21]	MMA	FR	BATC	SOL	A/IND	EXP	HOM	T	PR		NN	BB
[22]	E	FR	TUB	HighP	A/IND	EXP	CO	T/P/FR	$\rho/M_{ m w}/MI/X$		NN	BB
[23]	E	FR	TUB	HighP	A	SIM		T/FR/P	$M_{\rm w}/LCB/SCB/X$		PLS	BB
[24]			SemiBA	C	IND	EXP		35PV	., , ,		PLS	BB
[25]	E	FR	TUB	HighP	A/IND	SIM		T	$T/M_{ m w}/MI$		NPE	ME + EC
[26]	PET	COND	BATC	BULK	A/IND	EXP		TO	$MT/RC/\eta$		EKF/OB	ME + EC
[27]	MMA	FR	CSTR	SOL	A	EXP	HOM	h/r/T	$M_{ m w}/X$		EKF	ME + EC
[28]	MMA	FR	BATC	SOL	A	EXP	HOM	RC/X	CL	CL	EKF+NN	ME + BB
[29]	E/B	ZN	CSTR	SOL	A/IND	EXP	CO	RC/T	MI	MI	NN	ME + BB
[30]	S/BR	FR	SemiBA	EML	A	SIM	CO	T	$M_{ m w}/CLD$	PS/RC	EKF/PLS	ME + BB
[31]	E/*	ZN	CSTR	SOL	IND	EXP	CO	T/RC/X	MI/ρ	MI/ρ	LE	
[32]	E/B	ZN	CSTR	SOL	IND	EXP	CO	T/RC	MI	MI	EKF	ME + EC
[33]	E	ZN	CSTR	SOL	IND	EXP	CO	RC	MI/ ho		EKF	ME + EC

- ^a MON (monomer): ethylene (E), butene-1 (B), methyl mehtarcylate (MMA), styrene (STY).
- b Kinetics: Ziegler–Natta (ZN), free radical (FR), condensation (COND).
- ^c Reac-T: continuous stirred tank (CSTR), batch (BATC), semibatch (SemiBA), tubular (TUB), fluidized bed (FB).
- d Reac-M: solution (SOL), gas-phase (GAS), bulk (BULK), emulsion (EML), high pressure (HighP).
- e Research: academia (A), industry (IND).
- ^f Poly-T: homo-polymerization (HOM), co-polymerization (CO), ter-polymerization (TER).
- ^g FMV (frequently measured variable): temperature (T), concentration (RC) conversion (X) density (ρ), viscosity (η), torque (TO).

(1)

- ^h Estimate: production rate (PR), chain length (CL), heat transfer coeff (HT), melt index (MI), particle size (PS), molecular weight (M_w) molecular weight distribution (M_wD) , long chain branching (LCB), short chain branching (SCB), density (ρ) .
 - i IMV, infrequently measured variable.
- ^j Tech: linear estimator (LE), observer (OB), Extended Kalman filter (EKF), nonlinear parameter estimation (NPE), neural net (NN), partial least square (PLS).
 - ^k Model: mechanistic model (ME), empirical correlation model (EC), black box model (BB).

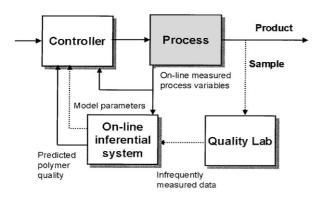


Fig. 4. On-line inferential system for a polymer.

$$\ln(MI_i) = 3.5 \ln \left\{ k_0 + k_1 \frac{[H_2]}{[C_2]} + k_2 \frac{[C_3]}{[C_2]} + k_3 \frac{[C_4]}{[C_2]} + k_4 \frac{[R]}{[C_2]} \right\}$$
$$+ k_5 \left(\frac{1}{T} - \frac{1}{T_0} \right)$$

The cumulative property model was given by

$$\frac{\mathrm{d}(MI_c(t)^{-0.286})}{\mathrm{d}t} = \frac{1}{\tau(t)}MI_i(t)^{-0.286} - \frac{1}{\tau(t)}MI_c(t)^{-0.286}$$
 (2)

where MI_c and MI_i denotes MI of cumulative and instantaneous polymers, respectively. ln(.) means natural logarithm. The value, -0.286, comes from the empirical relationship between MI and average molecular weight, i.e. $\bar{M}_w \propto MI^{-0.286}$.

In polyolefin production plants, it is well known that the concentration ratio of hydrogen to monomer strongly affects the *MI*. Likewise, the ratio of comonomer to monomer deeply affects the density of the polymer. Plant engineers have been acquiring steady state plant data and developing regression models that relate *MI* to the concentration ratios in the form:

$$\log(MI) = \alpha_1 \log \left(\frac{[H_2]}{[C_2]}\right) + \beta_1, \log(MI) = \alpha_2 \log \left(\frac{[C_3]}{[C_2]}\right) + \beta_2$$

where log(.) means common logarithm.

Extending these correlation models, we developed a quality model to infer MI for slurry HDPE reactor [32].

$$\log(MI_i) = \beta + \alpha_1 \log \frac{[H_2]}{[C_2]} + \alpha_2 \log \frac{[C_3]}{[C_2]} + \alpha_3 \log \frac{[C_4]}{[C_2]} + \alpha_4 \log[R] + \alpha_5 \log(T)$$
(3)

$$\frac{\mathrm{d}\log(MI_c(t))}{\mathrm{d}t} = \frac{1}{\tau(t)}\log(MI_i(t)) - \frac{1}{\tau(t)}\log(MI_c(t)) \tag{4}$$

The instantaneous MI model, Eq. (3), is formulated in the form of a linear combination of logarithmic concentration ratios rather than the logarithm of a linear combination of the ratio, Eq. (1).

In our second inferential scheme [29], the ℓ^{∞} Wave-Net, which is an artificial neural network proposed by Kourlouris [34], was used to develop an instantaneous property model and incorporated with Eq. (2), to infer MI of loop HDPE reactors. The developed instantaneous MI model was of the form:

$$MI_i^{-0.286} = \text{Wave-Net}\left(\frac{[H_2]}{[C_2]}, \frac{[C_2]}{[C_2]}, \frac{[C_6]}{[C_2]}, \frac{[R]}{[C_2]}\right)$$
 (5)

where Wave-Net(x) is constructed by a weighted sum of family wavelets function of x:

Wave-Net(x) =
$$\sum_{m=0}^{L} \sum_{k} c_{mk} \Psi_{mk}(x) + \sum_{k} d_{Lk} \Phi_{Lk}(x)$$
 (6)

L represents the coarsest scale, with c_{mk} and d_{Lk} being the coefficients of the multi-dimensional wavelets Ψ and scaling functions Φ .

There were some neural network applications in attempt to relating the instantaneous polymer properties with process variables. However, few are capable of learning on-line. By taking advantage of the fact that Eq. (6) is the linear function of the coefficients to be trained, the coefficients of the Wave-Net can be adapted on-line by the irregularly spaced quality measurements and on-line measured process variables.

We have been testing three schemes to assess their usability in several industrial plants: single loop-reactor; a train of two reactors plant where high density polyethylene (HDPE) is produced; a slurry CSTR reactor for HDPE; HDPE reactor with metallocene catalysts. One of the sets of test data is shown in Fig. 5.

Fig. 6 shows MI values estimated by three different models. They needed an appropriate parameter adaptation or on-line learning mechanism for practical use. Fig. 7 illustrates the estimated MI with an Extended Kalman filter. The three models did not differ significantly from

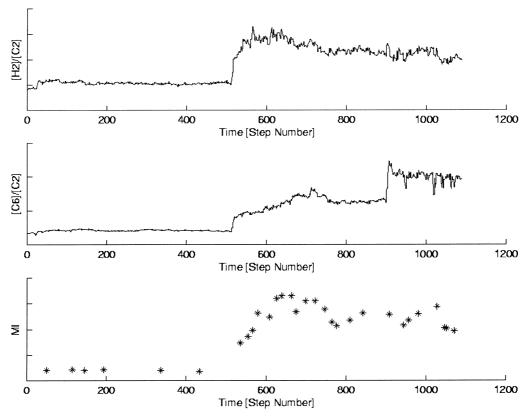


Fig. 5. Process data and MI measurement of a HDPE plant.

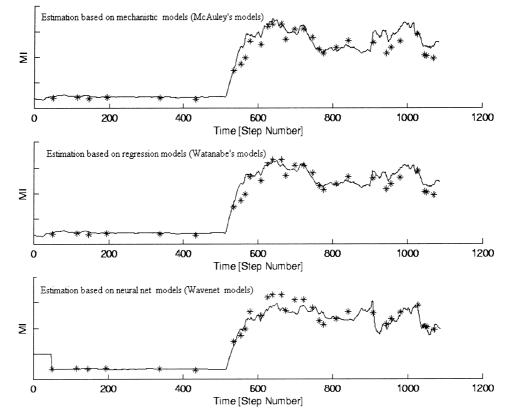


Fig. 6. Estimation by three different models.

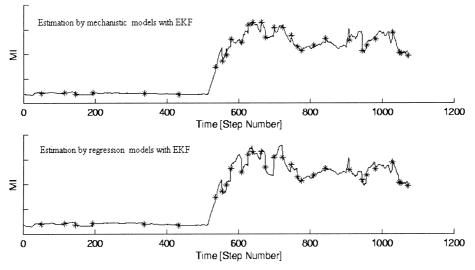


Fig. 7. MI estimation by EKF.

each other in estimation accuracy on the learning data. However, the risks of extrapolation by neural net type models were perceived when comparing the function of obtained instantaneous *MI* model of Wave-Net with those of other models (Fig. 8).

In the region where training data exist, the function expressed by Wave-Net has a profile similar to those of

other models. But, outside the region, three functions show different shapes. When neural-net type models are used for the property inferential system, a considerable amount of learning data is required or on-line learning function must be equipped. It is clearly dangerous to use neural net model for estimating the properties of a new grade.

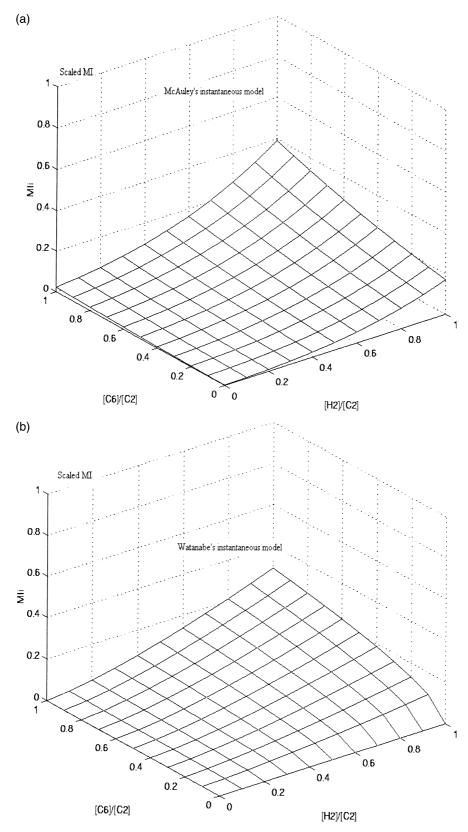


Fig. 8. Function of instantaneous models: (a) mechanistic + empirical models; (b) empirical regression models.

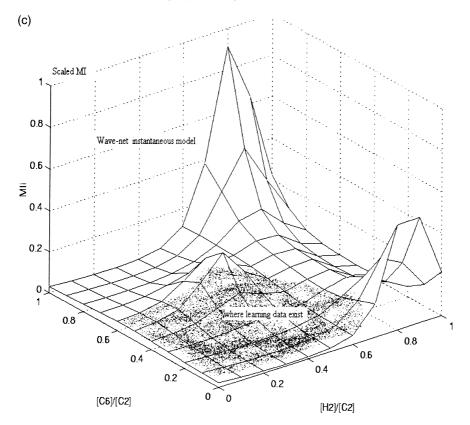


Fig. 8. (c) Function of instantaneous models: neural net model.

4.3. On-line sensors during extrusion

Recently, several advances have also been made in the development of on-line sensor at extruder units. On-line sensors for rheological properties such as viscosity and yield stress were developed [35]. Using fiber-optic linked devices, such as Raman and NIR, new sensors were also developed for monitoring polymer properties [9,10,36,37]. Among them, near infrared (NIR) spectroscopy has greatly expanded capabilities with the help of recent advances in chemometrics. Watari et al. utilized the NIR to measure the density of polyethylene on-line [38]. In the sense that it is incorporated with multivariate schemes, such as PLS and PCA, NIR on-line sensing can be regarded as a softsensor with a black box model.

In the future, by incorporating on-line soft sensors installed around reactors with those installed around extruder, polymer quality can be monitored over the entire plant as illustrated in Fig. 9.

5. Polymer reactor control

5.1. Grade changeover operation

As mentioned in the introductory section, one polymer production plant has been forced to produce various grades of high-quality polymers. This requires a socalled grade changeover operation in which frequent changes in operating condition are necessary. However, these grade changes often result in relatively large settling times and/or overshoots, and consequently significant amounts of off-specification polymers are produced. From the economical point of view, it is very important to reduce the amount of off-spec polymer produced during the grade changeover operation. "Time optimal" operation alone cannot accomplish the reduction of off-spec polymers. For example, drastic change in instantaneous polymer properties, such as

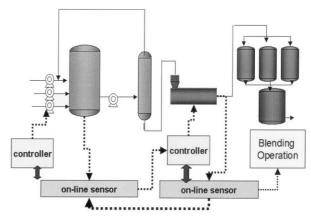


Fig. 9. Incorporation of on-line quality sensors.

average molecular weight, during changeover operation may help shorten the changeover time but it easily deteriorates the quality of total polymer. From a polymer quality viewpoint, changeover operation while keeping instantaneous polymer properties in a certain range is important as illustrated in Fig. 10 [39].

Furthermore, safety is also an important aspect to be considered during grade changes, since certain kinds of grade changeover require drastic change in operating conditions, resulting in run-away reaction in the reactor. Thus, for an ideal setting, it is crucial, during a grade changeover operation, to change the operating conditions as safely and quickly as possible while keeping the quality within an acceptable range. At the same time, it is equally crucial, during the following period in which the same grade is being produced, to maintain a stable product quality against some inevitable disturbances. To that end, what is needed is a sophisticated control system equipped with an ability to provide optimal grade changeover operation trajectories, to track those trajectories accurately, and to perform regulatory control efficiently.

Debling et al. tested several patterns of grade transition operation for several reactors on their dynamic simulator, POLYRED [40]. To obtain the optimal trajectory of grade changeover operation, off-line optimization is often performed by using nonlinear programming methods [41-43]. McAuley and MacGregor proposed optimal grade transition strategies for a gas phase polyethylene reactor by solving an optimization problem with a lumped parameter process model [42]. Ohshima et al. also derived an optimal grade changeover trajectory and tested it on a loop and a gas phase polypropylene reactors in series [43]. The optimal operation trajectory was calculated by using dynamic programming with the simplified quality models among MI, density, and gas concentrations. For a train of reactors, the optimal grade changeover was not attained by simply changing the operating conditions from the steady state value of one grade to another, as shown in Fig. 11.

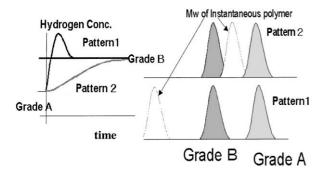


Fig. 10. Changes in instantaneous property during grade changeover operation.

Hydrogen Conc. at 1st reactor

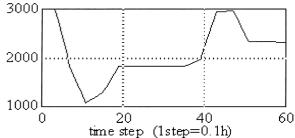


Fig. 11. An optimal trajectory of H₂ concentration in the reactor.

Recently, Takeda [44] applied the control vector parameterization method to the dynamic simulator, POLYLED, to derive the optimal grade changeover strategies for bimodal loop reactors. The optimized material flows show a pattern similar to our results.

In view of the recent advances in optimization schemes, it is anticipated that real time optimization will be put to practical use so as to optimally operate the entire polymer plant in the presence of model/plant mismatch and disturbances.

5.2. A control system for a polymer plant

With the availability of relatively cheap computer power, several control schemes including non-linear controllers have been proposed for polymer reactors. An enormous amount of work has been done on polymerization reactor controls, which is impossible to review in a short paper. Thus, the rest of this paper focuses on control systems for grade changeover operation of a polyolefin reactor.

When one develops a control system to perform grade changeover operation for the polymer production plant, there are at least two different approaches to be considered. One is an iterative open-loop optimization scheme, i.e. receding horizon control [27,45,46]. The other is a combination of feedforward and feedback controls [47,48].

In the iterative open-loop optimization scheme, a new optimal trajectory is recomputed by using nonlinear process models, and the first input action calculated by the open-loop optimization is implemented every time new measurements or estimates of controlled and state variable become available. To calculate the input action one can use a linear model constructed at every moment by performing a step response test on the prepared nonlinear models.

The concept of combination of feedforward and feedback controllers is as follows. The optimal trajectory of both manipulated and controlled variables (polymer property such as MI) for grade changeover is pre-calculated or prepared as a database of empirical knowledge. When the setpoint in polymer property is

changed, the optimal trajectory of manipulated variables calculated off-line is introduced to the plant in a feedforward manner. Because of the presence of disturbance and model/plant mismatch, controlled variables deviate from the desired optimal trajectory. Robust feedback controllers take care of the deviation.

McAuley et al. developed a nonlinear controller for a gas phase fluidized bed polyethylene reactor in the framework described above [47]. In their system, an optimal trajectory for a grade changeover is determined from the off-line dynamic optimization. This trajectory is tracked using a nonlinear controller, which is developed on the basis of global input—output linearization of a simplified process model.

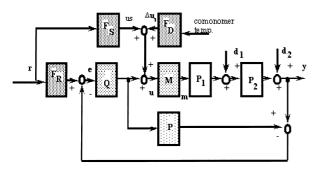


Fig. 12. Control structure for grade changeover.

The structure of control system that we developed for controlling *MI* at our polyethylene plant consisted of a feedforward controller and a linear-feedback controller as illustrated in Fig. 12 [48].

The overall control system is realized in the form of a multirate cascade system. In the inner loop controller, which is expressed by P_1 , the monomer and catalyst feed flow rate, etc., are manipulated to control concentration ratios, [H₂]/[C₂], in the reactor every 15 min. The transfer function P_2 represents the dynamics between the concentration ratio and the logarithmic MI of product polymer. The setpoint of MI, r, is sent to the outer loop, and the outer loop controller controls the MI value of the product polymer every 2 h by manipulating the setpoint of the concentration ratios, u. The MI estimate by soft sensor is used in the controller. When the setpoint in MI is changed, a feedforward element, F_s , calculates an optimal trajectory for the manipulated variable and moves the input following the trajectory. That is, the setpoint of the concentration ratio is changed in a feedforward manner. At the same time, F_R generates an optimal trajectory of the controlled variable, MI, so as to calculate deviation of the measured controlled variable from its optimal trajectory. The deviation, e, is fed to a linear IMC controller, Q, with a linear process model, \tilde{P} (i.e. model of MP_1P_2), obtained from the linearization around optimal trajectories. The M is the

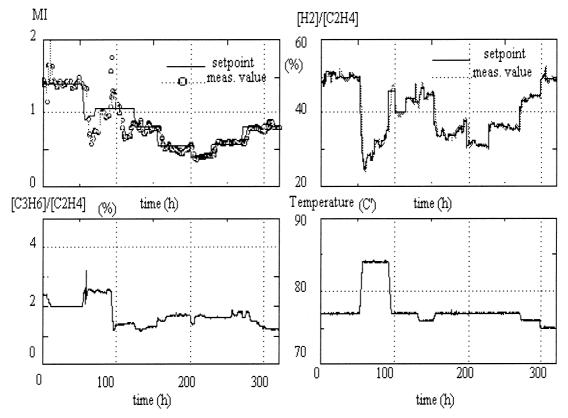


Fig. 13. Grade changeover operation of a HDPE plant.

compensator by which the dynamics between the setpoint and the $\log MI$ can show an overdamped response. If F_s and F_R are inactive, the control system works as a regulatory control. Fig. 13 shows a series of the grade changeover operation. The control system was activated after 120 h have elapsed.

6. Optimal blending operation

As illustrated in Fig. 1, blenders and storage tanks are installed downstream of the production line for damping out quality variability. It is an admission of the fact that the reduction of quality variability by the reactor

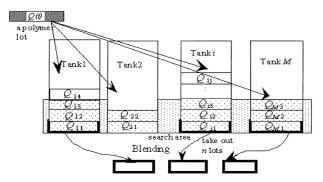


Fig. 14. Polymer silos.

control system alone is not good enough to satisfy the customers' strict demand for quality. Therefore, a cooperative blending operation should be pursued at the blending silos. However, the current blending operation is performed by skilful operators. Optimizing the blending operation could provide further reduction of quality variability. Fig. 14 illustrates a simplified structure of the blender and storage tanks process that we dealt with.

A certain amount of polymer produced at the reaction process is stored and made into one lot. At the same time, polymer quality is measured as a representative quality value of the lot. After measuring, the lot is assigned to one of the storage tanks and stored until blending. The blending operation is performed on a first-come-first-serve basis, taking three polymer lots from bottom of some tanks and blending them so as to make the average quality value of the blended polymer close to the target value.

The operator should decide how many lots to be taken out from which tanks. He should also decide to which tank a new polymer lot should be assigned so as to make the quality variability of blended polymer as small as possible. Both blending and tank assignment optimization problems are formulated as MILP problems [49].

A result of the optimization is shown in Fig. 15: (a) *MI* variability of the blended polymer operated by operator,

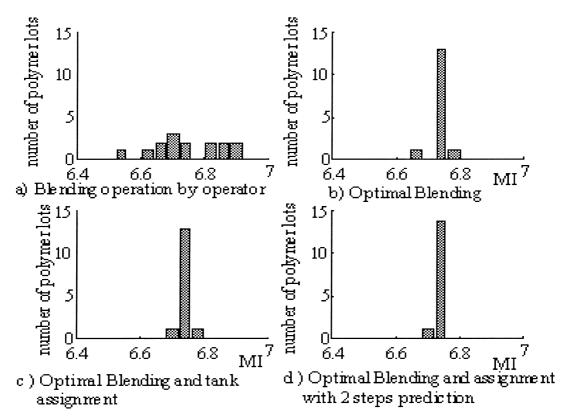


Fig. 15. Results of optimized blending operation.

(b) the result of the optimal blending operation and (c) the result of the simultaneous blending and assignment optimization. Fig. 15(d) is a result of optimal blending and assignment operation assuming that quality of two new polymer lots in future could be predicted. On-line property sensor will make the prediction possible.

As can be seen in these results, the optimal blending operation greatly helped reduce product quality variability. The optimization of blending units will lead to relaxation of constraints on reactor control, in other words, it will take some of the load off of the process control or it will lead to cost saving by reducing the number of storage tanks.

7. Conclusion

As discussed, quality control of a polymer plant is a very important and active area for future research. To attain the highest quality control in the production plant, it is indispensable to integrate technologies in three key areas, process control, sensing, and optimization, and to create an intelligent manufacturing system supervising the entire plant. The most important factor in developing such a system is quality modeling. Without it, practical quality control cannot be achieved.

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References

- [1] F. Rodriguez, Principles of Polymer Systems, Taylor & Francis, 1996, p. 2.
- [2] W.H. Ray, Polymerization reactor control, ACC 1985, 842–
- [3] J.F. MacGregor, A. Penlidis, A.E. Hamielec, Control of polymerization reactors: a review, Polymer Process Engineering 2 (2/3) (1984) 179–206.
- [4] J.F. MacGregor, Control of polymerization reactor, DYCORD '86, 1986, pp. 31–35.
- [5] C. Kiparissides, J. Morris, Intelligent manufacturing of polymers, ESCAPE 6, 1996a.
- [6] C. Kiparissides, Polymerization reactor modeling: a review of recent developments and future directions, Chem Engng Sci 51 (10) (1996) 1637–1659.
- [7] B.A. Ogunnaike, A contemporary industrial perspective on process control theory and practice, DYCORD+95, 1995, pp. 1–8.
- [8] K. Soga, M. Kaku, N. Kashiwa, Polymerization process technology—polyolefin, Dainippon Tosho (1993) (in Japanese).

- [9] D.C.H. Chien, A. Penlidis, On-line sensors for polymerization reactor, JMS-Rev, Macromol. Chem. Phys. C30 (1) (1990) 1–42.
- [10] C. Kiparissides, E.G. Chatzi, O. Kammona, Recent hardware sensors developments for monitoring polymerization reactions, DYCOPS-5, 1998, pp. 72–79.
- [11] M. Embiruçu, E.L. Lima, J.C. Pinto, A survey of advanced control of polymerization reactors, Polymer Engng. and Sci. 36 (4) (1995) 433–447.
- [12] L.L. Bohm, P. Gobel, O. Lorenz, T. Tauchnitz, PE-HD-production using a complex model based state observer, Dehema-Monograplien, Bond 127 (1992) 257–273.
- [13] K.B. McAuley, J.F. MacGregor, On-line inference of polymer properties in an industrial polyethylene reactor, AIChE J. 37 (6) (1991) 825–835.
- [14] F.J. Doyle, Nonlinear inferential control for process applications, ADCHEM-97, 1997, pp. 170–181.
- [15] M.T. Eaton, N.L. Ricker, Extended kalman filtering for particle size control in a fed-batch emulsion polymerization reactor, ACC, 1995, pp. 2697–2701.
- [16] T.J. Crowley, K.Y. Choi, On-line monitoring and control of a batch polymerization reactor, J. of Proc. Control 6 (2/3) (1996) 119–127.
- [17] B.A. Ogunnaike, On-line modeling and control of an industrial terpolymerization reactor, Int. J. of Control 59 (3) (1994) 711– 729.
- [18] C. Scali, M. Morretla, D. Semino, Control of the quality of polymer products in continuous reactors, J. of Process Control 7 (5) (1997) 357–369.
- [19] G.R. Sriniwas, Y. Arkun, F.J. Schork, Estimation and control of an a-olefin polymerization reactor, J. of Proc. Control 5 (5) (1995) 303–313.
- [20] D. Semino, M. Morretta, C. Scali, Parameter estimation in extended kalman filters for quality control in polymerization reactor, Comp. Chem. Engng 20 (1996) 913–918.
- [21] J. Zhang, X. Yang, J. Morris, C. Kiparissides, Neural network based estimations for batch polymerization, DYCORD+95, 1995, pp. 129-134.
- [22] W-H. Chan, C.A.O. Nascimento, Use of neural networks for modeling of olefin polymerization in high pressure tubular reactors, J. of Applied Polymer Science 53 (1994) 1277–1289.
- [23] J.F. MacGregor, C. Jaeckle, C. Kiparissides, M. Koutoudi, Process monitoring and diagnosis by multiblock PLS methods, AIChE J. 40 (5) (1994) 826–838.
- [24] T. Kourti, J. Lee, J.F. MacGregor, Experience with industrial application of projection methods, Computer Chem. Engng 20 (1996) 745–750.
- [25] C. Kiparissides, C.G. Verros, A. Pertsinidis, I. Goossens, On-line parameter estimator in high-pressure low density polyethylene, AIChE J. 42 (2) (1994) 440–465.
- [26] P. Appelhaus, S. Engell, Extended observer for the polymerization of polyethyelene terephthalatem, IFAC World Cong. M (1996) 175–180.
- [27] R.K. Mutha, W.R. Cluett, A. Penlidis, Nonlinear model-based estimator and control of a polymer, Poly. Reac. Engng Meeting, Palm Coast, 1997.
- [28] G. Guiochon, G. Defaye, C. Vidal, L. Caralp, Kalman filter and neural network for on-line estimation of polymer chain characteristics, DYCORD+95, 1995, pp. 287–297.
- [29] M. Ohshima, A. Koulouris, S. Tomita, G. Stephanopoulos, Wave-net based on-line quality inference system for polymerization processes DYCORD+95, 1995, 275–280.
- [30] Y. Yabuki, J.F. MacGregor, Product quality control in semibatch reactors using mid-course correction profile, ADCHEM97, 1997, pp. 189–193.
- [31] B. Lines, D. Hartlen, F.D. Paquin, S. Treiber, M. Tremblay, M. Bell, Polyethylene reactor modeling and control design, Hydrocarbon Processing (1993) June 119–124.

- [32] F. Watanabe, M. Ogawa, M. Ohshima, I. Hashimoto, Quality control systems for an industrial high density polyethylene process, AIChE mtg., St. Louis, 1993, paper no. 149m.
- [33] T.W. Karjala, H. Meerdink, A. Dems, B.C. Real-time estimation of polymer properties in an industrial polyethylene reactor, ACC, 1997, pp. 3063–3067.
- [34] A. Koulouris, Multi-resolution learning in nonlinear dynamic process modeling and control, Ph.D. thesis, MIT, 1995.
- [35] T.O. Broadhead, B.I. Nelson, J.M. Dealy, An in-line rheometer for molten plastics-design and steady state performance characteristics, Int. Polymer Processing 8 (2) (1993) 104–112.
- [36] C.H. Sohl, Just in time compounding using feedback control from a near infrared melt composition sensor, ANTEC'91, 1991, pp. 846–849.
- [37] A. Kettry, H.G. Hansen, Real-time analysis of molten ethylene vinyl acetate copolymers using near infrared spectroscopy, ANTEC'95, 1995, pp. 2824–2831.
- [38] M. Watari, N. Mitsui, H. Higashiyama, M. Tomo, On-line molten polymer measurement using near infrared fourier transfer spectroscopy, Tech. Info. of Yokogawa, 1996, TI11VOA1-11.
- [39] W.H. Ray, Modeling and control of polymerization processes, AIChE mtg, 1996, Chicago, paper no. 176.
- [40] J.A. Debling, G.C. Han, F. Kuijpers, J. Verburg, J. Zacca, W.H. Ray, Dynamic modeling of product grade transitions for olefin polymerization processes, AIChE J. 40 (3) (1994) 506–520.

- [41] S.S. Jang, W.L. Yang, Dynamic optimization of batch emulsion of vinyle acetate—an orthogonal polymerization initiator policy, Chem. Eng. Sci. 33 (1989) 515–528.
- [42] K.B. McAuley, J.F. MacGregor, Optimal grade transitions in a gas phase polyethylene reactor, AIChE J. 38 (10) (1992) 1564–1576.
- [43] M. Ohshima, I. Hashimoto, T. Yoneyama, M. Takeda, F. Gotcha, Grade transition control for an impact copolmerization reactor, ADCHEM'94, 1994, pp. 507–512.
- [44] M. Takeda, Optimal grade transition strategies for multistage olefin polymerization reactors with bimodal products, MS thesis, 1997, University of Wisconsin.
- [45] T. Peterson, E. Hernandez, Y. Arkun, F.J. Stork, A nonlinear DMC algorithm and its application to a semibatch polymerization reactor, Chem. Engng. Sci. 47 (4) (1992) 737–753.
- [46] P. Pladis, O. Kiparissides, Supervisory control of high pressure LDPE reactors, DYCOPS-5, 1998, pp. 205–210.
- [47] K.B. McAuley, J.F. MacGregor, Nonlinear product property control in industrial gas-phase polyethylene reactors, AIChE J. 39 (5) (1993) 855–866.
- [48] M. Ogawa, M. Ohsihma, F. Watanabe, K. Morinaga, I. Hashimoto, Quality inferential control of an industrial high density polyethylene process, IFAC World Cong. M (1996) 163–168.
- [49] M. Ohshima, M. Tanigaki, H. Ikenoue, S. Tomita, Optimal blending operation at polymer silos, Kagaku Kogaku Ronbunshu 23 (3) (1997) 384–390.