Analysis of the Dynamic Behavior of a Starch Foam Extrusion Process

Yogaraj Nabar, Ramani Narayan

Department of Chemical Engineering and Materials Science, Michigan State University, East Lansing, Michigan 48824

Received 8 October 2004; accepted 18 May 2005 DOI 10.1002/app.22942 Published online in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: The starch foam extrusion process was modeled as a multiple input multiple output (MIMO) process, and the dynamics of the process were studied as a response to step changes in the input variables such as starch feed rate, screw speed, moisture content (MC), and poly(hydroxy aminoether) (PHAE) feed rate. The responses were modeled as first-order responses with a time delay. The linearity of the process was determined over a range around the setpoint, and the parameters defining the first-order system such as gain " K ," time constant " τ ," and dead time " t_d " were determined in the linear range. The transfer function models can then be used in a predictive computer control system for on-line fine-tuning of the operating conditions. This could ensure a consistently high quality product even when low frequency disturbances are present in the system. It was observed that the time constants and the dead times recorded for both the pressure and torque responses did not exhibit significant variation within each manipulated or input variable tested, indicating a dynamic linearity with re-

INTRODUCTION

Foam plastic packaging is experiencing growing pressure from existing and proposed environmental and disposal regulations, and market-based sustainability initiatives. It presents a major disposal problem for companies and municipalities, as it is lightweight and bulky, and so does not lend itself to a viable economic and environmentally responsible recycling operation due to expensive handling and transportation costs. It is not biodegradable, which makes disposal in soil or composting operations untenable. Further, issues such as sustainability, industrial ecology, biodegradability, and recyclability are becoming major considerations in a company's product packaging design, especially with single use disposable packaging. There is, thus, a market need for bio-based, biodegradable foam plastic packaging that can be safely and effectively disposed in soil or in composting operations, but retains all of the current foam plastics performance requirements. In previous work, we have reported on the rationale, spect to each manipulated variable. It was also observed that for the same step-input variations in the manipulated variables, the torque loading on the twin-screw extruder exhibited a faster response (lower dead time), and also reached a steady state sooner (lower time constant). The MC and screw speed seem to be the most destabilizing variables, as they induce rapid responses in the process variables. The MC in the extruder was, hence, determined to be the most influential factor in the stability of the process, followed by screw speed and starch feed rate. PHAE feed rate was the least significant variable. Multiple step-input tests were carried out to determine the validity of the principle of superposition. The validity of the principle of superposition implied the linearity of the process in the domain tested. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 101: 3983–3995, 2006

Key words: process control; modeling; starch; foams; extrusion

design, and engineering of bio-based, biodegradable polymer materials. $1-4$

Starch, an anhydroglucose polymer from corn, offers a structural platform to manufacture sustainable, biodegradable foam packaging. Starch extrusion processes are multiple input multiple output (MIMO) complex systems. Harper⁵ gave a detailed description on the mechanics of extrusion. The phenomenon of starch foaming involves the physicochemical properties of starch, which are modified during extrusion. The rheological properties of the starch plastic are in turn reliant on these physicochemical properties,⁶ which affect the quality attributes of the foamed product.

Emphasis in starch extrusion research has focused on developing models to describe the complex processes in an extruder, predict some of the changes that occur, and aid in new process development.⁷ The control of such processes is directly linked to economic, qualitative, and scientific interests. As far as control is concerned, the process has to be modeled. Different approaches, which are roughly of two types, have been used to model extrusion processing: white-box modeling and black-box modeling.⁸ White-box modeling requires as much knowledge as possible to in-

Correspondence to: R. Narayan (narayan@msu.edu).

Journal of Applied Polymer Science, Vol. 101, 3983–3995 (2006) © 2006 Wiley Periodicals, Inc.

Figure 1 Starch foam extrusion process schematic with manipulated, process, and product variables.

corporate all the internal laws (physical, chemical, and biological), which rule the system. The result is that all the coefficients of the model may have a physical significance. However, a complete description, including temperature effects, non-Newtonian rheology, and dynamics, is indeed complicated, and therefore, simplifying assumptions are usually made. Also, to truly simulate the extrusion process, thermal and physical model parameters must be determined for each particular product and extruder-die combination. On the other hand, black-box modeling means that the model is empirical, reduced to a simple mathematical relationship between the inputs and outputs of the process. Such models are valid only over a definite range of experimental conditions, but they are often adequate to develop process control strategies. Concerning food extrusion, three different techniques of blackbox modeling have been implemented: residence time distribution (RTD), response surface analysis (RSA), and dynamical identification.⁹ Janssen¹⁰ and Martelli¹¹ presented engineering analyses of a twin-screw extrusion process, including discussions on RTD, melting mechanisms, power consumption, and operating characteristics of twin-screw extruders. RSA has been used by several researchers^{12–16} for the optimization of extrusion processing. This approach enabled correlation of the important processing variables to product quality without an engineering model. Results were generally limited to the experimental conditions because of the nonlinear response of the internal state of the extruder to externally manipulated variables such as screw speed, moisture, and feed rate. They have especially been used to establish static relationships.

A basic modeling technique applied to extrusion processing is transfer function modeling from input/ output data. Several researchers have studied the dynamic modeling and control of extrusion processes, based on their gross dynamic input– output behavior.17–24

In our previous paper, 25 we have reported on the twin-screw extrusion production of starch foams using poly(hydroxy aminoether) (PHAE) as the functional aid, as well as on the optimization of the process. In the present work, the dynamic response of

various process variables of a twin-screw extrusion starch foaming process or the sensitivity of the process parameters to the change in inputs were studied, both of which must be known for the implementation of process control. The dynamic characteristics are obtained with step tests on four independent extrusion operating variables: starch feed rate, moisture content (MC), screw speed, and PHAE feed rate. The response is monitored on the following outputs: melt pressure, melt temperature, and specific mechanical energy (SME). The corresponding system and its relevant manipulated, process, and product variables are shown in Figure 1.

EXPERIMENTAL

Materials

The type of starch used was hydroxypropylated high amylose cornstarch (70% amylose content). The starch was purchased from National Starch and Chemicals (Indianapolis, IN), under the trade name of HYLON 7. The density of HYLON 7 starch is 1.2 g/cm³. The inherent MC of the starch was 11.2% under ambient conditions. Water was used as the plasticizer as well as the blowing agent. The total MC of the starch was adjusted by adding water at the feed throat of the extruder with a volumetric pump to obtain the various tested levels of MC. Talc (Magnesium Silicate), used as the nucleating agent, was obtained from Luzenac (Ontario, Canada). It has a specific gravity of 2.76 and a bulk density of 150 kg/m 3 . The talc content was maintained at 1% for all the experiments. Poly(hydroxyamino ether) (PHAE) is an additive, which offers the adhesion and durability of epoxy resins, with the flexibility and processability of thermoplastic resins. PHAE was purchased from Dow Chemicals (Midland, MI), under the trade name BLOX 110. PHAE has a melt temperature of 75°C, and is produced by reacting liquid epoxy resin (LER) with hydroxy functional dinucleophilic amines and diglycidyl ethers of bisphenol-A, hydroquinone, or resorcinol (RDGE).^{26,27}

Figure 2 Twin-screw extrusion screw configuration.

Experimental design

The experimental setup used in this study was a twinscrew extrusion system. The twin-screw extrusion system consisted of an extruder driver with a speed control gearbox, a ZSK-30 twin-screw corotating extruder with a screw diameter of 30 mm, an L/D of 32, a positive displacement pump for injecting water into the extruder, and accurate single-screw feeders for feeding starch and PHAE. The screw configuration is shown in Figure 2. This specific screw configuration was selected to get the best physicomechanical properties based on our previous work.²⁵ A cylindrical filament die 2.7 mm in diameter and 8.1 mm in length, with a cooling sleeve, was assembled to the extruder. The sensors were mounted on the die to measure the temperature and pressure of the melt.

The dynamical behavior of the process was studied through the following outputs: melt pressure (*P*) and melt temperature (*T*). The specific mechanical energy (SME) was calculated using the following formula:

$$
SME = \frac{kW \times \text{torque} \times \frac{RPM_a}{RPM_r}}{\dot{m}}
$$
 (1)

where SME is the specific mechanical energy (kW h/kg); kW is the rated motor power (kW); torque is the motor load (decimal); RPM_a is the actual screw speed; RPM_r is the rated screw speed; and m is the mass flow rate (kg/s) .

The experiments consisted of different sequential variations around the following central set point²⁵: starch feed = 11.16 kg/h, MC = 17.42% (w/w) on an overall basis, screw speed $= 200$ rpm, and PHAE feed $= 0.78$ kg/h. Its corresponding response variables are process temperature, $T = 105^{\circ}$ C, pressure at the die, *P* $= 720$ psi, and SME $= 0.21$ kW h/kg (torque loading $= 72\%$). The experimental design for the single input variation runs is shown in Figure 3.

Each "forward step test" always succeeded by a "back step test" to let the process return to its original set point. The forward and back steps gave approximately the same results, suggesting that probably there is negligible hysteresis.

To get significant and comparable outputs, the overall range of stimulation imposed on each control variable was set according to the pre-estimated gain of that variable. At steady state (set point), a step test was performed on either one or multiple input variables. Once the output variables reached a steady value, the study was reduced to a static comparison between the output *Y(t*) and the input *U(t*), i.e., to the gain *K*.

$$
K = \frac{DY(t)}{DU(t)}
$$
 (2)

To compare the different steady state gains, it is possible to define a relative steady state gain K_R , where

$$
K_R = \left[\frac{DY(t)}{Y(t)}\right] / \left[\frac{DU(t)}{U(t)}\right]
$$
 (3)

Linearity

It is important to assess the linearity or nonlinearity of a process, as it helps to understand and predict its response to any variation in input. The hypothesis of linearity is an implicit and necessary prerequisite to most of the classical techniques of process control. A linear system must exhibit a constant steady state gain, whatever be the magnitude of step test applied.

Dynamic (transient) responses

Once a step-input variation in the starch feed rate, screw speed, MC, and PHAE feed rate was made (time, $t = 0$), the pressure and torque readings were

Figure 3 Single step-input step variation experimental design.

No.	Step change in starch feed rate $(\%)$	K_T	$K_{\rm RT}$	K_P	$K_{\rm RP}$	K_{torque}	$K_{\rm R, torque}$	$K_{\rm SME}$	$K_{\rm R, SME}$
1	-25.00	0.4	0.04	62.7	0.97	0.06	0.93	0.000	-0.01
2	-20.00	0.4	0.05	57.3	0.89	0.06	0.93	0.000	-0.01
3	-15.00	Ω	Ω	50.8	0.79	0.06	0.89	-0.001	-0.06
$\overline{4}$	-10.00	Ω	Ω	38.5	0.60	0.06	0.88	-0.001	-0.07
5	-5.00	Ω	Ω	32.3	0.50	0.06	0.86	-0.002	-0.08
6	$+5.00$	Ω	Ω	17.9	0.28	0.06	0.89	-0.001	-0.05
7	$+10.00$	Ω	Ω	39.4	0.61	0.06	0.89	-0.001	-0.05
8	$+15.00$	0.6	0.06	47.8	0.74	0.06	0.93	0.000	-0.01
9	$+20.00$	0.9	0.1	60.5	0.94	0.06	0.92	0.000	-0.01
10	$+25.00$	0.7	0.08	68.1	1.06	0.06	0.93	0.000	-0.01

TABLE I Steady State Process Gains for Step-Input Variations in the Starch Feed Rate

manually recorded every 5 s until a steady state was reached. A common assumption in many time series techniques is that the data are stationary. A stationary process has the property that the mean, variance, and autocorrelation structure do not change over time. These conditions were too drastic for these experiments, as the measurements of process variables were very noisy. It was assumed that the process would become stationary and, by graphical estimation, the time required to reach each steady state was calculated approximately.

Multiple input tests

Some multi-input experiments were carried out by performing step tests simultaneously on multiple manipulative variables. The principle of superposition was verified for multiple step input tests, within the linear domain. If the principle of superposition is satisfied, it implies that the system is linear within the domain tested. The variation of the process variables, namely, pressure, torque, and SME in response to the multivariable input step tests were measured at steady state. Pressure, torque, and SME were also calculated theoretically, using a linear combination (principle of superposition) of the steady state gains previously determined by single input step tests. The following formula was used,

$$
DY = Kstart feedD(startch feed)+ Kscreen speedD(screw speed)+ KMCD(MC)+ KPHAED(PHAE) (4)
$$

where, *Y* is pressure/torque/SME.

These experiments were conducted to verify if the linearity observed on each variable could be extrapolated through an additive law to a multivariate control. The values of pressure, torque, and SME obtained for the multiple input tests were fit to a model deemed

appropriate, using STAT-EASE "Design of Experiments 6.0" modeling software. A central composite response surface experimental design²⁸ was employed to investigate the influence of step-changes in the starch feed rate, screw speed, MC, and PHAE feed rate on the process variables (pressure, torque, and SME), which further govern the foam product functionalities (density, expansion ratio, and resilience). Regression analyses were employed to fit the experimental data to linear polynomials.²⁹ In these experiments conducted, the extruder almost always verified the conditions, which postulate that a bounded variation of the input induces a bounded variation of the output (BIBO stability). This means that, for and around the set point selected, the extruder remains far away from any uncontrollable change that could lead to an over-torque, or an over-pressure, and consecutively to a blockage of the screws (shutdown).

RESULTS AND DISCUSSION

Step-input variations in the starch feed rate

The absolute (K) and relative (K_R) steady state process gains for step-input variations in the starch feed rate are shown in Table I. For the range of the tested set points, the melt temperature *T* was hardly sensitive to the variations in starch feed rate because feed variations induce tiny variations in self-heating. The selfheating would be as a result of the shear imposed on the extruding material (viscous dissipation). The set temperature at the die was 105°C. The maximum temperature rise was 2°C for a 25% increase in starch feed. Similarly, the temperature decreased with a decrease in starch feed rate (though negligible). Hence, the values of G_R for this variable were positive. The temperature decreased by just 1°C for a 25% decrease in starch feed.

The melt pressure *P*, however, displayed significant variations due to an increase/decrease in the starch

steady state rrocess came for step mpar valuations in the Extrasion seren specie								
Step change in screw speed $(\%)$	K_{τ}	$K_{\rm RT}$	K_p	$K_{\rm RP}$	K_{torque}	$K_{\text{R,torque}}$	K_{SME}	$K_{\rm R, SME}$
-12.50	0.04	0.08	-2.9	-0.80	-0.005	-1.47	0.000	-0.28
-10.00	0.00	θ	-2.8	-0.78	-0.006	-1.54	0.000	-0.39
-7.50	0.00	θ	-2.8	-0.78	-0.005	-1.52	0.000	-0.40
-5.00	0.00	θ	-2.9	-0.81	-0.005	-1.50	0.000	-0.43
-2.50	0.00	θ	-3.2	-0.89	-0.006	-1.56	-0.001	-0.52
$+2.50$	0.00	Ω	-3.8	-1.06	-0.006	-1.67	-0.001	-0.71
$+5.00$	0.00	θ	-3.5	-0.97	-0.006	-1.56	-0.001	-0.63
$+7.50$	0.07	0.13	-3.5	-0.98	-0.005	-1.46	-0.001	-0.57
$+10.00$	0.05	0.10	-3.3	-0.92	-0.005	-1.31	0.000	-0.44
$+12.50$	0.04	0.08	-3.2	-0.88	-0.004	-1.21	0.000	-0.36

TABLE II Steady State Process Gains for Step-Input Variations in the Extrusion Screw Speed

feed rate. The increase in *P* with the starch feed would be basically due to an increase in the filling ratio, and *vice versa*. Thus, the values of K_R for this variable were positive. It was observed that the relative gains varied with the magnitude of the step test, and they increased as the size of the step change increased. Also, the values of the gains were different for the positive tests and the negative tests.

An increase in the starch feed rate leads to an increase in the motor load (torque) on the extruder drive, as a larger amount of material was being processed. The SME increases with an increase in the torque on the motor, while decreases with an increase in the starch feed rate. The net result was a decrease in the value of SME. On the other hand, a decrease in the starch feed rate gave lower values of torque, and finally a higher value of SME. Thus, the values of K_R for this variable were negative. Also, it was observed that the relative gains decreased with an increase in the magnitude of the step size.

Step-input variations in the screw speed

The absolute (K) and relative (K_R) steady state process gains for step-input variations in the screw speed are shown in Table II. Similar to the results observed for changes in the starch feed rate, the changes in temperature due to changes in screw speed were negligible, with the T changing by ± 1 °C with an increase/decrease in screw speed, respectively. These changes were due to an increase/decrease in the viscous dissipation associated with material being worked between the screws and the barrel.

An increase in screw speed leads to a decrease in pressure due to a decrease in the viscosity of the melt due to increased shear (equation), and *vice versa*. Thus, the values of K_R for this variable were negative. Also, it was observed that the values of the relative gains were almost constant over the range studied, indicating the possibility of linearity. Unlike the trend observed earlier in step changes in the starch feed rate, the values of K_R decreased with an increase in the magnitude of step size.

An increase in the screw speed decreased the torque, while both have opposing effects on the SME. The net result was a decrease in the SME, giving a negative value for K_R . Similarly, the torque increased with a decrease in the screw speed, finally resulting in an increase in the SME, giving negative values for K_R again.

Step-input variations in the moisture content (% MC)

The absolute (K) and relative (K_R) steady state process gains for step-input variations in the % MC are shown in Table III. For the three process variables (*T*, *P* and SME), the variations in MC induced greater responses than the variations in starch feed rate and the screw speed.

It was observed that the temperature decreased by 3°C, with only a 10% increase in the MC, and it increased by 2°C, with a 10% decrease in MC. This resulted in higher values of K_R as compared to those reported earlier for the step tests in screw speed and the starch feed rate.

Also, the pressure and SME values varied inversely with step changes in the MC. A decrease in MC leads to an increase in the viscosity of the melt, leading to an increase in the melt pressure and the torque (and thus SME). It was observed that the values for the relative gain with respect to MC were almost constant, implying a linear system. It was also observed that the step changes in MC influenced the SME input more than it affected the melt pressure, contradictory to the effects of changes in screw speed and the starch feed rate. A decrease in MC leads to an increase in the viscosity of the melt, leading to an increase in the melt pressure and the torque.

				\mathbf{r}					
No.	Step change in moisture content $(\%)$	K_T	$K_{\rm RT}$	K_P	$K_{\rm RP}$	K_{torque}	$K_{\text{R,torque}}$	K_{SME}	$K_{\rm R, SME}$
	-10.00	-114.8	-0.19	-3789.6	-0.92	-5.2	-1.25	-1.78	-1.47
$\overline{2}$	-8.00	-143.6	-0.24	-3588.7	-0.87	-5.2	-1.27	-1.79	-1.49
3	-6.00	-95.7	-0.16	-3445.1	-0.83	-5.4	-1.30	-1.82	-1.51
4	-4.00	-143.6	-0.24	-3445.1	-0.83	-5.6	-1.35	-1.88	-1.56
5	-2.00	Ω	0.00	-3732.2	-0.90	-6.3	-1.53	-2.09	-1.73
6	$+2.00$	Ω	0.00	-5167.7	-1.25	-5.5	-1.32	-1.82	-1.51
7	$+4.00$	-143.6	-0.24	-4449.9	-1.08	-5.3	-1.28	-1.78	-1.47
8	$+6.00$	-191.4	-0.32	-4402.1	-1.06	-5.1	-1.23	-1.70	-1.41
9	$+8.00$	-143.6	-0.24	-4234.6	-1.02	-5.0	-1.20	-1.66	-1.38
10	$+10.00$	-172.3	-0.29	-4191.6	-1.01	-4.7	-1.14	-1.59	-1.31

TABLE III Steady State Process Gains for Step-Input Variations in the Moisture Content

Step-input variations in the PHAE content

The absolute (K) and relative (K_R) steady state process gains for step-input variations in the PHAE feed rate are shown in Table IV. It was observed that the changes in PHAE content did not affect the temperature of the melt, except a change of ± 1 °C at a step change of $\pm 100\%$ in the PHAE content, respectively. Also, the step changes in the magnitude of PHAE content marginally affected the pressure, giving low values of relative gain K_R as compared to those obtained by step changes in starch feed, screw speed, and MC. A positive step change in the PHAE content leads to a decrease in pressure, and *vice versa*. Thus, the values of K_R for this variable were negative. However, the controlled variable that PHAE content influenced maximum is the SME input to the process. The SME decreased with an increase in the PHAE content and *vice versa*. The value of the relative gain for this variable K_R was maximum near the set point and decreased as the magnitude of the step change increased.

Thus, from the aforementioned results, it can be seen that the temperature was the least affected output, while pressure and SME were the most affected outputs for the present screw profile and operating conditions. For extruder control, MC seemed to be the most influencing variable, followed by screw speed, the starch feed rate, and PHAE feed rate.

Linear domains

From the aforementioned relative steady state gains, it can be seen that the system was typically nonlinear. However, it was possible to determine the minimal domain around the central set-point, within which the process could be assumed linear. It is possible that the process could be controlled within this domain, using linear control algorithms. It was possible to identify a linear domain by plotting the absolute variation of the output variable *versus* the absolute variation of the input variable. Figures $4(a) - 4(d)$ show the variations in pressure with different step variations in the starch feed rate, screw speed, MC, and PHAE feed rate, respectively. The variations in torque and SME with step-changes in these manipulated variables were plotted likewise, and are not shown.

The absolute and relative linear domains for the various response variables to step-input variations in the manipulated variables are shown in Table V (entries 1–12). In the case of pressure as the response

No. Step change in PHAE feed
rate $(\%)$ rate (%) *KT K*RT *KP K*RP *K*torque *K*R,torque *K*SME *K*R,SME 1 -100.00 -1.3 -0.01 -84.5 -0.09 -0.14 -0.15 -0.06 -0.22 2 -50.00 0.00 -81.9 -0.09 -0.19 -0.21 -0.08 -0.28 3 -25.00 0.00 -66.6 -0.07 -0.25 -0.27 -0.09 -0.34 4 -10.00 0.00 -76.8 -0.08 -0.26 -0.28 -0.09 -0.34 5 $+10.00$ 0 0.00 -64.0 -0.07 -0.29 -0.32 -0.10 -0.38 6 $+25.00$ 0.00 -61.4 -0.07 -0.31 -0.33 -0.10 -0.39 $7 \qquad \qquad +50.00 \qquad \qquad 0 \qquad \qquad 0.00 \qquad \qquad -79.4 \qquad \qquad -0.09 \qquad \qquad -0.20 \qquad \qquad -0.22 \qquad \qquad -0.07 \qquad \qquad -0.27$ 8 $+100.00$ -1.3 -0.01 -93.4 -0.10 -0.15 -0.17 -0.06 -0.21

TABLE IV Steady State Process Gains for Step-Input Variations in the PHAB Feed Rate

Figure 4 Linearity in pressure with step-input variations in the (a) starch feed rate, (b) screw speed, (c) moisture content, and (d) PHAE feed rate.

variable (entries $1-4$, Table V), the MC possibly had the smallest linear domain of the system, and therefore, seemed to be the most destabilizing variable of the system. PHAE feed rate, on the other hand, was the least destabilizing variable. Under the present operating conditions, the starch feed rate and the screw speed provided equivalent relative linear domains. Mulvaney et al. 17 stimulated the food extrusion process by step tests on screw speed, MC, and feed rate and followed the process response through pressure records. They implicitly made the hypothesis that the process is linear.

For torque as the process response variable (entries 5– 8, Table V), MC and screw speed provided narrow linear domains of $\pm 10\%$ and $\pm 12.5\%$, respectively. However, with respect to the variations in starch and PHAE feed rate, the process variations in torque loading were linear in a relatively wider range of $\pm 25\%$. With SME as the response variable (entries 9 –12, Table V), the screw speed seemed to be the most destabilizing variable, as it governed the load (torque) on the extruder drive. The MC had significant influence too, as it affected the viscosity of the extrudate, which in turn governed the torque on the motor. Sanei¹⁸ ob-

TABLE V Absolute and Relative Linear Domains

No.	Process variables	Control variables	Absolute linear domain	Relative linear domain $(\%)$
2 3 4	Pressure	Starch feed rate Screw speed moisture content PHAE feed rate Starch feed rate	± 1.67 kg/h ± 25 rpm $\pm 1.74\%$ ± 0.39 kg/h	±15.00 ±12.50 ± 10.00 ±50.00
5	Torque	Starch feed rate	± 2.8 kg/h	±25.00
6		Screw speed moisture content	± 25 rpm	±12.50
7		PHAE feed rate	$\pm 1.74\%$	± 10.00
8		Starch feed rate	± 0.20 kg/h	± 25.00
9	SME	Starch feed rate	± 1.67 kg/h	±15.00
10		Screw speed moisture content	± 15 rpm	±7.50
11		PHAE feed rate	$\pm 1.74\%$	±10.00
12		Starch feed rate	± 0.39 kg/h	±50.00

Figure 5 Transient response of pressure to different step-input variations in the moisture content modeled as a first-order process with a dead time (lag). (a) $+10\%$ step change in the moisture content, (b) $+8\%$ step change in the moisture content, $(c) + 4\%$ step change in the moisture content, (d) original steady state (0% step change in the moisture content), (e) -4% step change in the moisture content, (f) -8% step change in the moisture content, and (g) -10% step change in the moisture content.

served the change in viscosity (with an online rheometer) as a consequence of variations of screw speed. The author concluded that the process is linear under the range of screw speeds tested. Moreira et al.¹⁹ used step tests on screw speed and MC to study the dynamical behavior of the extruder through pressure. They performed step tests of medium magnitude, and also back steps to return to the initial set point. They suggested that the process might be nonlinear.

As the temperature was not affected considerably, the linear domains were not determined. The variation in temperature was characterized by small jumps (± 1) to 2°C), and hence, no particular trend was observed. Onwulata et al. 20 and Lu et al. 21 undertook fairly complete studies by adding temperature as an input variable and by following the effect of step tests both on product variables (like expansion ratio) and on process variables (like melt pressure). The authors used step tests of great amplitude and did not pronounce on the linearity of the system.

Dynamic responses

Figure 5 shows the transient response of pressure to some step-input variations in the MC. These transient responses of pressure (*P*) and torque to step-input variations in the manipulated variables were modeled as first-order processes (process gain, *K*; time constant, τ) with a dead time, t_d (lag) (FODT Model). Many processes are higher than first order (contain more than one lag term), and so any first-order model will

only be an approximation. However, the approximation is sufficient in most cases and many of the proportional integral derivative (PID) tuning procedures use a FODT process model. Costin et al.²² critically reviewed dynamic modeling and control of plasticating extruders, and concluded that the gross dynamic input/output behavior may be generally described by simple first-/second-order models. Chan et al.²³ studied the pressure responses to screw speed changes as a first-order function times a lead–lag function to effectively model responses with an initial increase or decrease, followed by an exponential decay to the final steady state value. Equation (5a) represents a firstorder process with a dead time in the time domain, while eq. (5b) represents that process in the Laplace domain, using "*s*" as the Laplace operator.

$$
P(t) = K_P[1 - e^{-(t - t_d \sqrt{\tau})} A_X \tag{5a}
$$

$$
P(s) = \frac{K_p e^{-1_A}}{\tau S + 1} X(s)
$$
 (5b)

Where, K_p is the steady state process gain for pressure as the response variable; *t* is the time constant for the first-order process; t_d is the dead time (lag); *X* is the manipulated variable (starch feed rate/screw speed/ MC/PHAE feed rate); and A_x is the amplitude of the step-input change in the manipulated variable.

The steady state process gain, apparent time constant, and apparent dead time were determined

TABLE VI

graphically based on the procedure developed by Ziegler and Nichols. 30 Table VI provides the firstorder time constants and the dead times, in addition to the steady state process gains for the transient responses of pressure with step input variations in the starch feed rate, screw speed, MC, and PHAE feed rate, while Table VII provides the parameters for the transient responses of the torque loading. The process gains have been discussed earlier in this section. The data for characterization of the process were easily obtained in the laboratory, and subsequent modeling and determination of parameters were straightforward. These models would be suitable for dynamic simulation of the process using commercially available computer software, which would enable optimization of the process via simulation. The individual transfer functions could be combined to give a complete multivariable description of the starch foam extrusion process.

It was observed that the time constants and the dead times recorded for both the pressure and torque responses did not exhibit significant variation within each manipulated or control variable tested. Thus, the system displayed linear dynamic characteristics with respect to each manipulated variable. It was also ob-

served that for the same step-input variations in the manipulated variables, the torque loading on the twin-screw extruder exhibited a faster response (lower dead time), and also reached a steady state sooner (lower time constant). For example, for step-input variations in MC, the dead time in the pressure response was approximately 20 –22 s and the time constant was approximately 10 –13 s. However, the dead time in the torque response was approximately 11–13 s, while the time constant was approximately $8-10$ s.

Also, the response in pressure was fastest to stepinput variations in the MC, followed by the screw speed and the starch and PHAE feed rates. The response in torque loading was also fastest to step-input variations in the MC as well as the screw speed, followed by the starch and PHAE feed rates. Thus, the MC and screw speed seem to be the most destabilizing variables, as they induce rapid responses in the process variables.

Multiple input tests

The experimental design shown in Table VIII was implemented for the multiple input step tests. The experimental (measured) and calculated values (from

TABLE VII Dynamic Properties (Torque)

	Process	Time					
	steady state	constant	Dead time				
	gain K _{torque}	(τ, s)	(t_d, s)				
	Step changes in starch feed rate (%)						
$+5.00$	0.06	21	16				
$+15.00$	0.06	26	15				
$+20.00$	0.06	23	15				
-5.00	0.06	19	20				
-15.00	0.06	22	18				
-20.00	0.06	19	19				
	Step changes in screw speed (%)						
$+5.00$	-0.006	13	10				
$+10.00$	-0.005	12	12				
$+12.50$	-0.004	15	10				
-5.00	-0.005	9	11				
-10.00	-0.006	14	10				
-12.50	-0.005	12	8				
	Step changes in moisture content $(\%)$						
$+4.00$	-5.3	10	12				
$+8.00$	-5.0	11	10				
$+10.00$	-4.7	7	15				
-4.00	-5.6	11	12				
-8.00	-5.2	11	14				
-10.00	-5.2	9	13				
Step changes in PHAE feed rate (%)							
$+25.00$	-0.31	22	19				
$+50.00$	-0.21	26	16				
$+100.00$	-0.15	25	23				
-25.00	-0.25	16	20				
-50.00	-0.20	20	21				
-100.0	-0.14	32	22				

Starch feed rate $(\%)$	Screw speed $(^{\circ}\!\!/\!_0)$	Moisture content (%)	PHAE feed rate $(\%)$
$+10$	$+10$	$+10$	$+10$
$+10$	$+10$	$+10$	θ
$+10$	$+10$	$\overline{0}$	$+10$
$+10$	$\overline{0}$	$+10$	$+10$
θ	$+10$	$+10$	$+10$
$+10$	$+10$	θ	$\boldsymbol{0}$
$\overline{0}$	$+10$	$+10$	0
0	$\overline{0}$	$+10$	$+10$
$+10$	0	θ	$+10$
$+10$	0	$+10$	$\boldsymbol{0}$
θ	$+10$	$\mathbf{0}$	$+10$
-10	-10	-10	$^{-10}$
-10	-10	-10	θ
-10	-10	$\overline{0}$	-10
-10	$\boldsymbol{0}$	-10	-10
$\mathbf{0}$	-10	-10	-10
-10	-10	θ	$\boldsymbol{0}$
$\boldsymbol{0}$	-10	-10	θ
$\boldsymbol{0}$	$\overline{0}$	-10	-10
-10	0	θ	-10
-10	$\overline{0}$	-10	$\boldsymbol{0}$
$\overline{0}$	-10	0	-10

TABLE VIII Design for Multiple Input Tests

principle of superposition) of pressure, torque, and SME are shown in the Figures 6–8, respectively. The principle of superposition was fairly satisfied in the control variables, the error margin being low, especially in the case of the pressure readings. It was also observed that the pressure readings obtained from the experiment were always higher than those calculated by the principle of superposition. The validity of the principle of superposition in the aforementioned multiple input tests suggested that the process was sufficiently linear in the domain tested.

Figure 6 Measured (recorded) values of pressure compared to the calculated (from "principle of superposition") values for the different multiple step-input variation runs.

Figure 7 Measured (recorded) values of torque compared to the calculated (from "principle of superposition") values for the different multiple step-input variation runs.

A linear model was fit using STATEASE "Design of Experiments 6.0" modeling software with pressure (*P*), torque, and SME as the response data, and the starch feed rate, screw speed, MC, and PHAE feed rate as the factors (manipulated variables). The final model equations in terms of the coded factors "A" (starch feed rate), "B" (screw speed), "C" (MC), and "D" (PHAE feed rate) are shown in eqs. (6a)–(6c), while the final model equations in terms of the actual factors are shown in eqs. (7a)–(7c). Significance level was defined as $P < 0.05$.

$$
P = 738.03 + 54.45A - 70.55B - 73.56C - 6.05D
$$
\n
$$
\tag{6a}
$$

$$
Torque = 0.73 + 0.078A - 0.082B - 0.064C - 0.023D
$$
 (6b)

Figure 8 Measured (recorded) values of SME compared to the calculated (from "principle of superposition") values for the different multiple step-input variation runs.

nipulated variables.

$$
SME = 0.21 + 5.432E - 03A - 4.28E
$$

$$
- 03B - 0.018C - 9.655E - 03D
$$
 (6c)

$$
P = 1693.75 + 48.79 \text{ (start feed rate)}
$$

- 3.528 (screw speed) - 4215.37 (MC)
- 77.58 (PHAE feed rate) (7a)

\n
$$
\text{Torque} = 1648 + 0.0695 \, \text{(starch feed rate)} \\
- 4.115E - 03 \, \text{(screw speed)} - 3.684 \, \text{(MC)} \\
- 0.29 \, \text{(PHAE feed rate)} \, \text{(7b)}\n \end{array}
$$
\n

\n
$$
SME = 0.483 + 4.87E - 03
$$
 (start feed rate)
\n $- 2.14E - 04$ (screw speed) - 1.058 (MC)
\n $- 0.124$ (PHAE feed rate) (7c)\n

Figure 9(a) shows the response surface of pressure for the starch feed rate and screw speed as manipulated variables, while Figure 9(b) shows its response surface for the MC and PHAE feed rate as input variables. Thus, Figure 9 shows that variations in the MC, screw speed, and starch feed rate induced significant variations in pressure as compared to those due to step-changes in the PHAE feed rate. Figure 10 shows the comparison between the actual values of pressure obtained and the values from the linear model developed. The variance analysis for these data revealed a determination coefficient (R^2) of 0.9943 (*P* $<$ 0.0001). Similarly, the R^2 values for torque and SME were 0.9502 $(P < 0.0001)$ and 0.9533 $(P < 0.0001)$, respectively. Thus, multiple regression analyses showed a significant linear effect of the manipulated variables on the response variables. The coefficients of the manipulated (input) variables in eqs. (7a)–(7c) are similar to the values of the steady state process gains for the corresponding single step-input tests performed earlier, suggesting the linearity of the process within the domain tested.

The nonlinearity of the process was demonstrated by Cayot et al. 24 by the observation of the transient responses. Majority of the responses were reported to be of first order with a delay. Second-order responses were also recorded by Cayot et al.,²⁴ but no correlation was developed between the response and the manipulated variables.

CONCLUSIONS

The starch foam extrusion process was modeled as a **Figure 9** Response surface of pressure for the various ma- MIMO process, and the dynamics of the process were

Figure 10 Actual values of pressure obtained versus values from the linear model developed.

studied as a response to step changes in the input variables such as starch feed rate, screw speed, MC, and PHAE feed rate. The responses were modeled as firstorder responses with a time delay. The linearity of the process was determined over a range around the setpoint, and the parameters defining the first-order system such as gain "*K*," time constant "τ," and dead time "t_d" were determined in the linear range. The transfer function models can then be used in a predictive computer control system for on-line fine-tuning of the operating conditions. This could ensure a consistently high quality product even when low frequency disturbances are present in the system. It was observed that the time constants and the dead times recorded for both the pressure and torque responses did not exhibit significant variation within each manipulated or control variable tested. Thus, the system displayed linear dynamic characteristics with respect to each manipulated variable. It was also observed that for the same step-input variations in the manipulated variables, the torque loading on the twin-screw extruder exhibited a faster response (lower dead time), and also reached a steady state sooner (lower time constant). The response in pressure was fastest to step-input variations in the MC, followed by the screw speed and the starch and PHAE feed rates. The response in torque loading was also fastest to step-input variations in the MC as well as the screw speed, followed by the starch and PHAE feed rates. Thus, the MC and screw speed were the most destabilizing variables, as they induce rapid responses in the process variables. The MC in the extruder was, hence, determined to be the most influential factor in the stability of the process, followed by screw speed and starch feed rate. PHAE feed rate was the least significant variable.

Multiple step-input tests were carried out to determine the validity of the principle of superposition. The validity of the principle of superposition implied the linearity of the process in the domain tested. The hypothesis of linearity is an implicit and necessary prerequisite to most of the classical techniques of process control. A linear model was fit using STATEASE "Design of Experiments 6.0" modeling software, with pressure (*P*), torque, and SME as the response data, and the starch feed rate, screw speed, MC, and PHAE feed rate as the factors (manipulated variables). Multiple regression analyses showed a significant linear effect of the manipulated variables on the response variables.

References

- 1. Narayan, R. Kunststoffe 1989, 79, 1022.
- 2. Narayan, R. Am Chem Soc Symp Ser 1992, 476, 1.
- 3. Narayan, R. In Polymers from Agricultural Coproducts; Fishman, M. L., Friedman, R. B., Huang, S. J., Eds.; ACS Symposium Series 575; American Chemical Society: Washington, DC, 1994; p 2.
- 4. Narayan, R. In Paradigm for Successful Utilization of Renewable Resources; Sessa, D. J., Willett, J. L., Eds.; AOCS Press: Champaign, IL, 1998; p 78.
- 5. Harper, J. M. CRC Crit Rev Food Sci Nutr 1979, 11, 155.
- 6. Eerikainen, T.; Linko, P. In Extrusion Cooking; Mercier, C., Linko, P., Harper, J. M., Eds.; American Association of Cereal Chemists: St Paul, USA, 1989; p 157.
- 7. Janssen, L. P. B. M. In Food Engineering and Process Applications, Vol. II: Unit Operations; Le Maguer, M., Jelen, P., Eds.; Elsevier Applied Science: London, 1986; p 115.
- 8. Della Valle, G.; Tayeb, J. In Proceedings of Agoral, Dijon, April 16 –17, 1991.
- 9. Cayot, N. Doctoral Thesis, Université de Bourgogne, 1992.
- 10. Janssen, L. P. B. M. Twin Screw Extrusion; Elsevier/North Holland: New York, 1978.
- 11. Martelli, F. G. Twin Screw Extruders: A Basic Understanding; Van Nostrand Reinhold: New York.
- 12. El-Dash, A.; Gonzales, R.; Ciol, M. In Extrusion Cooking Technology; Jowitt, R., Ed.; Elsevier Applied Science: London, 1983; p 51.
- 13. Frazier, P. J.; Crawshaw, A.; Daniels, N. W. R.; Eggitt, P. W. R. In Extrusion Cooking Technology; Jowitt, R., Ed.; Elsevier Applied Science: London, 1983; p 1.
- 14. Olkku, J.; Hagqvist, A. In Extrusion Cooking Technology; Jowitt, R., Ed.; Elsevier Applied Science: London, 1983; p 27.
- 15. Owusu-Ansah, J.; Van de Voort, F. R.; Stanley, D. W. Can Inst Food Sci Technol J 1984, 17, 65.
- 16. Richburg, L. L.; Garcia, A., III. Presented at the Summer Meeting of the American Society of Agricultural Engineers, Rapid City, SD, June 26 –29, 1988.
- 17. Mulvaney, S. J.; Hsieh, F.; Onwulata, C.; Brent, J., Jr.; Huff, H. E. Presented at the International Winter Meeting of the American Society of Agricultural Engineers, Chicago, December 13–16, 1988.
- 18. Sanei, A. Doctoral-Engineer Thesis, Université de Technologie de Compiègne, 1990.
- 19. Moreira, R. G.; Srivastava, A. K.; Gerrish, J. B. Food Control 1990, 1, 179.
- 20. Onwulata, C. I.; Mulvaney, S. J.; Hsieh, F.; Heymann, H. J Food Sci 1992, 57, 512.
- 21. Lu, Q.; Hsieh, F.; Mulvaney, S. J.; Tan, J.; Huff, M. E. Lebensmittel Wissenschaft und Technologie 1992, 25, 261.
- 22. Costin, M. H.; Taylor, P. A.; Wright, J. D. Polym Eng Sci 1982, 22, 393.
- 23. Chan, D.; Nelson, R. W.; Lee, L. J. Polym Eng Sci 1986, 26, 152.
- 24. Cayot, N.; Bounie, D.; Baussart, H. J Food Eng 1995, 25, 245.
- 25. Nabar, Y. U.; Schindler, M.; Narayan, R. Polym Eng Sci, to appear.
- 26. Winkler, M. S.; Berry, T. S.; Kirkpatrick, D. E. U.S. Pat. 6,365,079 (2002).
- 27. Silvis, C. H.; White, J. E. U.S. Pat. 5,275,853 (1986).
- 28. Myers, R. H. In Response Surface Methodology; Allan and Bacon: Boston, 1971.
- 29. Chavez-Jauregui, R. N.; Silva, M. E. M. P.; Areas, J. A. G. J Food Sci 2000, 65, 1009.
- 30. Ziegler, J.; Nichols, N. Trans ASME 1942, 64, 759.