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R.P. SINGH  
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# MULTIRESPONSE OPTIMIZATION OF ACID CASEIN PRODUCTION

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## ABSTRACT

*A multivariate skimmilk extrusion process, designed to produce an acid co-precipitate was studied in terms of minimizing residual lactose, ash and fines. An experimental model system was utilized to simulate the extrusion process and evaluated using response surface methodology to assess the relationship between the responses (fines, residual lactose and minerals) and the process variables (concentration, pH, temperature, agitation, washing time and wash water ratio). Compromise optimum conditions were derived using the Generalized Distance Approach (GDA) and an Extended Response Surface Procedure (ERSP) which made use of the SAS RSREG procedure with and without constraints. The GDA procedure produced good results in terms of providing an optimum for a general acid casein process, while the ERSP allowed more extensive analysis of the data in terms of assessing specific processing conditions. Although more computing intensive, the ERSP conferred additional flexibility in determining optimal conditions for special situations such as extrusion processing. Both approaches are useful for process engineering, with the GDA being a more general tool while the ERSP is advantageous when the GDA procedure becomes limiting.*



## INTRODUCTION

The manufacture of acid casein from skimmilk powder (SMP) is a primary industrial process which serves to provide the base material for the further processing of milk proteins into soluble caseinates, mainly used by the food industry (Muller 1971; Richert 1975; Muller 1982; Southward 1985, Fichtali *et al.* 1989a). The overall quality of acid casein depends on the conditions associated with the coagulation and washing of the curd, with residual lactose and ash being important quality attributes, and the minimization of fines being a major economic consideration.

We have been studying extrusion processing as a continuous means of producing acid casein from SMP (Barraquio *et al.* 1988), however, the end product did not meet international standards. It was clear from this initial work that a more detailed investigation was required of the coagulation and washing procedures in relation to the extrusion process. These procedures involve a number of variables, including pH and concentration for the coagulation step, and temperature, time, agitation and wash water ratio (WWR) for the washing step. Such a complex multivariate system can only be studied effectively using Response Surface Methodology (RSM), an approach which has been enhanced by the availability of multiresponse optimization routines (Khuri and Conlon 1981), specifically the Generalized Distance Approach (GDA). This procedure was assessed in relation to obtaining a multivariate compromise optimum for the coagulation/washing process for acid casein in general and compared to an Extended Response Surface Procedure (ERSP) through the use of the SAS RSREG procedure with and without constraints as devised by Fichtali.

## MATERIAL AND METHODS

The details of the experimental protocol and analyses have been described elsewhere (Fichtali *et al.* 1989a,b). In essence skimmilk powder was coagulated under various conditions of pH and concentration (C), and subsequently washed under variable conditions of temperature (T), agitation (rpm), time (t) and wash water ratio (WWR). The casein fines were separated from the wash water by centrifugation and the product was freeze dried and analyzed for lactose and ash.

### Experimental Design and the Regression Model

A central composite design with three blocks (Box and Draper 1987) for six factors and with six center points was used for this study. The input variables considered in the experiment, their original values and coded levels are presented in Table 1. The design consisted of 50 treatment combinations, including 5 levels

TABLE 1.  
THE ORIGINAL AND CODED LEVELS OF THE INPUT VARIABLES FOR THE SKIMMILK  
COAGULATION AND WASHING PROCESS.

Variables		Coded Levels				
		-2	-1	0	1	2
pH	X1	4.1	4.3	4.5	4.7	4.9
Temperature	X2 (°C)	40	45	50	55	60
Concentration	X3 (%)	10	20	30	40	50
Time	X4 (min)	5	10	15	20	25
Mixer speed	X5 (rpm)	150	200	250	300	350
Wash water ratio	X6 (g/g)	3	4	5	6	7

for each variable which were fixed to allow the investigation of a wide range of experimental conditions within practical limits. Using the Statistical Analysis System (SAS) RSREG Procedure (SAS; Institute Inc. Cary, North Carolina), the 50 coded levels were regressed against each of the responses to obtain the coefficients for a set of three second-order regression equations which described the contribution of each input variable to the selected responses. The general form of a second-order regression model is:

$$y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i=1}^{k-1} \sum_{j=2}^k \beta_{ij} x_i x_j + \epsilon$$

Where  $x_1, x_2, \dots, x_k$  are the input variables which influence the response  $y$ ;  $\beta_0, \beta_i$  ( $i = 1, 2, \dots, k$ ),  $\beta_{ij}$  ( $i = 1, 2, \dots, k; j = 1, 2, \dots, k$ ) are unknown parameters, and  $\epsilon$  is random error.

### Generalized Distance Approach

Contour plots of individual responses can be used to optimize one response at a time against two selected independent variables. When two or more response variables are considered simultaneously, the optimization problem becomes substantially more complex and conditions which are optimal for one response may not be optimal for another, or may be physically impractical. In addition, the optimum may be a function of more than two independent variables. Optimization of the multiresponse function can be achieved using the Generalized Distance Approach (Khuri and Conlon 1981; Khuri and Cornell 1987). This method permits one to find compromise conditions for the input variables that are somewhat favourable to all responses. This implies that the multiresponse function deviates as little as possible from the individual optima and the deviation is

formulated as a distance function which is minimized over the experimental region. Mathematically, if the responses are considered to be correlated, this distance function can be expressed as:

$$\rho[\hat{y}(x), \phi] = \left[ \frac{(\hat{y}(x) - \phi)' \hat{\Sigma}^{-1} (\hat{y}(x) - \phi)}{z'(x) (Z'Z)^{-1} z(x)} \right]^{1/2}$$

where:

$x$  is defined as a  $1 \times k$  vector of the coded input variables

$z(x)$  is a vector of order  $p \times 1$  of the same form as a row of  $Z$ , but is evaluated at the point  $x$

$\hat{y}(x)$  is the vector of predicted  $r$  responses at the point  $x$

$\phi$  being the vector of individual optima

$\hat{\Sigma}$  is an estimate of the variance-covariance matrix of the responses given by:

$$\hat{\Sigma} = \frac{Y' [I_n - Z(Z'Z)^{-1}Z'] Y}{(n-p)}$$

where  $Y$  is the  $n \times r$  multiresponse data matrix.

It is to be noted that the matrix  $Z$  is such that all the responses are represented by the models:

$$y_i = Z\beta_i + \epsilon_i \quad i = 1, 2, \dots, r$$

and  $Z$  is assumed to be of order  $n \times p$  and rank  $p$ .

The minimization of the  $\rho$  function with respect to  $x$  over the experimental region will produce a compromise optimum for the multivariate system under consideration. The input data for the Multiple Response (MR) program developed by Conlon and Khuri (1988) are the design and response matrix, which are used to calculate the regression equations and the optima for each response. Once the individual optima have been obtained, the GDA is used to calculate the compromise optimum. A variable which can be adjusted in this search is the design radius (the extreme limits of the coded variables), which can be reduced to narrow the search. Since our design is not rotatable, the radius is a required parameter in the program. The details of the search method and convergence criteria used in the program have been discussed by Khuri and Conlon (1981).

### **Extended Response Surface Procedure (ERSP) for Multiresponse Optimization**

An alternative approach to finding a multiresponse optimum is to use an ERSP with constraints. Because of practical experimental considerations, the original experimental RSM design is restricted to 50 variable combinations. A search for an optimum under these conditions is limited because the grid comprising the variable combinations is quite crude and the optimum may be missed. By writing a small subroutine, one can generate a much finer grid using second order regression equations by reducing the interval between the coded variables (i.e., 0.25 instead of 1.00). The expanded data set considers all the variable combinations and an extensive new set of responses values is calculated. If the computing power is available, even more combinations can be generated by reducing the coded level interval to even smaller values to increase the precision of the search. The response of interest is then sorted in ascending order using a sort routine which is part of the RSREG procedure. The maximum or minimum of the sorted response can be determined with or without constraints on the remaining responses. In addition to this, the search in the design space can also be limited by constraining the input variables to selected ranges. Assuming the relations originally derived from the experimental data are sufficiently precise, this approach allows one to optimize a selected variable with constraints on either input variables or the remaining responses. The end result is an optimum with constraints, which may also be considered a type of multivariate response optimum.

## **RESULTS AND DISCUSSION**

The complete experimental design in terms of coded values and their respective responses is presented in Table 2. Table 3 presents the regression equations and their estimated regression coefficients obtained based on this data. All the coefficients of determination ( $R^2$ ) are significant at  $P < 0.01$  with only lactose having a coefficient accounting for less than 95% of the variation observed. Figure 1 illustrates in graphical form the type of problem to be solved. Here pH and concentration are the variables under consideration, with the remaining variables fixed and the responses are presented as overlapping contour plots. In this circumstance, we can visually estimate a multiresponse optimum in relation to fines, lactose content and ash. The shaded region in Fig. 1 can be considered the best conditions meeting the criteria of minimal fines, lactose and ash. Khuri's procedure uses a mathematical approach to find a more general solution, which is not restricted in terms of the number of variables under consideration, as in the case of the response surface plot.

TABLE 2.  
EXPERIMENTAL DESIGN AND THE MULTIRESPONSE EXPERIMENTAL DATA  
OBTAINED FOR SKIMMILK

$X_1$	$X_2$	$X_3$	$X_4$	$X_5$	$X_6$	$Y_1$	$Y_2$	$Y_3$
-1	-1	-1	-1	-1	-1	1.48	1.53	0.300
1	-1	-1	-1	1	-1	3.97	2.58	0.114
-1	1	-1	-1	1	-1	1.36	1.27	0.182
1	1	-1	-1	-1	-1	7.47	2.40	0.334
-1	-1	1	-1	-1	1	6.27	2.91	1.050
1	-1	1	-1	1	1	8.41	3.87	0.123
-1	1	1	-1	1	1	7.84	2.62	0.159
1	1	1	-1	-1	1	5.54	4.23	0.680
-1	-1	-1	1	-1	1	1.73	1.85	0.103
1	-1	-1	1	1	1	4.64	2.29	0.086
-1	1	-1	1	1	1	1.59	1.40	0.103
1	1	-1	1	-1	1	8.41	2.09	0.101
-1	-1	1	1	-1	-1	6.94	2.81	0.532
1	-1	1	1	1	-1	12.20	3.80	0.134
-1	1	1	1	1	-1	8.67	2.66	0.248
1	1	1	1	-1	-1	4.90	4.10	0.288
0	0	0	0	0	0	1.63	3.45	0.197
0	0	0	0	0	0	1.24	3.25	0.270
-1	-1	-1	-1	1	1	3.00	1.72	0.087
1	-1	-1	-1	-1	1	6.86	2.49	0.108
-1	1	-1	-1	-1	1	1.89	1.33	0.120
1	1	-1	-1	1	1	5.97	2.17	0.132
-1	-1	1	1	1	-1	9.37	2.57	0.888
1	-1	1	-1	-1	-1	7.72	4.52	0.433
-1	1	1	-1	-1	-1	3.30	2.50	0.352
1	1	1	-1	1	-1	10.50	3.89	0.559
-1	-1	-1	1	1	-1	2.80	1.57	0.172
1	-1	-1	1	-1	-1	1.75	1.51	0.097
-1	1	-1	1	-1	-1	1.75	1.51	0.157
1	1	-1	1	1	-1	6.30	2.11	0.126
-1	-1	1	1	1	1	12.40	1.94	0.134
1	-1	1	1	-1	1	11.60	4.06	0.136
-1	1	1	1	-1	1	6.39	2.43	0.207
1	1	1	1	1	1	9.16	3.66	0.122
0	0	0	0	0	0	1.22	3.10	0.236
0	0	0	0	0	0	1.26	3.25	0.234
-2	0	0	0	0	0	8.05	1.64	0.257
2	0	0	0	0	0	18.20	2.70	0.312
0	-2	0	0	0	0	1.24	3.10	0.179
0	2	0	0	0	0	1.38	3.12	0.242
0	0	-2	0	0	0	1.44	1.95	0.055
0	0	2	0	0	0	13.7	4.49	0.188
0	0	0	-2	0	0	1.34	3.39	0.548
0	0	0	2	0	0	1.56	2.71	0.087
0	0	0	0	-2	0	0.88	3.19	0.205
0	0	0	0	2	0	3.10	2.84	0.092
0	0	0	0	0	-2	1.18	3.45	0.473
0	0	0	0	0	2	1.82	3.06	0.153
0	0	0	0	0	0	1.41	3.29	0.227
0	0	0	0	0	0	1.35	3.27	0.230

$Y_1$  = Fines (%);  $Y_2$  = Ash (%);  $Y_3$  = Lactose (%)

TABLE 3.  
LEAST SQUARES FIT AND REGRESSION ESTIMATES FOR SKIMMILK

Parameters	Regression Coefficients		
	Y <sub>1</sub>	Y <sub>2</sub>	Y <sub>3</sub>
intercept	1.3010*	3.3439**	0.2235**
X <sub>1</sub>	1.6475**	0.4898**	-0.0266
X <sub>2</sub>	-0.4205	-0.0483	-0.0135
X <sub>3</sub>	2.1970**	0.6878**	0.1010**
X <sub>4</sub>	0.4435*	-0.0963*	-0.0962**
X <sub>5</sub>	0.5435*	-0.0793	-0.0477*
X <sub>6</sub>	0.1385	-0.0343	-0.0535*
X <sub>1</sub> X <sub>1</sub>	2.9788**	-0.3218**	0.0186
X <sub>2</sub> X <sub>2</sub>	0.0213	-0.0868	0.0001
X <sub>3</sub> X <sub>3</sub>	1.5875**	-0.0593	-0.0222
X <sub>4</sub> X <sub>4</sub>	0.0563	-0.1018*	0.0268
X <sub>5</sub> X <sub>5</sub>	0.1913	-0.1106*	-0.0154
X <sub>6</sub> X <sub>6</sub>	0.0688	-0.0506	0.0257
X <sub>1</sub> X <sub>2</sub>	0.1675	0.0122	0.0906**
X <sub>1</sub> X <sub>3</sub>	-0.8713**	0.1847**	-0.0315
X <sub>1</sub> X <sub>4</sub>	0.0538	-0.0603	0.0040
X <sub>1</sub> X <sub>5</sub>	-0.5413*	-0.0072	0.0033
X <sub>1</sub> X <sub>6</sub>	-0.2088	-0.0047	0.0099
X <sub>2</sub> X <sub>3</sub>	-0.6325*	0.0372	-0.0299
X <sub>2</sub> X <sub>4</sub>	-0.3338	0.0497	0.0155
X <sub>2</sub> X <sub>5</sub>	0.1925	0.0266	0.0114
X <sub>2</sub> X <sub>6</sub>	0.0263	-0.0134	0.0057
X <sub>3</sub> X <sub>4</sub>	0.2950	-0.0253	-0.0618*
X <sub>3</sub> X <sub>5</sub>	1.0813**	-0.0822	-0.0299
X <sub>3</sub> X <sub>6</sub>	0.1175	-0.0522	-0.0041
X <sub>4</sub> X <sub>5</sub>	-0.0775	-0.0009	0.0188
X <sub>4</sub> X <sub>6</sub>	0.0950	-0.0234	-0.0034
X <sub>5</sub> X <sub>6</sub>	-0.2675	-0.0303	-0.0481
R <sup>2</sup>	0.95**	0.96**	0.81**
RMSE	1.35	0.27	0.13

Y<sub>1</sub> = Fines(%); Y<sub>2</sub> = Ash (%); Y<sub>3</sub> = Lactose (%); RMSE = Root Mean Square Error  
\*Significant at p<0.05; \*\* Significant at p<0.01

For the calculations, the data set presented in Table 2 was entered into the Multiple Response program and a search implemented for the compromise minimum using design radii of 2.00 and 1.75. The output of the program provides optima for the factors investigated and computes the corresponding responses from them (Table 4). The compromise optima obtained for searches for both radii were similar and it is likely that the narrower search is slightly more accurate as more error can accumulate when extreme design values are used. The compromise optimum response conditions provide a very useful solution, defining six variables leading to a product which meets international specifications for acid casein in terms of lactose (<0.1%), ash (<2.0%), while minimizing fines, the latter being important from an economic viewpoint. This is clearly a valuable

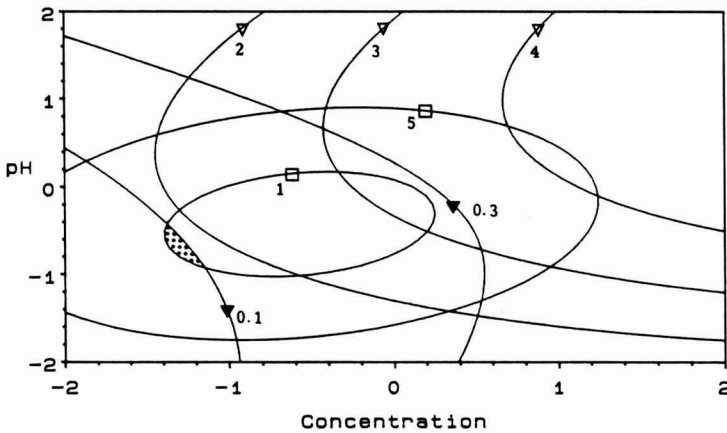


FIG. 1. SUPERIMPOSED MULTIPLE RESPONSE CONTOUR PLOT OF PREDICTED PERCENT LACTOSE ( $\blacktriangledown$ ), ASH ( $\nabla$ ) AND FINES ( $\square$ ) AS A FUNCTION OF pH AND CONCENTRATION  
 Temperature = 55°C; time = 15 min; rpm = 250; wash water ratio = 4.

solution in relation to defining and developing a process for the production of acid casein.

One can foresee circumstances where the compromise optimum may not provide a solution which meets specifications or where one may be forced to work outside these optimal values. Extrusion processing, which requires a solids content of 20% or more cannot be carried out using the calculated compromise optimum conditions. In this situation, the additional capability of adding constraints would be useful to allow a search for a solution which defines or limits specific criteria. Such an approach was devised by making use of an ERSP based

TABLE 4.  
 MULTIRESPONSE FACTOR OPTIMA CALCULATED USING THE GENERALIZED DISTANCE APPROACH AND THEIR SUBSEQUENT PREDICTED RESPONSES FOR TWO RADII ( $r$ )

Variables	Optimal Values	
	$r=2.00$	$r=1.75$
pH	4.33	4.34
Temperature	45°C	46°C
Concentration	12.3%	14.9%
Wash Time	14.4 min	15.8 min
rpm	263	259
water ratio	4.8	4.9
Responses	Optimal Values	
Fines	1.70%	1.11%
Ash	1.55%	1.76%
Lactose	-0.029%	0.011%

on the SAS RSREG procedure and a routine to generate a more detailed surface for the search. To do this, the program was first instructed to minimize fines but to simultaneously restrict lactose and ash to <0.05% and <1.5%, respectively. A variety of additional constraints were then imposed in relation to the extrusion process. Specifically, optimization was studied within the data set using the following criteria: Run 1, the use of a selected pH range; Runs 2–5, specific pH values; Runs 6–11, selected concentrations; and Run 12, a speciality high ash product.

Table 5 presents the constraints placed on the variables for runs 1–12 and the resulting optimal solutions. Thus, the solution for Run 1, which mimics extrusion

TABLE 5.  
MINIMIZATION OF FINES USING THE EXTENDED RESPONSE SURFACE PROCEDURE  
USING DEFINED CONSTRAINTS ON RESPONSES AND INPUT VARIABLES

Run#	Constraints					
	pH	T(°C)*	C(%)	t(min)	rpm	WWR
1	4.3-4.5	40-60	10-25	10-20	200-250	4-5
2	4.3-4.3	40-60	10-25	10-20	200-250	4-5
3	4.4-4.4	40-60	10-25	10-20	200-250	4-5
4	4.5-4.5	40-60	10-25	10-20	200-250	4-5
5	4.6-4.6	40-60	10-25	10-20	200-250	4-5
6	4.1-4.6	40-60	25-25	10-20	200-250	4-5
7	4.1-4.6	40-60	30-30	10-20	200-250	4-5
8	4.1-4.6	40-60	35-35	10-20	200-250	4-5
9	4.1-4.6	40-60	40-40	10-20	200-250	4-5
10	4.1-4.6	40-60	45-45	10-20	200-250	4-5
11	4.1-4.6	40-60	50-50	10-20	200-250	4-5
12	4.3-4.7	40-60	10-50	10-20	200-250	4-5

Run#	Solutions								
	pH	T	C	t	rpm	WWR	F(%)	A(%)	L(%)
1	4.32	59	22	20.0	200	4.0	-0.44	1.48	0.016
2	4.30	60	23	20.0	200	4.0	-0.43	1.42	0.003
3	4.40	60	18	20.0	205	4.0	0.31	1.46	0.050
4	4.50	59	15	16.5	200	4.6	3.26	1.48	0.050
5	4.60	40	11	16.5	250	4.0	4.07	1.49	-0.120
6	4.28	60	25	20.0	200	4.0	-0.32	1.47	0.005
7	4.22	60	30	20.0	200	4.0	1.17	1.44	0.005
8	4.18	60	35	20.0	200	4.0	3.51	1.43	0.013
9	4.16	60	40	20.0	200	4.0	6.20	1.50	0.024
10	4.14	60	45	20.0	200	4.4	10.23	1.50	0.001
11	4.12	60	50	19.5	200	4.0	14.37	1.50	0.037
12	4.35	60	45	20.0	200	4.7	4.01	3.11	0.047

\*Full range, F = Fines, A = Ash, L = Lactose



conditions, is slightly better than the compromise solution (Table 4) obtained using the Multiple Response program in terms of minimizing each response, but still allowing the use of higher SMP concentrations. For Runs 2–5, the effect of increasing pH forces a reduction of the concentration but also results in an increase in minimum fines. In the case of increasing concentration from 25 to 50% (Runs 6–11), the pH required decreases, however, minimum fines reach unacceptable levels. For the manufacture of a specialty high ash casein with low lactose content (Run 12), the key factor appears to be the use of high concentrations. The ERSP is a broader tool in that it allows one to explore the data set more fully and assess basic trends as a function of selected constraints.

An attempt was made to compare the two methods more directly by using a totally unconstrained input variables ERSP search with lactose and ash restricted to <0.05% and <1.5%, respectively. The results of the unconstrained input variables search are presented in Table 6 and can be compared to the compromise optima presented in Table 4. The solutions are quite different, with the ERSP results being at the limits of the design values for the majority of input variables. Most of the optima are impractical to implement, but do result in improved minimization of fines, lactose and ash. The ESRP solutions also indicate that the effect of the design interval between the coded levels appears to be negligible, but that the design radius does affect the results. These differences can readily be explained in terms of the search approach. In the Generalized Distance Approach, the compromise optimum depends on individual optima of the responses and how far they are from each other in the design space, and as a result, the

TABLE 6.  
COMPUTATION OF MINIMUM FINES WITH UNCONSTRAINED INPUT VARIABLES  
AND LACTOSE AND ASH UNDER CONSTRAINTS USING THE EXTENDED RESPONSE  
SURFACE PROCEDURE FOR TWO DESIGN RADII

(a) Radii = 2.00

I	N	pH	T	C	t	rpm	WWR	F(%)	A(%)	L(%)
1	15625	4.5	40	10.0	5	350	7	-3.6	1.10	-0.39
0.5	531441	4.5	40	15.0	5	350	7	-3.7	1.40	-0.25
0.25	24137569	4.45	40	12.5	5	350	7	-3.9	1.18	-0.28

(b) Radii = 1.75

1	4096	4.35	58.8	12.5	6.3	313	6.3	-1.6	1.34	-0.06
0.5	262144	4.45	58.8	12.5	6.3	338	6.8	-2.9	1.41	-0.24
0.25	11390625	4.45	58.8	12.5	6.3	338	6.8	-2.9	1.41	-0.24

I = Interval between coded levels; N = Number of predicted data points generated by SAS; F = Fines; A = Ash; L = Lactose.

compromise optimum may be far from the 'ideal' optimum (Khuri and Cornell 1987). Using ERSP, the search for minimum fines under the defined constraints will be closer to the optimum, which in turn can be further localized by increasing the number of data points in the design space.

The ERSP approach requires a large number of data points and considerable computing time, i.e., Run #1 consumed 40 min of CPU time (~ \$375/h). The GDA on the other hand can be run on an IBM PC at negligible cost on readily available hardware. The computing costs for the ERSP are high because the number of variables considered in this study were exceptionally large. In general, most problems of this nature consider between 2 to 4 variables, which would reduce computing time exponentially.

Both calculation procedures have merit, the GDA being useful for a generalized compromise optimum, while the ERSP is useful in specific situations where constraints are a necessity, as in the case of extrusion. The GDA optimum has been shown to be a very workable solution for the conventional acid casein process which is run on a relatively low solids basis. Extrusion processing requires high solids and hence optimal conditions can only be found using constraints. This ability to set constraints is certainly an advantage in such circumstances, providing the flexibility to explore the experimental data in relation to producing a tailor made end product.

## CONCLUSION

An attempt has been made to extend the basic information available from standard multivariate response surface data in terms of optimizing a skimmilk coagulation/washing process. It has been shown that the GDA program developed by Conlon and Khuri (1988) could be applied to obtain a useful compromise optimum for a standard acid casein process. The GDA result may in fact not provide a viable compromise optimum and in such circumstances a useful alternate solution can be obtained by an ERSP search as long as the models under consideration are second order. ERSP presents a useful way of deriving additional information from the experimental data set, especially where constraints are likely to be a prerequisite. Both procedures are useful tools to the process engineer and should be considered as adjuncts to standard RSM techniques.

## ACKNOWLEDGMENTS

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# **FUZZY EXPERT SYSTEMS: A PROTOTYPE FOR CONTROL OF CORN BREAKAGE DURING DRYING**

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## **ABSTRACT**

*A prototype fuzzy expert system was developed to predict the breakage susceptibility of corn for certain drying conditions and to provide recommendations for dryer control. The control decisions were based on predicted levels of breakage, drying conditions, and the recommendations of corn drying experts. Drying temperature, initial moisture content, equilibrium moisture content, and level of the wet product holding bin were used as the process information for providing recommendations for drying temperature and flow rate adjustment. Two example drying processes were tested. The results of the study showed that (1) the predicted corn breakage levels matched the experimental results and (2) the control recommendations by the system were consistent with domain experts.*

## **INTRODUCTION**

An expert system can be defined as a computer system which can mimic a human expert's heuristic and thought processes to solve complex problems. It relies both on data input and on an expert's knowledge and heuristics (Zimmermann 1987). An expert system is typically made up of three parts: a knowledge base, a working memory, and an inference engine. The knowledge base contains the domain expert's knowledge for use in problem solving. The working memory is used to store information. The inference engine uses the domain knowledge together with acquired information to provide an expert solution. In terms of the method of knowledge representation, expert systems can be divided as rule-based systems and frame-based systems. The rule-based system is ideal for representation of surface knowledge which would become conceptually very complex as the knowledge goes deeper. A frame-based system is more suitable

to represent deep knowledge which usually represents expertise and heuristics (Doheny and Monaghan 1987). When an expert system applies fuzzy relations, either parallel or sequential to the production rules, this expert system is defined as a fuzzy expert system (Buckley and Tucker 1987). Suitable applications for expert systems are: interpreting and identifying, predicting, diagnosing, designing, planning, monitoring, debugging, testing, instructing or training, and controlling (Wolfgram, *et al.* 1987).

Drying temperature has a significant effect on corn quality (Brooker *et al.* 1974). High temperature, low relative humidity drying combined with a high initial moisture content will lead to high levels of breakage susceptibility and stress cracks. To control levels of breakage susceptibility and stress cracks during drying processes, it is necessary to determine the influence of the controllable parameters, the flow rate through the dryer and drying temperature, on corn quality and to utilize experts' experience and heuristics for adjusting the operation of a dryer. Since there are many factors such as weather, corn variety, etc. that will also affect corn quality, the influences of the controllable parameters have some degree of uncertainty. A fuzzy expert system is specially suitable for dealing with such uncertainty and imprecision.

The prototype fuzzy expert system developed in this study was a combination of predicting corn breakage susceptibility, diagnosing the principal causes of breakage, and identifying the most suitable dryer adjustments. This system would be able to predict levels of breakage susceptibility under particular drying conditions and provide recommendations for adjusting the operation of the dryer.

## OBJECTIVES

The objectives of this study were: (1) to develop a fuzzy comprehensive evaluation model for determining the influence of drying conditions on the level of breakage susceptibility of dried corn, (2) to develop a fuzzy expert system to be used by operators of cross-flow grain dryers, and (3) to use corn breakage susceptibility control as an example to show how a fuzzy expert system can be applied in food process engineering.

## MODEL OF FUZZY COMPREHENSIVE EVALUATION

Corn breakage susceptibility is the potential for kernal fragmentation or breakage when subjected to impact forces during handling or transport, and there are laboratory procedures to measure breakage susceptibility (AACC 55-20, 1983). The influence of drying conditions on the breakage susceptibility of dried corn is not easily modeled because there are many other uncertain related effects. It is very difficult to predict such influences by applying common mathematical

tools. Fuzzy sets theory is a useful tool to describe ambiguous phenomena, search fuzzy relationships, and represent uncertain influences (He 1983). Such characteristics allow prediction of the level of breakage susceptibility of dried corn under important but uncertain influences by applying the model of fuzzy comprehensive evaluation.

Chen *et al.* (1983) developed a fuzzy model of multifactorial comprehensive evaluation. This fuzzy model consists of three key elements, namely a factor  $U$ , a grading set  $V$ , and the single factor grading.

$$U = \{u_1, u_2, \dots, u_n\} \quad (1)$$

$$V = \{v_1, v_2, \dots, v_m\} \quad (2)$$

The single factor grading is a fuzzy image  $f$  from the factor set  $U$  to the grading set  $V$ :  $U \rightarrow V$ . For a certain single factor  $u \in U$ , there exist a fuzzy grading  $B(u) \in F(V)$ . The fuzzy image  $f$  represents a fuzzy relation  $R_f$ . It can be represented as a fuzzy membership matrix  $R \in \mu_{n \times m}$ .  $R$  is a fuzzy transformation from  $U$  to  $V$ . For a comprehensive evaluation problem with  $n$  factors and  $m$  grading levels, the fuzzy membership matrix  $R$  can be written as:

$$R = \begin{matrix} r_{11} & r_{12} & \dots & r_{1m} \\ r_{21} & r_{22} & \dots & r_{2m} \\ \dots & \dots & \dots & \dots \\ \dots & \dots & \dots & \dots \\ r_{n1} & r_{n2} & \dots & r_{nm} \end{matrix} \quad (3)$$

The set  $(U, V, R)$  forms the model of fuzzy comprehensive evaluation. Given a fuzzy subset  $X \in U$  which contains the weighting factors assigned to each evaluating factor concerned, the model will provide comprehensive grades  $y_j \in Y$  for each quality factor by finding the maximum value of minimum fuzzy memberships of each predefined quality level in  $R$ :

$$Y = X \circ R \quad (4)$$

where

$$y_j = \bigvee_{i=1}^m (x_i \wedge r_{ij}) \quad (5)$$

The weighting factors in subset  $X$  are not defined arbitrarily, but by expertise, knowledge, experience, experimental results, and other related information.

In this study, a fuzzy grade statistical method (Li 1988) was applied to determine weighting factors and fuzzy memberships. To perform the fuzzy com-

prehensive evaluation, both drying conditions and predicted quality factors were divided into some pre-defined ranges. The ratio of the number of samples distributed within a defined range of drying conditions to the total number of samples is defined as the weighting factor. The ratio of the number of samples distributed within a certain level of breakage susceptibility at a particular range of drying conditions to the number of samples distributed within that range of drying conditions is defined as the fuzzy membership.

The weighting factor of a certain range of a drying factor was

$$M_{ij} = \frac{\sum n_{ij}}{\sum N} \quad (6)$$

The fuzzy membership function of a certain range of a particular drying variable on the breakage level of dried corn was

$$\mu_{Rijk} = \frac{n_{ijk}}{\sum n_{ij}} \quad (7)$$

We applied 4 sets experimental data on corn breakage susceptibility from a laboratory study (Weller 1987). Three of the data sets were used to determine weighting factors and fuzzy memberships for the fuzzy comprehensive evaluation model. The fourth set of the data was used to evaluate of the model.

In this study, three drying process variables, drying temperature, product initial moisture content, and equilibrium moisture content for the conditions created by the dryer, were selected as factors for evaluation. The breakage susceptibility and drying variables were all divided into three levels of high, moderate, and low. The levels were defined as in Table 1. For more precise prediction or a broader range of conditions, more levels can be defined.

TABLE 1.  
CLASSIFICATION OF BREAKAGE SUSCEPTIBILITY AND DRYING CONDITIONS

Breakage Susceptibility and Drying Conditions	Defined Levels		
	Low	Moderate	High
Breakage Susceptibility (%)	< 4.0	4.0 to 8.0	> 8.0
Temperature (°C)	< 45	45 to 70	> 70
Initial MC (%wb)	< 20	20 to 26	> 26
Equilibrium MC (%wb)	< 13	13 to 14	> 14

Figures 1-3 show sample distributions within the defined levels of breakage susceptibility for dried corn for the three drying variables. As defined above, weighting factors and fuzzy memberships for different ranges of different drying variables could be easily obtained from these sample distributions. As an example, the number of samples within the moderate range of equilibrium moisture content was 23 and the total number of samples was 36 (Fig. 3). The weighting factor for the moderate equilibrium moisture content, therefore, was

$$M_{e2} = 23/36 = 0.64 \tag{8}$$

The fuzzy memberships for high, moderate, and low levels of breakage susceptibility under the moderate range of equilibrium moisture content were

$$\begin{aligned} \mu_{Rem1} &= 8/23 = 0.35 \\ \mu_{Rem2} &= 14/23 = 0.61 \\ \mu_{Rem3} &= 1/23 = 0.04 \end{aligned} \tag{9}$$

Table 2 shows weighting factors and fuzzy memberships for each range of breakage susceptibility for each drying variable.

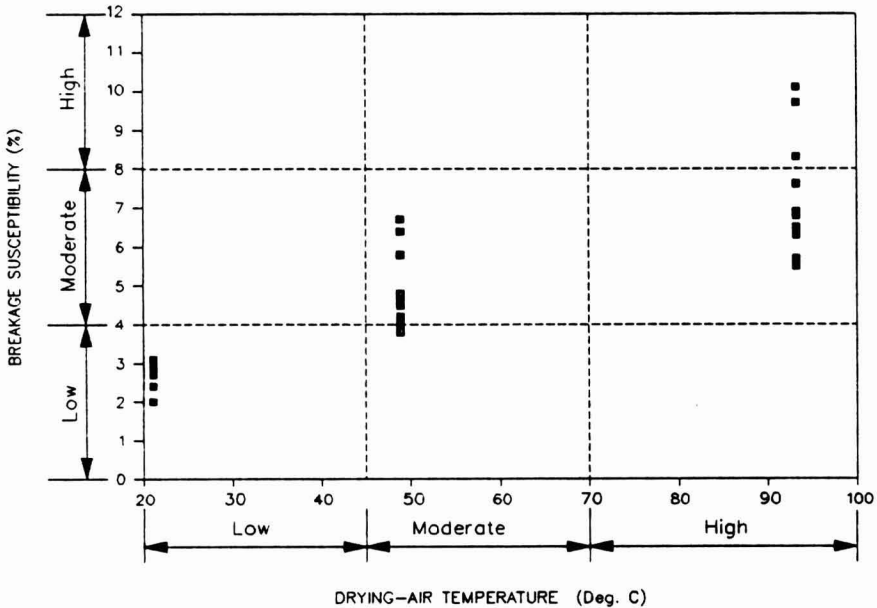


FIG. 1. DISTRIBUTION OF DRIED CORN SAMPLES FOR LOW, MODERATE, AND HIGH BREAKAGE SUSCEPTIBILITY AND FOR LOW, MODERATE, AND HIGH-DRYING TEMPERATURE (from Weller 1987)



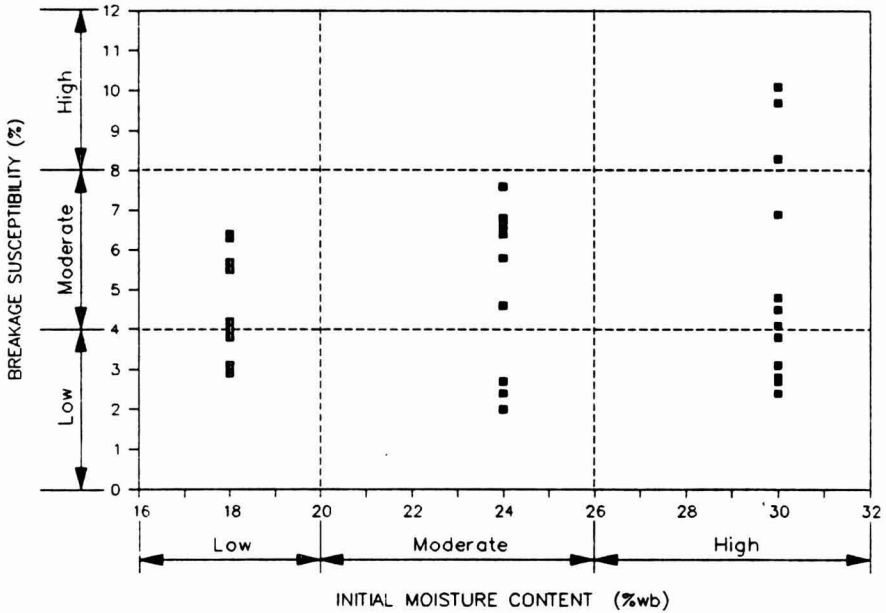


FIG. 2. DISTRIBUTIONS OF DRIED CORN SAMPLES FOR LOW, MODERATE, AND HIGH BREAKAGE SUSCEPTIBILITY AND FOR LOW, MODERATE, AND HIGH INITIAL MOISTURE CONTENT (from Weller 1987)

After weighting factors and fuzzy memberships were determined, levels of breakage could be predicted on the basis of drying conditions. For example, when the drying temperature was 48.9 °C, the initial moisture content was 30.0 % w.b. and the equilibrium moisture content was 14.0 % w.b., the process was classified as moderate drying temperature, high initial moisture content, and moderate equilibrium moisture content. From Table 2, the weighting factor was 0.33 for drying temperature, 0.33 for initial moisture content, and 0.64 for equilibrium moisture content. The fuzzy weighting factor vector was

$$X = (0.33 \quad 0.33 \quad 0.64) \tag{10}$$

Like the weighting factor vector of drying variables, the corresponding fuzzy memberships for the fuzzy prediction of breakage levels under given drying conditions could be obtained from Table 2. The fuzzy membership matrix for this example was

$$R = \begin{bmatrix} 0 & 0.75 & 0.25 \\ 0.42 & 0.58 & 0 \\ 0.35 & 0.61 & 0.04 \end{bmatrix} \tag{11}$$

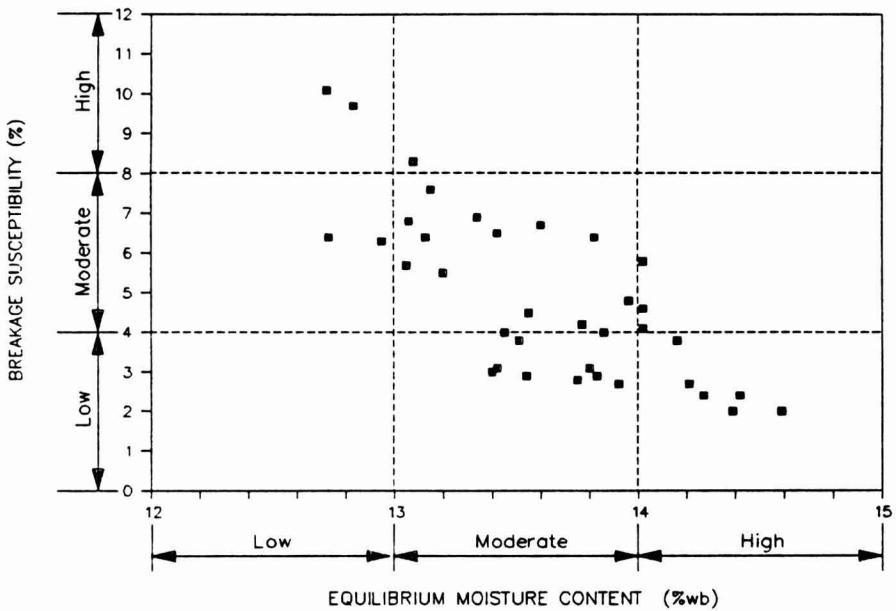


FIG. 3. DISTRIBUTIONS OF DRIED CORN SAMPLES FOR LOW, MODERATE, AND HIGH BREAKAGE SUSCEPTIBILITY AND FOR LOW, MODERATE, AND HIGH EQUILIBRIUM MOISTURE CONTENT (from Weller, 1987)

Degrees of likelihood for different levels of breakage susceptibility of dried corn were determined by a process of fuzzy comprehensive evaluation. In this example, the vector of fuzzy grade,  $Y$ , was predicted through a fuzzy transformation:

$$\begin{aligned}
 Y &= X \circ R \\
 &= (0.33 \quad 0.33 \quad 0.64) \circ \begin{bmatrix} 0 & 0.75 & 0.25 \\ 0.42 & 0.58 & 0 \\ 0.35 & 0.61 & 0.04 \end{bmatrix} \\
 &= \{(0 \vee 0.33 \vee 0.35) (0.33 \vee 0.33 \vee 0.61) (0.25 \vee 0 \vee 0.04)\} \\
 &= \{0.35 \quad 0.61 \quad 0.25\}.
 \end{aligned}$$

Normalizing the result, the fuzzy grade vector was:

$$Y = \{0.29 \quad 0.50 \quad 0.21\} \quad (12)$$

Values in the normalized  $Y$  vector showed that the likelihood was 0.29 for high level, 0.50 for moderate level, and 0.21 for low level of breakage suscep-

TABLE 2.  
WEIGHTING FACTORS OF DRYING CONDITIONS AND FUZZY MEMBERSHIPS OF  
DRYING CONDITIONS FOR CORN BREAKAGE SUSCEPTIBILITY

Drying Condition	Range of Condition	Weighting Factor of Condition Range	Fuzzy Membership of Condition to Breakage Susceptibility
Drying Temperature	High	$M_{11} = 0.33$	$\mu_{Rt11} = 1.00$ $\mu_{Rt12} = 0$ $\mu_{Rt13} = 0$
	Moderate	$M_{12} = 0.33$	$\mu_{Rt21} = 0.17$ $\mu_{Rt22} = 0.83$ $\mu_{Rt23} = 0$
	Low	$M_{13} = 0.33$	$\mu_{Rt31} = 0$ $\mu_{Rt32} = 0.75$ $\mu_{Rt33} = 0.25$
Initial Moisture Content	High	$M_{11} = 0.33$	$\mu_{Ri11} = 0.42$ $\mu_{Ri12} = 0.58$ $\mu_{Ri13} = 0$
	Moderate	$M_{12} = 0.33$	$\mu_{Ri21} = 0.25$ $\mu_{Ri22} = 0.75$ $\mu_{Ri23} = 0$
	Low	$M_{13} = 0.33$	$\mu_{Ri31} = 0.42$ $\mu_{Ri32} = 0.33$ $\mu_{Ri33} = 0.25$
Equilibrium Moisture Content	High	$M_{e1} = 0.11$	$\mu_{Re11} = 0$ $\mu_{Re12} = 0.50$ $\mu_{Re13} = 0.50$
	Moderate	$M_{e2} = 0.64$	$\mu_{Re21} = 0.35$ $\mu_{Re22} = 0.61$ $\mu_{Re23} = 0.04$
	Low	$M_{e3} = 0.25$	$\mu_{Re31} = 0.67$ $\mu_{Re32} = 0.33$ $\mu_{Re33} = 0$

tibility of dried corn. The moderate level of breakage susceptibility had the highest likelihood for the evaluated drying condition. So the drying process would be most likely to result in a moderate level of breakage susceptibility. The experimental result was a breakage susceptibility of 4.8% for this process (Weller 1987), which confirmed the prediction.

Similar to the example discussed above, Table 3 lists both the prediction and measured levels of dried corn breakage susceptibility for 12 sample conditions.

TABLE 3.  
EVALUATION OF BREAKAGE SUSCEPTIBILITY OF DRIED CORN  
(prediction and measured levels)

Drying Temp. (°C)	Initial MC (%wb)	Equilibrium MC (%wb)	Prediction Result	Actual Breakage	Prediction Matches?
71.1	18.0	13.59	moderate	5.3	Yes
71.1	18.0	13.32	moderate	5.7	Yes
71.1	18.0	13.50	moderate	4.9	Yes
71.1	18.0	13.33	moderate	5.9	Yes
71.1	24.0	13.29	moderate	7.9	Yes
71.1	24.0	13.16	moderate	7.7	Yes
71.1	24.0	13.67	moderate	6.5	Yes
71.1	24.0	13.49	moderate	6.7	Yes
71.1	30.0	13.39	moderate	7.0	Yes
71.1	30.0	13.86	moderate	5.7	Yes
71.1	30.0	13.83	moderate	5.5	Yes
71.1	30.0	13.81	moderate	5.2	Yes

All prediction matched experimental results. The model of fuzzy comprehensive evaluation on corn breakage susceptibility, therefore, was reliable for these conditions. The model was used in the fuzzy expert system to perform fuzzy reasoning for determining the predicted level of breakage susceptibility with the highest likelihood.

### THE PROTOTYPE FUZZY EXPERT SYSTEM

The fuzzy expert system was developed with a shell of Intelligence/Compiler (Intelligence Ware, Inc. 1987). The shell can support forward chaining, backward chaining, and inexact reasoning rules. Forward chaining reasons from known facts to the resulting conclusions. Backward chaining reasons from known conclusions to the facts that caused them. Inexact reasoning checks the degree of confidence for each rule and can be applied in both forward chaining and backward chaining. This fuzzy expert system applied an indirect fuzzy reasoning inference by using inexact inference to find the certainty value of each statement and then used backward chaining inference to evaluate product rules.

After levels of breakage susceptibility of dried corn were predicted by the fuzzy relations between levels of breakage susceptibility and ranges of drying variables, the system provided a recommendation with the highest confidence factor for adjusting drying processes by imitating experts' heuristics.

There were two types of fact bases used in this system. One was a fact base of fuzzy relations which contained fuzzy memberships of high, moderate and low levels of breakage susceptibility of dried corn to various ranges of drying

variables. It also contained weighting factors of different ranges of drying variables. Both fuzzy memberships and weighting factors were determined prior to the development of the system. The other fact base was the working memory of the fuzzy expert system. It contained the possible decisions the system could make including grades of breakage susceptibility of dried corn and recommendations for adjusting drying processes.

The knowledge base of the system contained rules which were used: (1) to determine the likelihood of levels of corn breakage susceptibility for various ranges of different drying variables, and (2) to find the degree of confidence of recommendations for adjusting the drying process. The knowledge base also contained logic-based rules for classifying the value of each drying variable.

The top level goal of this fuzzy expert system was to provide advice for adjustment of drying processes. It was supported by classifying the range of drying variables, determining the fuzzy relationship between drying variables and corn breakage levels, and searching for suitable recommendations for dryer control. Figure 4 shows the structure of the decision making processes of the system.

The input information to the system were (1) drying variables and (2) the level of wet corn in a predrying holding bin. The level of corn in the wet-holding bin was an important practical consideration that was used to determine whether the drying rate could be decreased or should be increased. For an example analysis, the system was restricted to a drying temperature range less than 100°C, corn initial moisture content higher than 15.5% w.b., and equilibrium moisture content lower than 15.5% w.b. The drying variables were divided into ranges of high, moderate, and low as defined in the preceding section. The level of wet corn in a wet-holding bin was also defined as full, medium, and low.

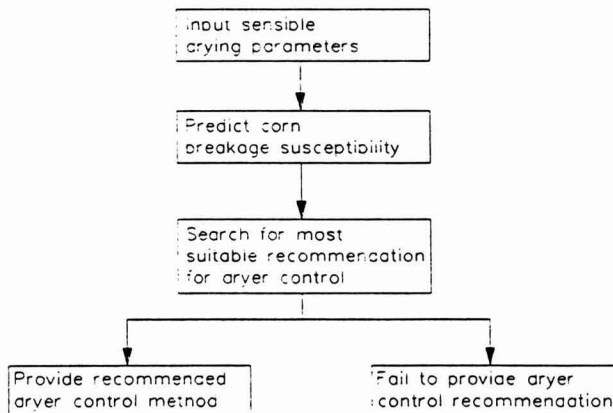


FIG. 4. STRUCTURE OF THE DECISION PROCESSES OF THE FUZZY EXPERT SYSTEM

The prediction of the breakage susceptibility utilized a fuzzy reasoning process. The structure of the predicting process is shown in Fig. 5. The confidence factor in the inexact inference played the same function as the membership in fuzzy reasoning processes.

Because fuzzy memberships varied with drying conditions, the fuzzy reasoning inference acquired appropriate memberships and weighting factors based on pre-defined ranges of drying conditions and represented them in the inexact reasoning reference as confidence factors. It also determined the likelihood of levels of corn breakage susceptibility by means of selecting the highest confidence factor. Since the fuzzy reasoning of the system was performed by combining inexact and exact reasoning, the confidence of the fuzzy reasoning was represented by the confidence factor of inexact reasoning.

A two-step process was applied to avoid unnecessary evaluations. The first step was to select appropriate fuzzy relationships for drying variables. The second step was to assign memberships and weighting factors to confidence factors of corresponding premises and then to determine the likelihood of each level of breakage susceptibility.

The control recommendations for drying were based on dryer operation expertise. This expertise would provide dryer control recommendations on the basis of either: (1) drying-rate-first strategy, or (2) energy-saving-first strategy. With the drying-rate-first strategy, the system would provide dryer control recommendations for raising drying rate as much as possible with a restriction of producing a low or moderate level of breakage susceptibility of dried corn. With the energy-saving-first strategy, the system would provide dryer control recommendations for reducing energy consumption as much as possible. In general, whether to choose a drying-rate-first strategy depended on the level of wet corn in the wet-holding bin.

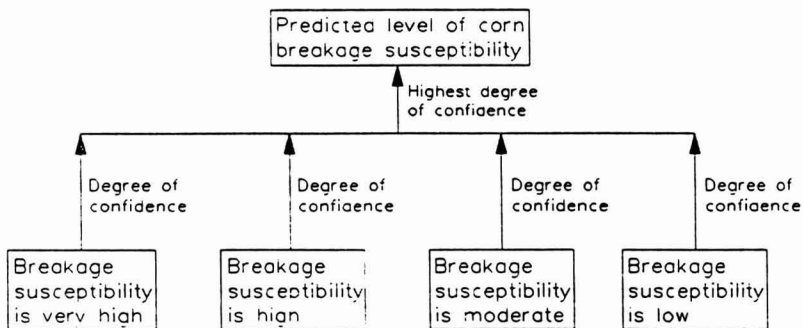


FIG. 5. STRUCTURE OF THE PROCESS FOR PREDICTING THE LEVEL OF CORN BREAKAGE SUSCEPTIBILITY

The fuzzy expert system applied inexact reasoning inference to determine confidence factors of all recommendations for dryer control. Usually, several of the recommendations were feasible for a particular drying process. The one with the highest value of confidence was selected as the system recommended dryer control adjustment. Figure 6 shows the process for making a control recommendation.

## EVALUATION OF THE PROTOTYPE FUZZY EXPERT SYSTEM

To test the fuzzy expert system, human experts (the authors) provided their recommendations for dryer control based on the predicted level of corn breakage and the level of the wet-holding bin. Then, the same information was input to the computer to check whether the recommendations provided by the system matched the human experts' recommendations.

Two hypothetical corn drying processes were tested in this evaluation procedure. One of them had a drying temperature of 48.9°C, corn initial moisture content of 30.0%, equilibrium moisture content of 14.0%, and level in wet corn holding bin of full. The second process had a drying temperature of 71.1°C, corn initial moisture of 18.0%, equilibrium moisture content of 13.6%, and level in wet corn holding bin of medium.

The system predicted that both drying processes would have a moderate level of breakage susceptibility (Table 4) which matched the experimental results obtained by Weller (1987). The recommendations for process adjustments pro-

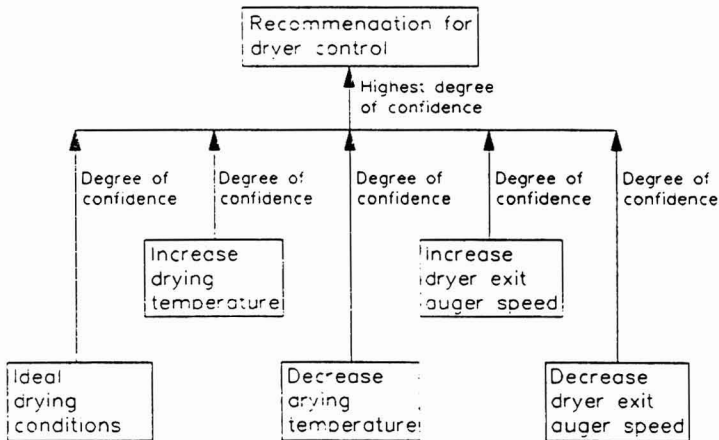


FIG. 6. STRUCTURE OF THE PROCESS FOR DETERMINING A DRYER CONTROL RECOMMENDATION

TABLE 4.  
COMPARISON OF FUZZY EXPERT SYSTEM OUTPUTS TO EXPERIMENTAL DATA  
AND TO THE AUTHORS' EXPERTISE

Drying condition	Corn breakage level		Recommended drying process adjusting	
	Predict	Measured	Expert system	Human experts
A	Moderate	4.8 %	keep the current drying conditions	keep the current drying conditions
B	Moderate	5.3 %	lower product flow rate & temperature	lower product flow rate & temperature

Note: Drying condition A: drying temperature = 48.9 °C, initial moisture content = 30.0 %, equilibrium moisture content = 13.9 %, wet corn level in bin = high.

Drying condition B: drying temperature = 71.1 °C, initial moisture content = 18.0 %, equilibrium moisture content = 13.6 %, wet corn level in bin = medium.

vided by the system were the same as what the human experts provided. Table 4 compares both the predicted and experimental levels of the breakage susceptibility as well as recommendations for process adjustments given by the fuzzy expert system and by the human's expertise. For the hypothetical process were drying condition A, both the expert system and the human experts recommended keeping the drying temperature and the product flow rate at the current levels. The confidence factor for the expert system recommendation was 0.85. For the hypothetical process with drying condition B, the expert system provided a recommendation of reducing the drying temperature and the flow rate by one control level. The confidence factor was 0.89. It matched the human experts' recommendation. The fuzzy expert system performed accurately for these two example processes.

After the knowledge base has been developed and tested, it was compiled as a stand-alone fuzzy expert system by the shell of Intelligence/Compiler. The stand-alone system might be used by dryer operators for (1) predicting levels of breakage susceptibility of dried corn and (2) determining an expert-system recommended-best process adjustment for particular drying situations.

By combining the exact and inexact reasoning inferences, the shell of Intelligence/Compiler can support fuzzy reasoning satisfactorily. Although the knowledge acquisition and representation techniques available are adequate, this shell cannot provide the conclusion of a reasoning process at early stages of execution as supporting knowledge for later reasoning stages. It requires much more computer time to execute the fuzzy reasoning process.



## CONCLUSIONS AND RECOMMENDATIONS

This work developed a fuzzy expert system to control the breakage susceptibility of dried corn during drying processes. Feasibility of such a fuzzy expert system was demonstrated for (1) predicting breakage susceptibility levels of dried corn in terms of drying variables and (2) providing a recommendation with the highest confidence value for dryer control to reduce corn breakage susceptibility.

The fuzzy expert system predicted levels of breakage susceptibility of dried corn for various drying conditions. The workable range of drying variables were divided into levels of high, moderate, and low. The predictions agreed well with experimental results. Therefore, this type of fuzzy expert system might be used to help dryer operators to adjust drying processes.

To obtain more accurate evaluations and to provide more precise information for control strategy analyses, more levels of quality factors and drying variables should be defined.

The fuzzy relations between drying conditions and corn breakage susceptibility can only be determined on the basis of both experimental results and humans' expertise. It will be helpful to develop a combined rule-based and frame-based system to represent the deep knowledge. The rule-based files contain the fuzzy relationships between drying conditions and corn quality factors, and the frame-based files represent the expert's expertise and heuristics.

This study applied laboratory test data. Since each particular type of commercial dryer has its own influence on dried corn quality, a practical system should be developed for each type of commercial dryer.

In addition to breakage susceptibility, drying processes can also affect stress cracking, kernel discoloration, starch separation, oil recovery, and protein quality. This fuzzy expert system could be extended to include more quality factors. The dryer control strategy, therefore, could be recommended on the basis of the drying conditions as well as the various quality factors required for a particular end use.

Finally, other food processing operations might benefit from this approach since quality control for food products during processing is based on both objective information such as process variables and subjective information such as human expertise and experience.

## NOMENCLATURE AND SYMBOLS

- U = factor set
- X = weighting factor vector
- x = weighting factor for factor i

- $V$  = grade set  
 $Y$  = comprehensive grade vector  
 $y_i$  = likelihood for grade  $i$   
 $R$  = fuzzy membership matrix  
 $R_i$  = fuzzy membership vector  
 $r_{ij}$  = the contribution of factor  $i$  on grade  $j$   
 $M_{ij}$  = the weighting factor of  $j$ -th range of  $i$ -th factor  
 $\mu_{R_{ijk}}$  = the fuzzy membership of  $j$ -th range of  $i$ -th factor to  $k$ -th grade  
 $\Sigma N$  = total number of sample  
 $\Sigma n_{ij}$  = number of sample distributed in  $j$ -th range of  $i$ -th factor  
 $n_{ijk}$  = number of sample distributed in  $k$ -th grade at  $j$ -th range of  $i$ -th factor  
 $\circ$  = fuzzy operation of the maximum in minimums  
 $\vee$  = fuzzy operation of maximum calculus  
 $\wedge$  = fuzzy operation of minimum calculus.

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# FLOW BEHAVIOR OF TOMATO SAUCE WITH OR WITHOUT PARTICULATES IN TUBE FLOW<sup>1</sup>

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## ABSTRACT

*Viscosity and density are two properties of fluids that are affected by temperature changes. The flow behavior of tomato sauce (7° and 14° Brix) in a tube viscometer was studied at varying concentrations of added particulates (2 and 5 wt%) at temperatures in the range of 70 to 95°C. The results indicate that the Power Law model described the flow behavior of the non-Newtonian fluids better than the Casson model. The effects of temperature, concentration of carrier fluids and particulates on the flow behavior indices and viscosity was studied. The effect of temperature on the flow behavior index (n) and consistency index (K) were modeled as exponential relationships. The values of n and K for 7° Brix sauce ranged from 0.30 to 0.86 and 0.04 to 0.34, respectively. For the 14° Brix sauce these values were 0.27 to 0.53 and 0.20 to 0.60 respectively. The values of n decreased and K increased with increase in concentration of solids and particulates. The relative changes in K and n were lower for higher concentrations of solids and particulates. It was found that temperature had a greater effect on K than on n.*

*The effects of temperature on viscosity was modeled as an Arrhenius type equation from which the activation energies were calculated. The combined effects of temperature, concentration of solids in the sauce and added particulates on apparent viscosity can be predicted by a nonlinear model.*

## INTRODUCTION

In recent years there has been a wide interest in the high temperature short time (HTST) processing of fluid foods with and without particulates. Prediction of flow behavior for these foods is complicated. The design and evaluation of

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food processing equipment for fluid foods is dependent on the flow characteristics of the product.

In the food industry, most non-Newtonian fluids are pseudoplastic fluids. The relationship between the shear stress and the shear rate for these fluids is usually described by the Power Law:

$$\tau = K (\dot{\gamma})^n \quad [1]$$

The value of  $n$  for pseudoplastic fluids lies between 0 and 1.

Most fluids with marked non-Newtonian behavior have such high consistencies that turbulent flow in tubular heat exchangers is difficult to attain and therefore these will usually have laminar flow. In laminar flow of pseudoplastic fluids in tubes there are two limiting cases: (1) for a flow behavior index of  $n = 0$ , a uniform velocity profile exists and (2) for a flow behavior index of  $n = 1$ , a parabolic velocity profile is obtained. For all the intermediate values of  $n$ , the velocity profile becomes somewhere between uniform and parabolic.

Both viscosity and density of fluids are dependent on temperature, and therefore a variation in temperature over the path of flow process will affect the flow behavior. Consequently for most pseudoplastic fluids,  $K$  and  $n$  are temperature dependent. Usually,  $K$  is a stronger function of temperature than  $n$ . The strong temperature dependence of  $K$  causes the velocity profile to undergo substantial changes in a tubular heat exchanger. The temperature difference between bulk and wall generates a density gradient. This causes a free convection which further complicates fluid flow and heat transfer.

Much literature is available on flow behavior measurements of foods using viscometers like the rotating cylinder (Brekke *et al.* 1978; Crandall *et al.* 1982; Harper and Lebermann 1962; Harper and Sahrigi 1965; Mizrahi and Berk 1970, 1972; Rao 1977; Rao *et al.* 1974, 1981), and cone and plate types (Davis 1973) and tube viscometers (Sarvacos 1968; Vitali and Rao 1982). However, most of these researchers have limited their study to product temperatures of 60°C or less. Few studies involve higher temperatures (Filkova *et al.* 1987; Sarvacos 1968). Very little data is available on the flow behavior of non-Newtonian fluid foods with added particulates (Rao 1987).

The majority of food fluids contain several constituents. One of these is a substrate (continuous phase) and the other or others may be distributed through the substrate as particles in suspension or large molecules in solution (dispersed phase). A number of expressions may be found scattered about in the literature which purport to explain the effect of concentration of suspended matter on the rheological properties of the suspensions. Some of these correlations are purely empirical while others are based on the hydrodynamic approach of Einstein or the network approach (Adam and Delsanti 1977; Goodwin 1975; Napper and Hunter 1972; Simha 1940; Tanford 1961). All these correlations predict the

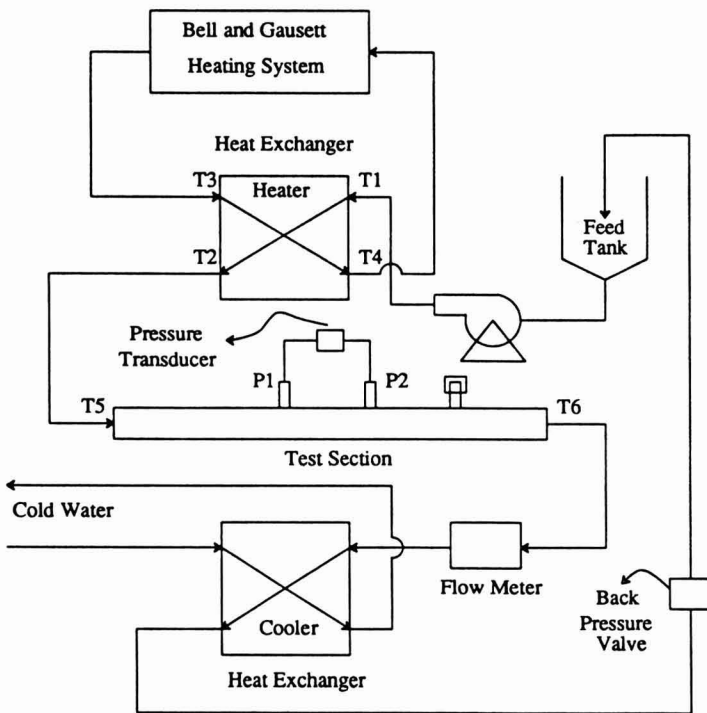
viscosity of polymer solutions or suspensions of noninteracting species. However, the rheological behavior of food materials at the microstructural level is difficult to predict because of a large number of interacting components present in the system.

The specific objective of this study was to investigate the effect of temperatures in the range of 65 to 100°C on the flow behavior of fluid foods with and without added particulates in a tubular heat exchanger as a function of different experimental conditions obtained by varying shear rates, solid contents of tomato sauces, percentage of particulates (by weight) and particulates.

## MATERIALS AND METHODS

### Experimental Setup

A schematic representation of the experimental setup is shown in Fig. 1.



T1 to T6 - Temperature measurement points

P1, P2 - Differential pressure ports

FIG. 1. SCHEMATIC REPRESENTATION OF EXPERIMENTAL SETUP

## Test Section

The heart of the system is the test section. Two test sections were made of stainless steel, each 3.048 m (10') long and 1.54 and 2.44 cm in diameter (internal), respectively. Tri clover fittings were welded on both ends. Three legs (0.318 cm i.d., 0.635 cm o.d) were welded to three holes at 0.6096 m (2'), 1.2192 m (4') and 2.4384 m (8') from an end. The three legs had threaded ends to which caps could be screwed on. The whole tube was insulated.

It is important to consider the entrance effect which arises due to changes in velocity profile and abrupt changes in shear distribution when the product is forced from a large diameter tube to a smaller diameter tube. This was eliminated by using a long entrance region (1.212 m).

Any two legs were used to measure the pressure difference and the third leg was closed with a cap. Rubber tubes were connected from each of the two legs to the ends of the pressure transducer to form an U-shape. The rubber tube connections were clamped tightly to avoid any possibility of leakage.

## Heating Unit

A double tube heat exchanger was used to heat the product (3.81 cm i.d., 5.08 cm o.d). Heating of the product was done in a counter current fashion. Hot water up to 132°C (270°F) as a heating medium was obtained from a Bell and Gaussett heating system.

## Cooling Unit

The cooling unit was a specially designed heat exchanger. It was a 4.572 m (15') copper tube (2.875 cm (1 $\frac{1}{8}$ " o.d, 2.54 cm (1" i.d) immersed in a 0.208 m<sup>3</sup> (55 gallon) drum. Cold water was run continuously from the bottom of the drum. This was used to cool the product to a temperature below its boiling point. At temperatures above 99°C (210°F), ice was dumped into the drum to enhance cooling.

## Data Acquisition System

An Omega WA-FAI system attached to an IBM personal computer (Compaq) was used to collect data. The inputs recorded were inlet and outlet temperatures of the hot water and product, the volumetric flow rate, the differential pressure, the inlet and outlet temperatures of the product through the test section.

## Pumps and Accessories

A Waukesha pump (Model D025831SS, Waukesha foundry Company, Waukesha, Wisconsin) was used to pump the product through the system. Copper-Constantan thermocouple were used to read the various temperatures.

## Flow Meter

The flow meter used was a Micro-Mag Transmitter (1103T Model B, Taylor Instrument Inc., New York). The output of the flow meter was a 0–25 mA signal which was converted to a 0–25 mV signal by connecting an  $1\Omega$  resistance across the positive and negative terminals. The flow meter was calibrated against actual volumetric flow rate as measured by a bucket and a stop watch.

## Pressure Transducer

The pressure difference between any two points of the test section was measured by a wet/wet differential pressure transducer (Model number PX820-010DV, Omega Engineering, Inc., Stamford, CT). Its output signal was calibrated against the actual static pressure caused by the height of a water column.

## Model Food Systems

Tomato sauce was used as a fluid food. Frozen thawed peas and dry soybeans from the food stores at Purdue University were chosen as particulates. Studies were conducted on two types of particulates mixed in the model fluids.

## Preparation Of Model Fluids

All tomato sauce samples were prepared from a standard tomato paste of 31° Brix (supplied by Naas Foods, Portland, Indiana). Water in required amounts was added to the original paste, and the blend was well mixed to get the desired 7° and 14° Brix solid concentrations.

To prepare food products with particulates, a known amount of particulates was added to the sauce. Two concentrations of particulates were studied: 2 % and 5 % (by weight).

## Experimental Procedure

The various experimental conditions are shown in Table 1. The hot water flow rate was maintained constant at  $0.1135\text{ m}^3$  (30 gpm). The average dimensions of the particulates were as follows:

TABLE 1.  
EXPERIMENTAL CONDITIONS

Variables	Values
Carrier fluids	Tomato sauce
Particulates	Frozen peas and Dry soybeans.
Volumetric flow rate	$3.154\text{e-}05 - 3.785\text{e-}04\text{ m}^3/\text{s}$
Product temperature ( °C )	71.1, 82.2, 93.3 and 99.0
Solids content of fluids:	
Tomato sauce (° Brix)	7, 14
Particulate concentration ( by weight )	2 %, 5 %



Soybeans: Major diameter: 0.76 cm

Minor diameter: 0.56 cm

Peas: Mean diameter: 0.84 cm

The temperature of the same sample was increased and then decreased for different flow rates. Fresh samples were used for each of the different solids and particulate concentrations.

### Calculation Procedure

**Flow Behavior.** The relationship between shear rate and shear stress is obtained from the measurement of pressure gradient and volumetric flow rate. This method is based on the following assumptions: (a) flow is steady, (b) flow is laminar, (c) fluid properties are time independent, (d) fluid exhibits no slippage at the wall, (e) fluid is incompressible, (f) fluid velocity has no radial or tangential components, (g) fluid viscosity is not influenced by pressure and (h) measurement is conducted under isothermal conditions.

The entrance length ( $L_e$ ) for laminar flow of fluids through pipes is given by the following approximate expression (Kays 1966):

$$\frac{L_e}{D} = \frac{N_{Re}}{20} \quad [2]$$

The volumetric flow rate is given by the following expression:

$$Q = \pi \left( \frac{\Delta P}{2K \Delta L} \right)^{\frac{1}{n}} \left( \frac{n}{3n+1} \right) R^{\frac{3n+1}{n}} \quad [3]$$

The shear stress at the wall is given by:

$$\tau_w = \frac{D \Delta P}{4 \Delta L} \quad [4]$$

Skelland (1967) has presented in detail the Rabinowitsch-Mooney equation to calculate the rate of shear at the wall which is:

$$\dot{\gamma} = \left( \frac{3n+1}{n} \right) \left( \frac{Q}{\pi R^3} \right) \quad [5]$$

The apparent viscosity is given by:

$$\mu_a = K (\dot{\gamma})^{n-1} \quad [6]$$

### Flow Behavior Studies

The Power Law and Casson models were fitted to study the flow behavior of tomato sauce.

$$\text{Power Law } \tau = K (\dot{\gamma})^n \quad [7]$$

$$\text{Casson model } \tau^{1/2} = K_0 + K_1 \dot{\gamma}^{1/2} \quad [8]$$

The parameters that were studied to describe the individual and combined effects of temperature and concentrations of both, dissolved solids and particulates on apparent viscosity, flow behavior index and consistency index are shown in Table 2.

## RESULTS AND DISCUSSION

### Flow Models For Tomato Sauce

**Power Law.** Shear stress versus shear rate (log-log scale) data are plotted for 7° Brix tomato sauce in Fig. 2 for a temperature range of 22–94°C and a shear rate range of 250–1200  $s^{-1}$ . It is noted that at 22.2°C, the data are well described by the Power Law model. At higher temperatures, there is a scatter of points thereby showing a deviation from the power law. The shear stress-shear rate relationships and the  $R^2$  values for the fit are shown in Table 3.

It is observed (Fig. 2) that at low shear rates (less than 450  $s^{-1}$ ), a rapid drop in wall shear stress was observed suggesting a threshold value for shear rate.

TABLE 2.  
TEMPERATURE AND/OR CONCENTRATION RELATIONSHIPS OF  
FLOW BEHAVIOR PARAMETERS

Effect of temperature on apparent viscosity at constant shear.	$\mu_a = \mu_0 \exp\left(\frac{-E_a}{RT}\right)$
Effect of temperature and concentration of solids and particulates on apparent viscosity.	$\mu_a = \mu_0 C_1^a \exp\left(\frac{-E_a}{RT}\right) + C_2^b$
Effect of temperature on flow behavior index.	$n = n_0 \exp(-aT)$
Effect of temperature on consistency index.	$K = K_0 \exp(-aT)$

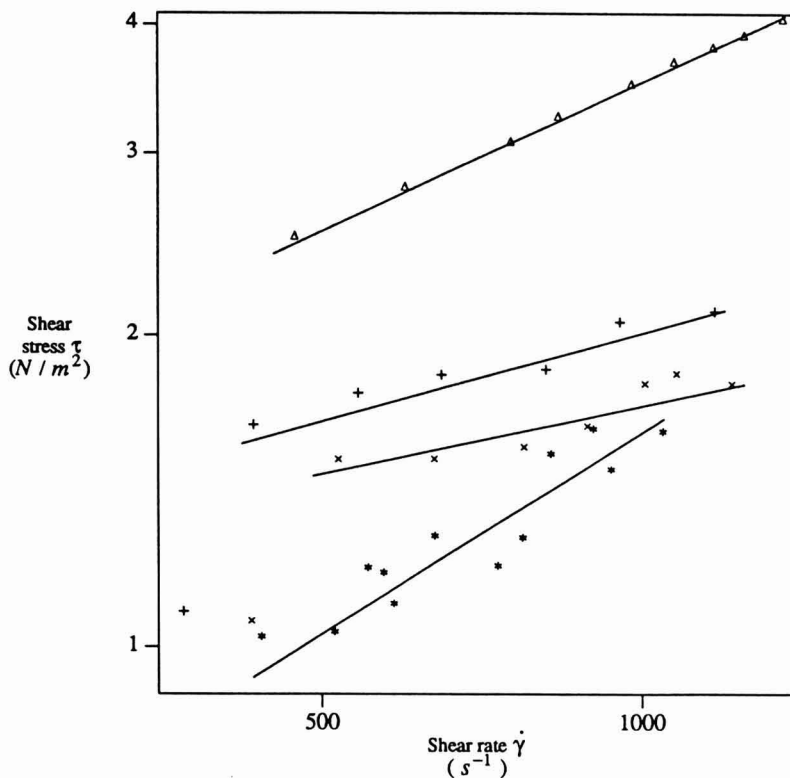


FIG. 2. APPLICABILITY OF POWER LAW MODEL FOR 7° BRIX TOMATO SAUCE  
 $\Delta$  (22.22°C), + (71.11°C),  $\times$  (82.22°C), \* (93.33°C)

TABLE 3.  
 APPLICABILITY OF POWER LAW MODEL FOR TOMATO SAUCES

Temperature (°C)	7° Brix Sauce		14° Brix Sauce	
	Model	$R^2$	Model	$R^2$
22.22	$\tau = 0.1471 (\dot{\gamma})^{0.4621}$	0.99	$\tau = 1.9121 (\dot{\gamma})^{0.2404}$	0.96
71.11	$\tau = 0.01379 (\dot{\gamma})^{0.6925}$	0.99	$\tau = 0.6016 (\dot{\gamma})^{0.3553}$	0.98
82.22	$\tau = 0.00413 (\dot{\gamma})^{0.8569}$	0.99	$\tau = 0.4204 (\dot{\gamma})^{0.4033}$	0.99
93.33	$\tau = 0.00415 (\dot{\gamma})^{0.8787}$	1.00	$\tau = 0.3240 (\dot{\gamma})^{0.4343}$	0.99
100	-	-	$\tau = 0.2787 (\dot{\gamma})^{0.4175}$	0.99

This might be due to slip at the wall surface at these low values of shear rates. Harper and Sahrighi (1965) reported that the behavior at low shear rates may be influenced by yield phenomena, time dependency and wall effects. The particulates act as a suspension of solid particles and these tend to migrate away from the walls. This results in lower concentration and lower effective viscosity within the tube ("Pinch effect", Rao 1977). The results obtained are shown in Table 3. Table 4 shows the effects of temperature, concentrations of dissolved solids and particulates on flow behavior index for 7° Brix and 14° Brix sauces.

Figure 3 shows the shear stress-shear rate relationship for 14° Brix tomato sauce. The curves are plotted for temperatures of 22 to 100°C and shear rates of 150 to 1300  $s^{-1}$ . A similar trend as described before (for 7° Brix tomato sauce) is observed. A threshold value of shear rate (about 300  $s^{-1}$ ) is observed here too beyond which there is a jump in the wall shear stress value. This could be explained by the slip at the wall under those conditions. However, an excellent correlation for other data points (at shear rates greater than 300  $s^{-1}$ ) was obtained.

Based on the data obtained for the shear stress-shear rate relationship, the effect of temperature on the flow behavior index ( $n$ ) and the consistency index ( $K$ ) was studied. The relationships observed are given in Tables 4 and 5, respectively. There is a good agreement of our results with those of Filkova *et al.* 1987. The effects of temperature and concentration of dissolved solids on the value of  $n$  for tomato sauces is shown in Fig. 4. The value of  $n$  increases with temperature as expected.

The effects of temperature, concentrations of dissolved solids and particulates on the values of  $n$  and  $K$  are shown in Table 4. By comparing Tables 4 and 5, it is seen that a change in temperature has a more prominent effect on the consistency index than on the flow behavior index. Figure 5 shows the effects

TABLE 4.  
EFFECT OF TEMPERATURE AND CONCENTRATION OF PARTICULATES  
ON VALUE OF  $n$

Product	$n$	$R^2$
7° Brix Sauce	0.3727 exp (0.0094 $T$ )	0.98
7° Brix Sauce + 2% soybeans	0.1699 exp (0.0092 $T$ )	0.96
7° Brix Sauce + 5% soybeans	0.1903 exp (0.0074 $T$ )	0.95
14° Brix Sauce	0.2010 exp(0.0081 $T$ )	0.99
14° Brix Sauce + 5% Peas	0.036 exp (0.0277 $T$ )	0.98

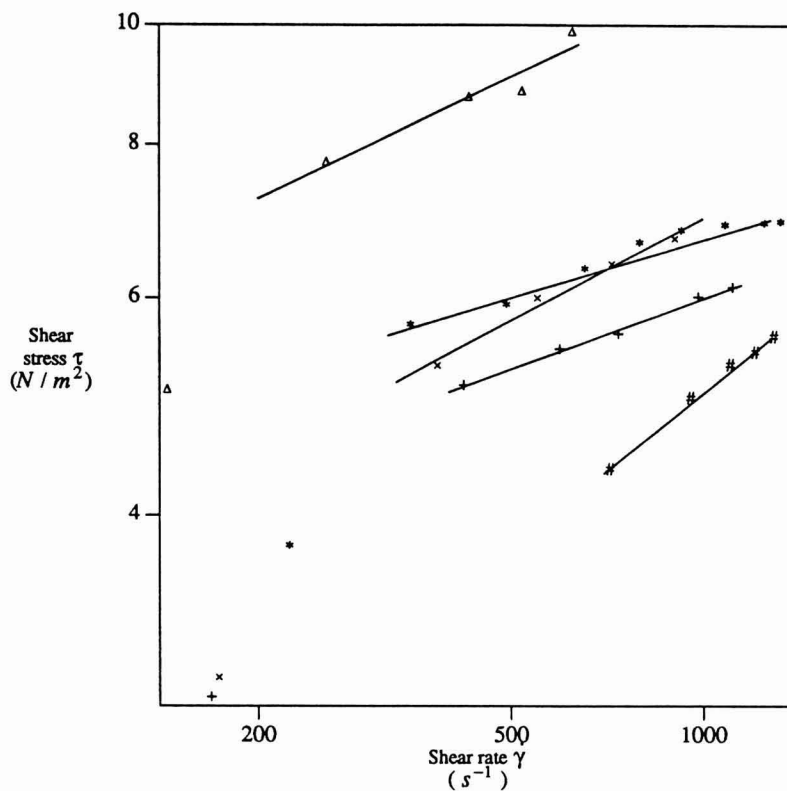


FIG. 3. APPLICABILITY OF POWER LAW MODEL FOR 14° BRIX TOMATO SAUCE  
 $\Delta$  (22.22°C), \* (71.11°C),  $\times$  (82.22°C), + (96.11°C), # (100°C)

TABLE 5.  
 EFFECT OF TEMPERATURE AND PARTICULATES ON VALUES OF  $K$

Product	$K$	$R^2$
7° Brix sauce	$0.5249 \exp(-0.0540 T)$	0.99
7° Brix Sauce +2% soybeans	$1.0056 \exp(-0.0229 T)$	0.97
7° Brix Sauce +5% soybeans	$1.8103 \exp(-0.0223 T)$	0.98
14° Brix Sauce	$3.5987 \exp(-0.0254 T)$	0.99
14° Brix Sauce +5% peas	$72.35 \exp(-0.0630 T)$	0.95

of temperature and added particulates on the value of  $n$ . It is observed that, on the addition of 5% soybeans, the relative change in  $n$  is smaller than when 2% soybeans are added. The same trend is observed for the values of  $K$  also (Fig. 6) whereas the value of  $n$  increases and value of  $K$  decreases with increase in temperature.

The effects of temperature on viscosity for different concentrations of sauces and particulates are shown in Table 6. The apparent viscosity was measured at a shear rate of  $750 \text{ s}^{-1}$ . In general, it was observed that the apparent viscosity decreased with increasing temperature and increased with increasing particulate concentration.

The increase in viscosity due to added particulates can be explained by the hydrodynamic approach. In case of the food system chosen in this study (tomato sauce with discrete particles), each particle will rotate in the shear field with an angular velocity equal to one-half of the shear rate,  $\dot{\gamma}$  (Prentice 1984). As two particulates approach each other, either by reason of one overtaking the other

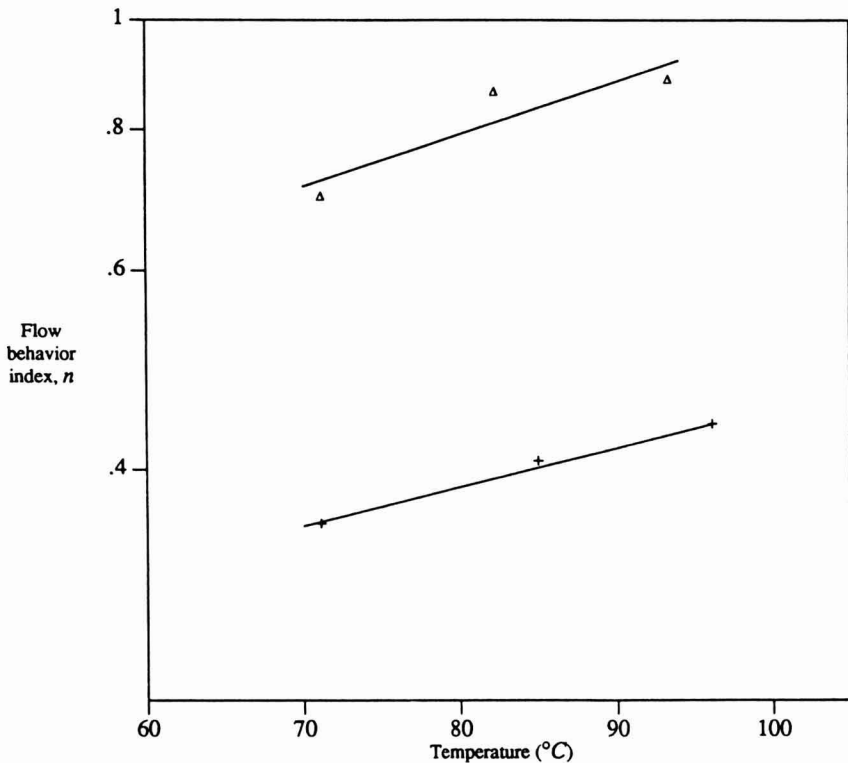


FIG. 4. EFFECT OF TEMPERATURE AND SOLIDS CONCENTRATION ON FLOW BEHAVIOR INDEX OF TOMATO SAUCE

$\Delta$  - 7° Brix, + - 14° Brix.

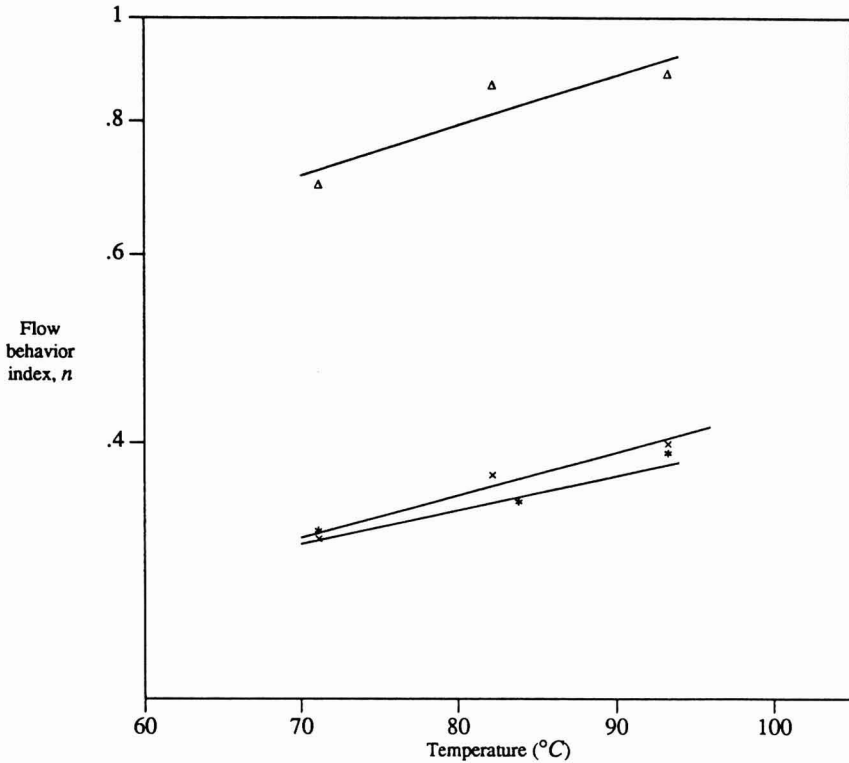


FIG. 5. EFFECT OF TEMPERATURE ON FLOW BEHAVIOR INDEX WITH AND WITHOUT PARTICULATES

$\Delta$  -7° Brix sauce,  $\times$  -2% soybeans,  $*$  -5% soybeans

or because of random fluctuations due to Brownian motion, the local shear rate in the medium between them would rise rapidly. This phenomenon allows fluid to show higher viscosity than that would be possible without particulates.

The activation energies of tomato sauce were calculated according to the Arrhenius equation (Eq.9).

$$\mu = \mu_0 e^{\frac{E_a}{RT}} \quad [9]$$

The results are tabulated in Table 7. The activation energies tend to decrease with increase in total solids concentration of sauces and concentration of particulates. These values are higher than those reported by Rao *et al.* (1981). One reason for the difference could be that different types of tomatoes were used in the two studies. Another reason could be that the experiments of Rao *et al.* (1981) were conducted using a rotational viscometer where it is assumed that

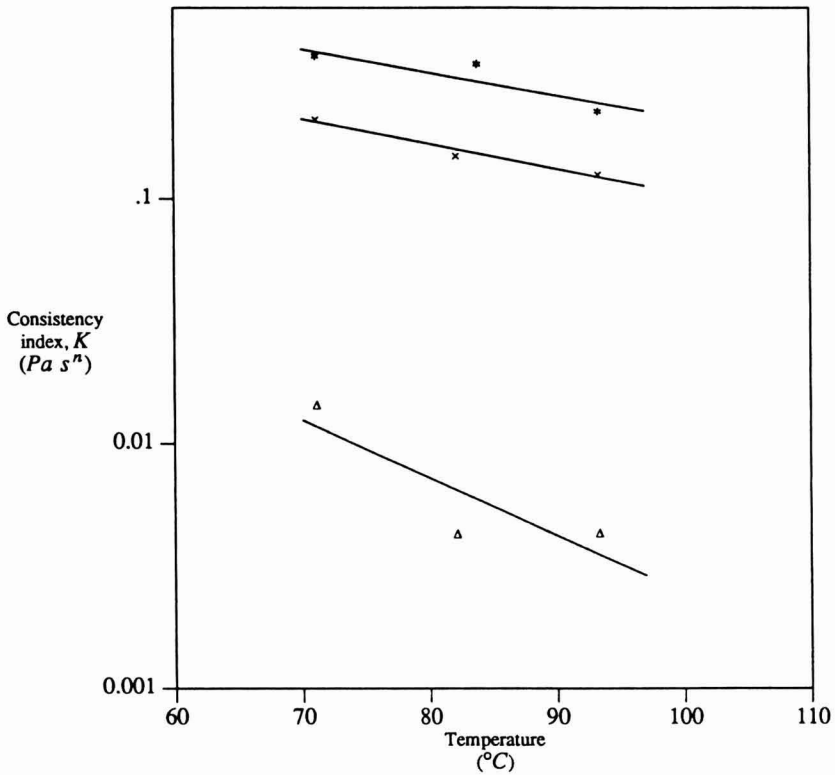


FIG. 6. EFFECT OF TEMPERATURE ON CONSISTENCY INDEX WITH AND WITHOUT PARTICULATES

Δ - 7° Brix sauce, × - 2 % soybeans, \* - 5 % soybeans

TABLE 6. EFFECT OF TEMPERATURE ON APPARENT VISCOSITY AT A SHEAR RATE OF 750 s<sup>-1</sup>

Product	App. Viscosity 10 <sup>3</sup> Pa.s	Temperature (°C)	Relationship $\mu = \mu_0 e^{-af}$
7° Brix tomato sauce	2.4876	71.11	9.932e-3exp(-0.019 T)
	2.0637	82.22	
	1.6187	93.33	
7° Brix sauce + 2% soybeans	2.2689	71.11	2.556e-3exp(-0.0016 T)
	2.2476	82.22	
	2.2047	93.33	
7° Brix sauce + 5% soybeans	5.1514	71.11	0.0157exp(-0.0159 T)
	4.1739	84.0	
	3.5378	93.33	
14° Brix sauce	8.6757	71.11	0.0139exp(-0.0063 T)
	8.5403	85.0	
	7.3652	96.11	
	5.9767	100.0	



TABLE 7.  
ACTIVATION ENERGIES AS CALCULATED BY EQ.9

Product	$E_a$ (kcal/mole K)	$\mu_0$ (Pa.s)	$R^2$
7° Brix sauce	4.817	2.192e-6	0.99
7° Brix + 2% soybeans	3.988	1.245e-5	0.97
7° Brix + 5% soybeans	3.981	1.504e-5	0.94
14° Brix sauce	2.858	1.402e-4	0.99

the fluid in the gap is being sheared only at the sides and not at the ends of the rotating cylinder (end effects).

Based on the data in Table 6, a nonlinear model for predicting the apparent viscosity as a function of temperature, suspended solids and added particulates was developed. This is an extension to the models suggested by Rao (1987) and is given by:

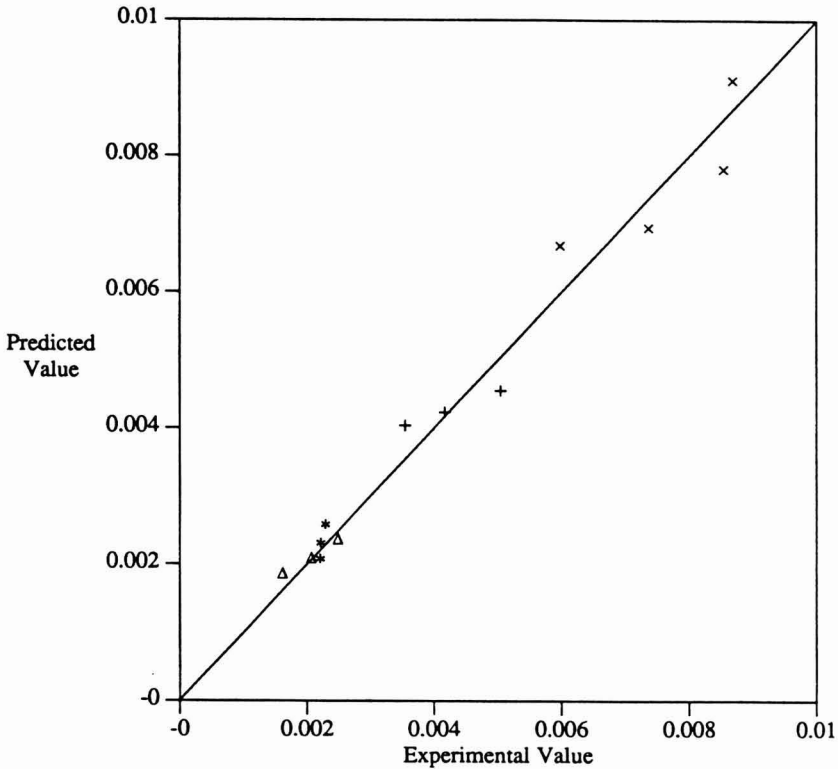


FIG. 7. PLOT OF PREDICTED VALUES (EQ.10) VERSUS EXPERIMENTAL VALUES OF  $\mu_a$  FOR TOMATO SAUCES

$\Delta$  - 7° Brix, \* - 2 % soybeans, + - 5 % soybeans,  $\times$  - 14° Brix

$$\mu_a = P_1 \exp(P_2 T)(C_1)^{P_3} + P_4 (C_2)^{P_5} \quad [10]$$

The constants P1 through P5 calculated by a nonlinear regression analysis using a statistical package (SAS 1985):

$$P_1 = 1.00e-06$$

$$P_2 = 1383.374$$

$$P_3 = 1.953$$

$$P_4 = 1.80e-05$$

$$P_5 = 2.9778$$

The predicted viscosity using Eq. 10 is plotted against the experimental value in Fig. 7. The effect of temperature and concentrations of solids and particulates is well described by the expression as can be seen from the plot. The sum of squares of the residuals for the 13 observations was 2.00e-06.

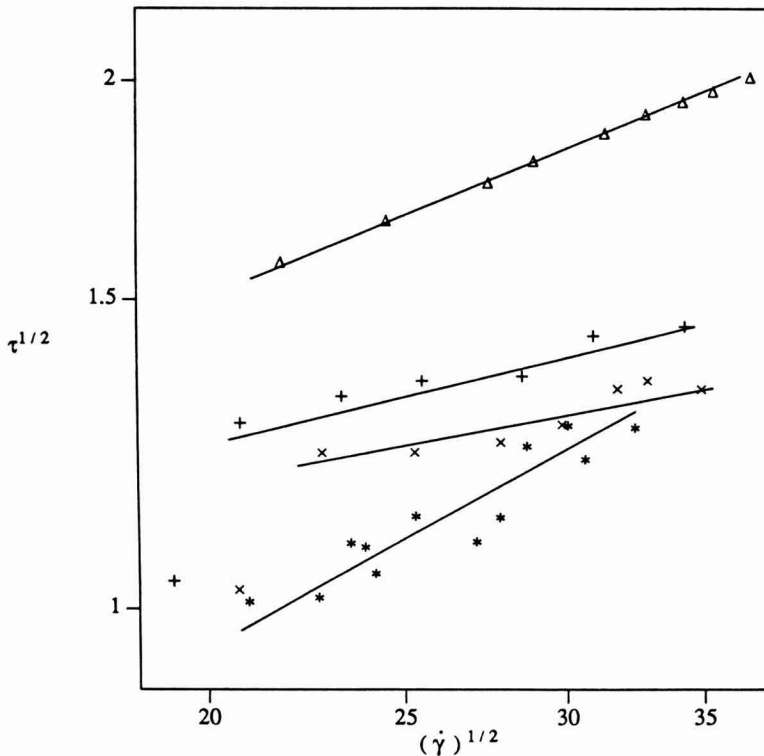


FIG. 8. APPLICABILITY OF CASSON'S MODEL FOR 7° BRUX TOMATO SAUCE  
 $\Delta$  (22.22°C), + (71.11°C),  $\times$  (82.22°C), \* (93.33°C)

### Casson's Model

The Casson's model (Eq.8) was fitted to the data for 7° and 14° Brix tomato sauces are shown in Fig. 8 and Fig. 9, respectively. The  $R^2$  values (Table 8) are much lower than the corresponding  $R^2$  values for Power Law model. Hence the Power Law model was preferred over the Casson model to describe the flow behavior of tomato sauces at these temperatures.

### CONCLUSIONS

The flow behavior of tomato sauces (7 and 14° Brix) with added particulates (2 and 5 wt%) was studied at temperatures in the range of 70 to 95°C. This study provides an insight into the flow behavior of heterogeneous foods at relatively high temperatures.

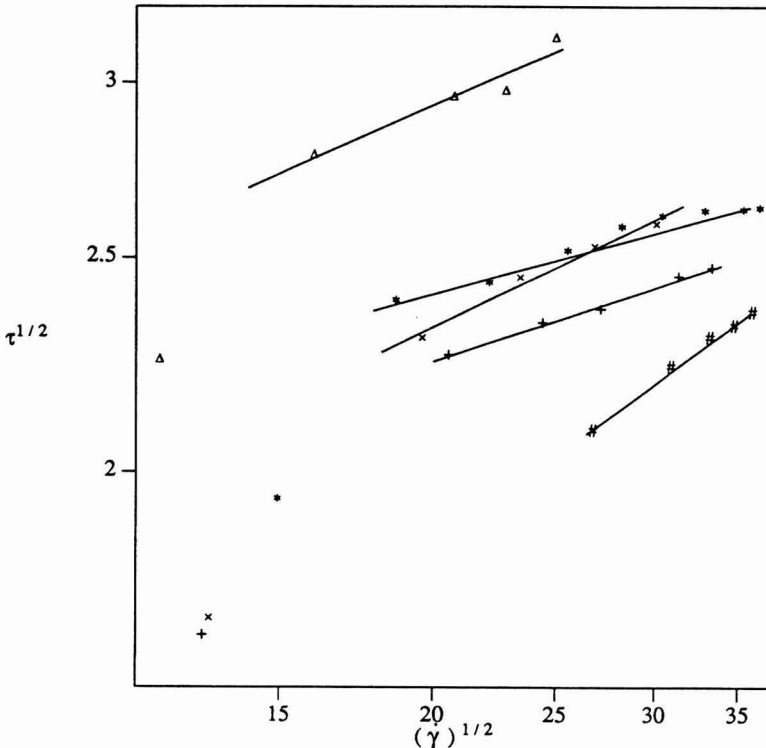


FIG. 9. APPLICABILITY OF CASSON'S MODEL FOR 14° BRIX TOMATO SAUCE  
 $\Delta$  (22.22°C), \* (71.11°C), × (82.22°C), + (96.11°C), # (100°C)

TABLE 8.  
COEFFICIENTS FOR CASSON MODEL EQ.8

Product	Temperature (°C)	$K_0$	$K_1$	$R^2$
7° Brix	22.22	0.9628	0.0286	0.9969
	71.11	0.7609	0.0213	0.7208
	82.22	0.4767	0.0245	0.8607
	93.33	0.7322	0.0184	0.7811
14° Brix	22.22	1.6197	0.0617	0.8969
	71.11	1.7727	0.0254	0.7503
	82.22	1.1690	0.0503	0.8947
	96.11	1.3567	0.0359	0.8504
	100.00	1.2930	0.0296	0.9912

The Power Law model describes well the shear rate-stress relationship for 7° Brix and 14° Brix tomato sauces at temperatures in the range of 70 to 95°C. The correlation coefficients were between 0.99 and 1.00.

The effect of temperature on the flow behavior index and consistency index using an exponential relationship shows that below a threshold shear rate ( $200s^{-1}$  for 7° Brix and  $450s^{-1}$  for 14° Brix), the observed data deviate from the model. This is explained by the slip at these conditions as described by the “pinch effect”.

A nonlinear model for apparent viscosity as a function of temperature was developed using a multiple regression program. An Arrhenius type equation was fitted to model the effects of temperature on viscosity. The values of activation energies calculated range from 3.9 to 4.82 kcal/mole K for 7° Brix.

The value of  $K$  increased and the value of  $n$  decreased with increase in added particulate concentrations. The relative changes in the values of  $K$  and  $n$  decreased with increase of particulate concentration.

The Casson model described the flow data well but had lower correlational coefficients than the power law model.

## NOMENCLATURE

### Symbols

$C_1$	= Concentration of dissolved solids (wt %)
$C_2$	= Concentration of particulates (wt %)
$E_a$	= Activation energy (kcal/mole K)
$K$	= Consistency index ( $Pa.s^n$ )
$L_e$	= Entrance length (m)
$n$	= Flow behavior index

$N_{Re}$	= Generalized Reynold's number
$P_1, \dots, P_5$	= Constants
$Q$	= Volumetric flow rate ( $m^3/s$ )
$R$	= Tube radius ( $m$ )
$R_G$	= Gas constant
$T$	= Temperature ( $^{\circ}C$ )
$\dot{\gamma}$	= Shear rate ( $s^{-1}$ )
$\Delta L$	= Differential length ( $m$ )
$\Delta P$	= Differential pressure ( $Pa$ )
$\mu_a$	= Apparent viscosity ( $Pa.s$ )
$\mu_0$	= Constant ( $Pa.s$ )
$\tau$	= Shear stress ( $Pa$ )

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# SENSITIVITY ANALYSIS OF ASEPTIC PROCESS SIMULATIONS FOR FOODS CONTAINING PARTICULATES<sup>1</sup>

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## ABSTRACT

*A sensitivity analysis of a computer model, simulating heat transfer into particulate foods processed in a continuous aseptic system was conducted to determine how the model reacts to the variations in selected product and process input parameters depending on simulation types, namely, "Total", "F<sub>0</sub> Hold" and "Hold Only". Three major output variables, namely, holding tube required to destroy 6 D of Clostridium sporogenes (PA3679), destruction of thiamine and inactivation of peroxidase were selected for the sensitivity analysis. Theoretical results indicated that basically no difference between the "Total" system approach and the "F<sub>0</sub> Hold" approach was found on the model prediction. Particle size, particle thermal properties such as density and specific heat were the most sensitive parameters (within the range investigated) among product parameters which influence holding tube length required, thiamine and peroxidase retention while fluid properties such as fluid thermal conductivity and viscosity were less susceptible to affect the model prediction regardless of simulation types. Among process parameters, product flow rate and product initial temperature seemed to be the most critical parameters within the range covered in this study while rotor speed seemed to be one of the least critical parameters to influence the model prediction regardless of simulation types.*

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## INTRODUCTION

In recent years, there has been a widespread growth of interest and research in the processing of low-acid foods ( $\text{pH} > 4.6$ ) containing particulates. One of the main concerns in designing the sterilization systems is to achieve commercial sterility while preserving maximum quality of the product. In this regard, aseptic processing is favored over traditional thermal processing (i.e., foods are hermetically packed in metal, glass, or plastic containers followed by heating in pressure retorts (for low acid foods) or in boiling water (for acid or acidified foods) in order to achieve commercial sterility). However, there are no commercially available low-acid foods containing particulates larger than 3.2 mm (major dimension) processed aseptically (Chandarana and Gavin 1989), mainly due to difficulties in experimentally verifying the commercial sterility of these products by physical means.

In contrast, commercial sterility in canning industry is assured by the reliable heat penetration data and often confirmed by use of inoculated packs (NCA 1968). However, no comparable methods for low-acid foods containing particulates processed aseptically are proven because it is currently impossible to measure actual temperatures in a moving particle within a continuously flowing fluid in a closed system under pressure. The Food and Drug Administration (FDA) would accept simulation models with appropriate experimental verification of achievement of sterility (Dignan *et al.* 1989) for verification of the filed process required for low-acid foods according to CFR 21:113 (Chang and Toledo 1989). In this regard, mathematical simulation models are useful in designing such processes and equipment by estimating particle temperatures during a heat/hold/cool system.

Several attempts have been made to model aseptic processing of foods containing particulates (de Ruyter and Brunet 1973; Manson and Cullen 1974; Dail 1985, Sastry 1986; Chandarana *et al.* 1989; Chandarana and Gavin 1989; Chang and Toledo 1989; Larkin 1989; Lee *et al.* 1990). de Ruyter and Brunet (1973) presented a model to estimate process conditions for continuous sterilization of foods containing particulates in a scraped surface heat exchanger (SSHE). Manson and Cullen (1974) also developed a computer model (finite difference method) describing the aseptic (thermal) processing of a food product containing discrete particulates in an SSHE and a holding tube. Although the latter two studies presented considerable insight into the critical parameters influencing sterilization of such products, unfortunately, both models assumed infinite heat transfer coefficient at the interface between particle and fluid. Consequently, it implied that Biot number was also assumed to be infinite, i.e., the particle surface temperature was equal to the average or bulk temperature of the continuous phase at all times, thus, overestimating the lethality delivered to the particles. However,

in real food system the range of Biot number is often between 0.1 and 40, that is, the resistance to heat transfer at the liquid-particle interface cannot be neglected (Heldman and Singh 1981).

Recently, Dail (1985) proposed that heating equations derived from the analytical solutions for conduction heat transfer into infinite slab and infinite cylinder could be substituted into Ball's formula method (Ball and Olson 1957) to calculate hold time for low acid foods containing particulates. Dail was unable to experimentally verify the approach, however. Larkin (1989) presented a modified Ball's formula method in evaluating aseptically processed (in an SSHE and a hold tube) foods containing particulates. Larkin used finite difference computation procedures accounting for time-dependent boundary conditions and developed a simplified approach for evaluating lethality accumulated in a continuous aseptic processing system.

Sastry (1986) developed a quantitative methodology for evaluating thermal process schedules for low-acid foods containing particles of any shape (in an SSHE and a holding tube). Finite element analysis was used to determine temperature distributions within particulate foods with time dependent boundary conditions. He found that particle size, residence time distributions within an SSHE and a holding tube, and convective heat transfer coefficients had significant effects on the thermal process schedule required to achieve commercial sterility. Chandarana *et al.* (1989) and Lee *et al.* (1990) also developed a mathematical model (finite difference method) to describe aseptic processing of particulate foods in a continuous sterilization system. Chandarana *et al.* concluded that parameters such as thermal, physical and rheological properties of the food to be processed, residence time distribution of the food within the processing system, and particle/fluid interface convective heat transfer coefficient were important in thermal process calculation of such products. Lee *et al.* (1990) agreed with Chandarana *et al.* and added that influence of effective overall heat transfer coefficient both for heater and holding tube was considerable in determination of minimum process time required to obtain 6 D for *Clostridium sporogenes* (PA3679) and 12 D for peroxidase to prevent regeneration.

Chang and Toledo (1989) also demonstrated that the explicit finite difference method could be used in simulating heating and microbial and enzyme inactivation under various conditions that might be encountered in sterilization of a product containing particulates. Chang and Toledo concluded that the choice of values for the particle/fluid surface heat transfer coefficient used in the simulation had a significant impact on the prediction of whether or not the product has been processed safely. It was also indicated that the temperature profile of the product containing particle was dependent on liquid to solid ratio as the mixture passes through the heat/hold/cool sections of a continuous sterilization system. Chandarana and Gavin (1989) further investigated various approaches to theoretically

design thermal processes for a commercially sterile model product. Chandarana and Gavin concluded that scheduling of a thermal process for a particle center  $F_0$ -value of 6.0 min could result in an effective  $F_0$ -value of as high as 78 min when neglecting the thermal contribution of a heat exchanger. In addition, it was indicated that such a conservative approach could lead to higher nutrient destruction of 14 to 19%, instead of 6 to 17% depending on the particle size.

With the use of a model the temperature distribution and the time/temperature profile of a particle in a system can be mathematically predicted from the knowledge of the size of the particle, thermal properties of both particles and liquid medium, residence time distribution of particles, and surface heat transfer coefficient between the liquid and the particle. The influence that each of these and other processing parameters has on the thermal process needs to be completely understood so that the proper critical control factors are known.

In evaluation of processes using such models, it is essential to know reliable experimental product and process parameters such as thermal and physical properties of both particle and fluid, heat transfer coefficient at the particle surface, particle residence time distribution before any meaningful calculation can be made. Some models have considered influence of these parameters, however, information concerning relative sensitivity among parameters to the model response is lacking. With the knowledge of parameter sensitivity, the final selection of good parameters can be made quickly without sacrificing the accuracy of stimulation.

The main objective of the study described herein was to determine relative sensitivity among parameters used in simulation of the aseptic processing of foods containing particulates. The model responses chosen were the holding tube length required to achieve 6 D reduction of *Clostridium sporogenes* (PA3679), retention of thiamine and peroxidase after heat/hold process.

## MATERIALS AND METHODS

### Model Aseptic Processing System

The model system simulated in this study consists of commercially available size of a scraped surface heat exchanger (0.836 m<sup>2</sup> SSHE; FranRica, Stockton, CA) for both heating and cooling the product and a holding tube. Heating was simulated by saturated steam while cooling by chilled water. Table 1 shows constant system specifications for SSHE and holding tube used in this study.

### Model Food Systems

The system was modeled using a cube shaped potato particle processed in a model aseptic processing system with water as a carrier fluid. The properties of particle and fluid are given in Table 2.

TABLE 1.  
CONSTANT SYSTEM SPECIFICATIONS USED IN  
THEORETICAL MODEL OF ASEPTIC PROCESS

Parameter	Specifications
<b>SYSTEM*</b>	
SSHE product tube inner diameter	30.48 cm (12.0 in)
SSHE product tube outer diameter	31.75 cm (12.5 in)
SSHE shaft diameter	20.32 cm (8.0 in)
SSHE length	1.03 m (3 ft)
Steam tube inner diameter	36.83 cm (14.5 in)
Number of scraped blades	12
Holding tube inner diameter	4.75 cm (1.87 in)
Holding tube outer diameter	5.08 cm (2.0 in)
<b>PROCESS</b>	
Steam temperature	160 °C (320 °F)
Ambient temperature	26.7 °C (80 °F)
Latent heat of steam	2082 kJ/kg (895 BTU/lb)
Steam convective heat transfer coefficient	7653 W/m <sup>2</sup> K (1348 BTU/h ft <sup>2</sup> °F)
Overall heat transfer coefficient in SSHE	1585 W/m <sup>2</sup> K (279 BTU/h ft <sup>2</sup> °F)

\* Specifications for the system were based on commercial size SSHE (FranRica, Stockton, CA).

### Mathematical Model

The mathematical model consists of the solution of the partial differential and energy balance equations iteratively for a particle cube (Chandarana 1988; Chandarana *et al.* 1989). The initial condition used was that of a known uniform temperature distribution throughout the food. The boundary condition for the particulates used was a time dependent and convective.

$$\rho C_{pp} \frac{\partial T_p}{\partial t} = k \left[ \frac{\partial^2 T_p}{\partial x^2} + \frac{\partial^2 T_p}{\partial y^2} + \frac{\partial^2 T_p}{\partial z^2} \right] \quad [1]$$

with the following initial and boundary conditions

$$\text{I.C. } T_p(x,y,x,0) = T_i$$

$$\text{B.C. } k \frac{\partial T_p}{\partial x}, \quad k \frac{\partial T_p}{\partial y}, \quad k \frac{\partial T_p}{\partial z} = h_p (T_f^j - T_p)$$

at the particle surface.

TABLE 2.  
REFERENCE SYSTEM SPECIFICATIONS USED IN  
THEORETICAL MODEL OF ASEPTIC PROCESS

Parameter	Specifications
<b>PRODUCT</b>	
Particle size	1.27 cm (0.5 in)
Particle loading	20%
Particle thermal conductivity <sup>a</sup>	0.556 W/m °C (0.321 BTU/h ft °F)
Particle density <sup>a</sup>	1070 kg/m <sup>3</sup> (66.80 lb/ft <sup>3</sup> )
Particle specific heat <sup>a</sup>	3.27 kJ/kg °C (0.781 BTU/lb °F)
Fluid thermal conductivity <sup>b</sup>	0.645 W/m °C (0.373 BTU/h ft °F)
Fluid density <sup>b</sup>	957 kg/m <sup>3</sup> (59.8 lb/ft <sup>3</sup> )
Fluid viscosity <sup>b</sup>	533 x 10 <sup>-6</sup> Pa s (0.533 cp)
<b>PROCESS</b>	
Product initial temperature	46.11 °C (115 °F)
Product flow rate	0.45 m <sup>3</sup> /h (2 GPM)
Rotor speed	150 rpm
Velocity ratio <sup>c</sup> in SSHE	0.75
Velocity ratio in holding tube	0.75
Particle surface convective heat transfer coefficient in SSHE	285 W/m <sup>2</sup> K (50 BTU/h ft <sup>2</sup> °F)
Particle surface convective heat transfer coefficient in holding tube	227 W/m <sup>2</sup> K (40 BTU/h ft <sup>2</sup> °F)

<sup>a</sup> Yamada (1970)

<sup>b</sup> Heldman and Singh (1981)

<sup>c</sup> Velocity ratio =  $V_{avg}/V_{max}$

The following equations were solved as suggested by Sastry (1986) assuming that  $k$  is independent of space coordinates:

for heat exchangers,

$$U_{ht} A_{ht} (T_s - T_f^j) = m_f C_{pf} (T_f^{j+1} - T_f^j) + h_p A_p N_p (T_f^j - T_{pm}) \quad [2]$$

$$T_p(x,y,z,t) \leq T_f^j(t)$$

for holding tube,

$$U_{hd} A_{hd} (T_f^j - T_e) = m_f C_{pf} (T_f^{j+1} - T_f^j) + h_p A_p N_p (T_f^j - T_{pm}) \quad [3]$$

for cooler,

$$U_c A_c (T_f^j - T_w) = m_f C_{pf} (T_f^{j+1} - T_f^j) + h_p A_p N_p (T_f^j - T_{pm}) \quad [4]$$

where

$$T_p = T_p(x, y, z, t)$$

$$T_f^j = T_f^j(t)$$

The three dimensional transient heat conduction Eq. (1) was solved numerically using an explicit finite difference scheme (Patankar 1980; Shih 1984). More detailed information in solving the heat transfer equation is given elsewhere (Chandarana 1988; Chandarana *et al.* 1989).

### Process Simulations

**Target Lethality and Process Calculation.** The theoretical processes were based on a target  $F_0$  of 7.2 min, which corresponds to a 6 D process of *Clostridium sporogenes* (PA3679), at the center of the particle leaving the holding tube. The lethality accumulated in the cooler was not included due to possible particle disintegration during cooling with present systems. The breakdown would result in faster cooling than anticipated, thus, less amount of lethality would be accumulated.

A holding tube length required to achieve the target lethality was then calculated for the model systems depending on input parameters and later subject to sensitivity analysis. The lethality accumulated at the center of the particle was calculated using Eq. (5). A decimal reduction time ( $D_{121.1^\circ\text{C}}$ ) of 1.2 min and a  $z$  value of 10.0°C for *Clostridium sporogenes* (PA3679) was used (Reed *et al.* 1951).

$$F = \int_0^t 10^{\frac{(T_o - T_{ref})}{z}} dt \quad [5]$$

**Nutrient and Enzyme Destruction.** In addition to the process calculation based on the target lethality, nutrient (thiamine) and enzyme (peroxidase) destruction were also studied. The respective decimal reduction time ( $D_{121.1^\circ\text{C}}$ ) and  $z$  values of 130 min and 25°C for thiamine (Feliciotti and Esselen 1957) and those of 3.09 min and 36.11°C for peroxidase (Yamamoto *et al.* 1962) were

used in the simulation. The destruction of these biochemicals in both particles and fluid for the whole system was determined by calculating an integrated sterilizing value according to Eq. (6) as recommended by Stumbo (1965) and Teixeira *et al.* (1969). The results were then subjected to the sensitivity analysis.

$$F_s = D_{\text{ref}} (\text{Log } a - \text{Log } b) \quad [6]$$

**Stimulation Approaches.** Chandarana *et al.* (1987a, 1987b) and Chandarana and Gavin (1989) have discussed three possible approaches to scheduling commercial processes for foods containing particulates processed aseptically. "Total" approach accounts for the process where particulate thermal and lethality credit can be achieved both in the SSHE and the holding tube. The second approach, "F<sub>0</sub> Hold", includes the thermal credit in the SSHE, no lethality credit in the SSHE, while including both thermal and lethality credit in the holding tube. The last approach, "Hold Only", excludes the thermal and lethality contributions of the SSHE and only includes the thermal and lethality accumulation in the holding tube. In addition, it should be noted that the model assumes no phase transitions during processing.

### Sensitivity Analysis

The essence of the sensitivity analysis is to introduce small perturbations in the various input parameters of the model and to study their relative effects on the output variables of interest (Kanwar and Johnson 1983). Three main output variables selected for the sensitivity analysis are the holding tube length required to destroy 6 D of *Clostridium sporogenes* (PA3679), retention of thiamine and peroxidase in the particle after processing depending on parameter variations and simulation approaches. The numerical values of the process parameters under study were changed in certain expected range and the analyses were conducted by comparing results from reference conditions given in Table 2. The range of parameters selected for the analysis is given in Table 3 and discussed in the following two sections.

**Product Parameters.** Particle size of 0.64 to 1.91 cm side cube was considered in the analysis. It seems to cover the general size range of particulates to be processed aseptically in an SSHE. Realistically, the maximum size cube to be processed in the present system would be the one with 1.91 cm sides considering its diagonal length with clearance in the SSHE. Particle loading was varied 10 to 30% of particulates, which seems to be the range where the pump works effectively.

Previous simulation study indicated that particle thermal properties greatly influenced the minimum process time to achieve 6 D for *Clostridium sporogenes* (PA3679) (Lee *et al.* 1990). However, the effect of individual variation in the

TABLE 3.  
RANGES OF PARAMETERS AND SPECIFICATIONS  
USED IN SENSITIVITY ANALYSIS

Parameter	Specifications
<b>PRODUCT</b>	
Particle size	0.64 - 1.91 cm (0.25 - 0.75 in)
Particle loading	10 - 30%
Particle thermal conductivity	0.500 - 0.611 W/m °C (0.289 - 0.353 BTU/h ft °F)
Particle density	1049 - 1092 kg/m <sup>3</sup> (65.46 - 68.14 lb/ft <sup>3</sup> )
Particle specific heat	3.20 - 3.34 kJ/kg °C (0.765 - 0.797 BTU/lb °F)
Fluid thermal conductivity	0.606 - 0.684 W/m °C (0.350 - 0.395 BTU/h ft °F)
Fluid density	917 - 997 kg/m <sup>3</sup> (57.2 - 62.3 lb/ft <sup>3</sup> )
Fluid viscosity	185 - 881 x 10 <sup>-6</sup> Pa s (0.185 - 0.881 cp)
<b>PROCESS</b>	
Product initial temperature	26.67 - 65.56 °C (80 - 150 °F)
Product flow rate	0.34 - 0.57 m <sup>3</sup> /h (1.5 - 2.5 GPM)
Rotor speed	100 - 200 rpm
Velocity ratio <sup>a</sup> in SSHE	0.5 - 1.0
Velocity ratio in holding tube	0.5 - 1.0
Particle surface convective heat transfer coefficient in SSHE	114 - 454 W/m <sup>2</sup> K (20 - 80 BTU/h ft <sup>2</sup> °F)
Particle surface convective heat transfer coefficient in holding tube	57 - 397 W/m <sup>2</sup> K (10 - 70 BTU/h ft <sup>2</sup> °F)

$$^a \text{Velocity ratio} = V_{\text{avg}}/V_{\text{max}}$$

properties such as thermal conductivity, density and specific heat was not investigated. The properties of potato were varied within a reasonable range by considering their variations due to the temperature change during process from the data given by Yamada (1970). For example, variation in the values of thermal conductivity, density and specific heat were  $\pm 10\%$  and  $\pm 2\%$  from the reference conditions, respectively.

Fluid properties such as thermal conductivity, density and viscosity were also considered. Properties of water are given by Heldman and Singh (1981) covering variations due to temperature change from 25 to 150 °C.

**Process Parameters.** In actual process, multiple SSHEs may be used to preheat the product. To simulate this process, variation of product initial temperature from 27 to 66 °C was investigated.

Little information is available on residence time distribution in SSHEs and holding tube for foods containing particulates. Taeymans *et al.* (1985a, 1985b) indicated that the flow pattern of the liquids containing solid particles (calcium alginate beads with water) deviated from the ideal piston flow. Recently, Berry (1989) studied residence time distribution of particles in a holding tube. The



particles used were 6, 10 and 13 mm rubber cubes (Linard, Linatex Corp. of America) with a specific gravity of 1.08. The fluid was a 1.5% carboxymethylcellulose (Ticalose CMC 5000 R, TIC Gums, Inc.) solution supplemented with 6% (by weight) rubber cubes. The ratio of the fastest particle residence time (FPRT) to bulk flow residence time in a holding tube remained fairly constant with values of 0.63, 0.56 and 0.59 for flow rates of 4, 8 and 12 L/min, respectively. FPRT ratios less than 0.5 were not observed, indicating that no particle traveled twice as fast as the bulk fluid. Sastry (1989) observed particles moving up to about 1.85 times the mean fluid velocity in the holding tube when polystyrene spherical particles of 95  $\mu$ m diameter and CMC solutions of 0.2, 0.5 and 0.8%, and particle concentrations of 4.5, 9, 13.5 and 18 particles/L were studied. In the present study, the velocity ratio,  $V_{avg}/V_{max}$ , for both SSHE and holding tube ranged from 0.5 to 1.0.

Another area of lacking information is that of the particle/fluid interface convective heat transfer coefficient. Limited data from Chandarana (1987) suggested a value of 284  $W/m^2C$ . Chandarana (1988) further investigated the coefficient for food particles immersed in static water and non-Newtonian fluids (2–5% Thermflo starch solutions) and found that the coefficient between potato cube and starch solution ranged from 84 to 94  $W/m^2C$  at 131°C depending on the particle size while the value between carrot cube and water was 389  $W/m^2C$  at 111°C. Chang and Toledo (1989) reported the heat transfer coefficients between surface of a sweet potato cube and water were 239 and 303  $W/m^2C$  at 0 and 0.86 cm/s relative velocity in an isothermal holding tube while it was 146  $W/m^2C$  in 35% sucrose at relative velocity of 0 cm/s. Lee *et al.* (1990) also indicated that the value of heat transfer coefficient beyond 500  $W/m^2C$  did not significantly reduce the process time required for 6 D process for *Clostridium sporogenes* (PA 3679). Therefore, the range used was 114 to 454  $W/m^2C$  and 57 to 397  $W/m^2C$  for SSHE and holding tube, respectively. Higher magnitude of the coefficient applied for SSHE than that for the holding tube was to account for the turbulence caused by rotation of the blades in an SSHE.

## RESULTS AND DISCUSSION

As the product passes through the aseptic processing system such as SSHE, it receives thermal energy from the heat transfer medium in the heat exchanger which causes the accumulation of lethality during the time it spends in the system. At present there are no effective procedures to assess the lethality accumulation if particles break up during cooling. Thus, it seems not to be prudent to credit any lethality accumulated during cooling from the public health point of view (Dignan *et al.* 1989) and no lethality during cooling was included for all simulations.

**Parameter Sensitivity**

Figures 1 and 2 show change in required holding tube length with respect to change in product parameter for simulation “Total”. Particle size has a considerable impact on required holding tube length. Increase in particle size from 1.27 to 1.91 cm (side of a cube) required 142% increase in holding tube length while decrease from 1.27 to 0.64 cm required 79% reduction in holding tube length to achieve specified sterility. Other parameters such as particle loading, particle thermal conductivity, fluid viscosity and fluid density affected the required holding tube length by approximately 7 to 14% with respect to their change. Next group of variables considered includes particle density, particle specific heat and fluid thermal conductivity whose effect on required holding tube length with respect to the change was less than 4%. It is interesting to observe that the responses are nonlinear, which indicates that there exists an optimum condition that requires a minimum holding tube length. It should be

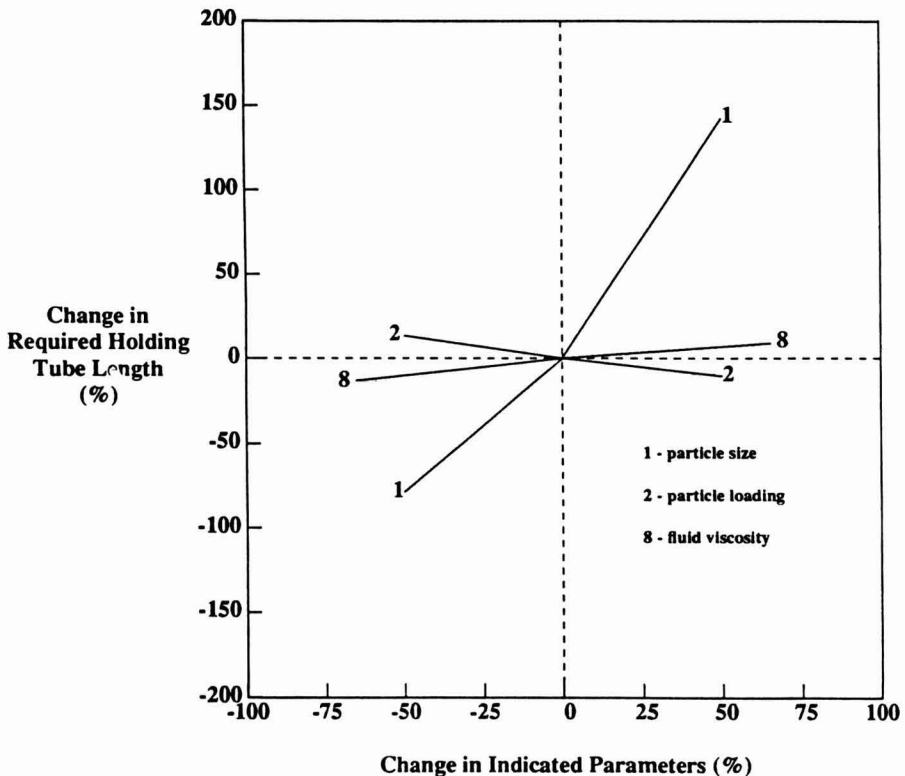


FIG. 1. PRODUCT PARAMETER SENSITIVITY TO HOLDING TUBE LENGTH REQUIRED FOR SIMULATION “TOTAL”

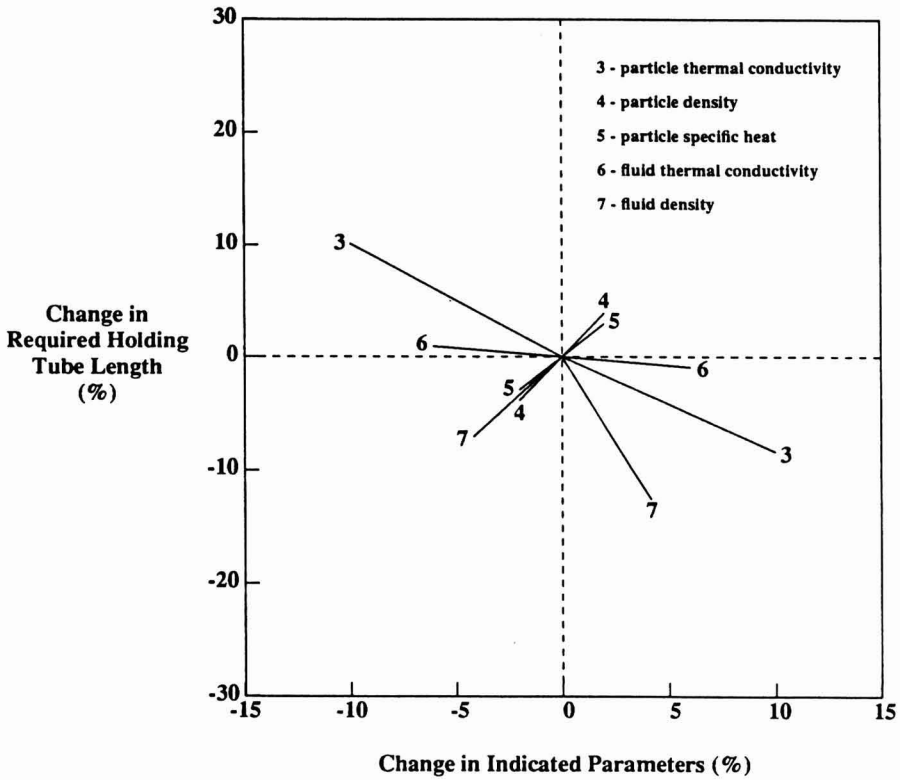


FIG. 2. PRODUCT PARAMETER SENSITIVITY TO HOLDING TUBE LENGTH REQUIRED FOR SIMULATION "TOTAL"

noted that their relative sensitivity could be different since each parameter varied in different range and will be discussed in relative sensitivity section. In addition, results from simulation "F<sub>0</sub> Hold" and "Hold Only" were not plotted due to similar pattern though different magnitude to those of "Total" simulations.

Figure 3 shows process parameter sensitivity to holding tube required for simulation "Total". The effect of change in product flow rate seems to have more influence on the required holding tube length than that of particle size. Increase in product flow rate from 0.45 to 0.57 m<sup>3</sup>/h required 208% longer holding tube while decrease from 0.45 to 0.34 m<sup>3</sup>/h required 87% shorter holding tube. Other parameters such as product initial temperature and velocity ratios in holding tube and SSHE affected approximately 25 to 58% of required holding tube length with respect to their change. Next group includes particle surface convective heat transfer coefficients in SSHE and holding tube and rotor speed

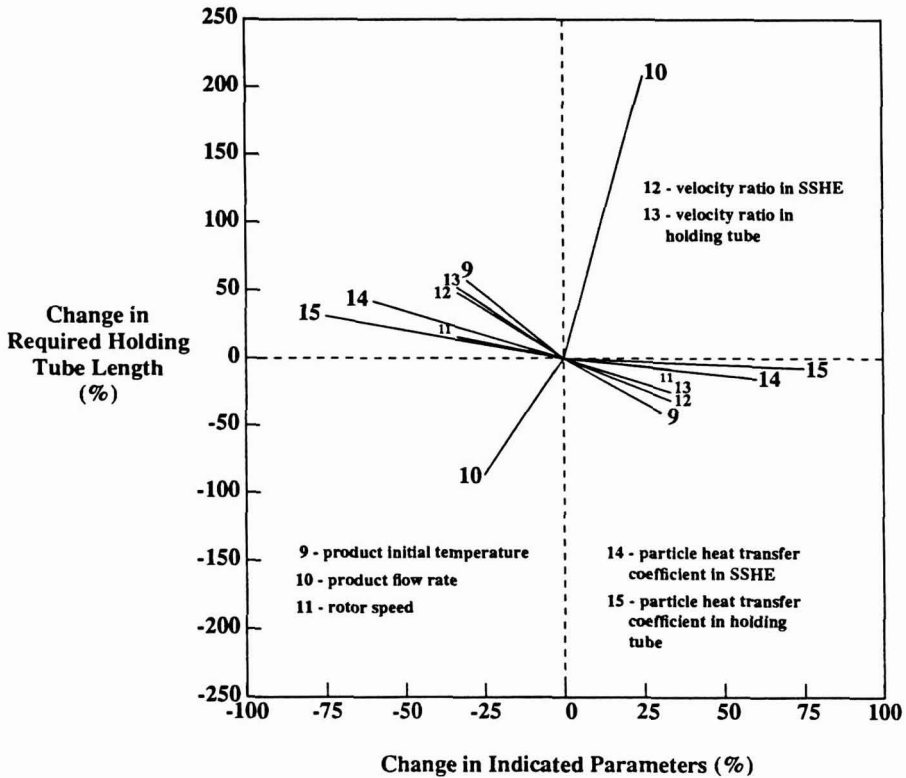


FIG. 3. PROCESS PARAMETER SENSITIVITY TO HOLDING TUBE LENGTH REQUIRED FOR SIMULATION "TOTAL"

whose effect on required holding tube length with respect to the change was between 8 and 42%.

Figures 4 and 5 present change in thiamine retention with respect to the change in product parameters for simulation "Total". It is noticed that the effect of parameters is similar to that for holding tube length required except it is in the opposite direction and of less magnitude. It is, because of considering thiamine retention rather than destruction and differences in  $z$  and  $D$  values, respectively. For example, increase in particle size from 1.27 to 1.91 cm (side of a cube) reduced the thiamine retention by 6% while decrease from 1.27 to 0.64 cm increased the thiamine retention by 4%. Particle thermal conductivity seems to be the next important parameter to impact thiamine retention among product parameters although its effect is small (less than 0.6%). Relative to other product parameters, particle loading and fluid viscosity has no effect on thiamine reten-

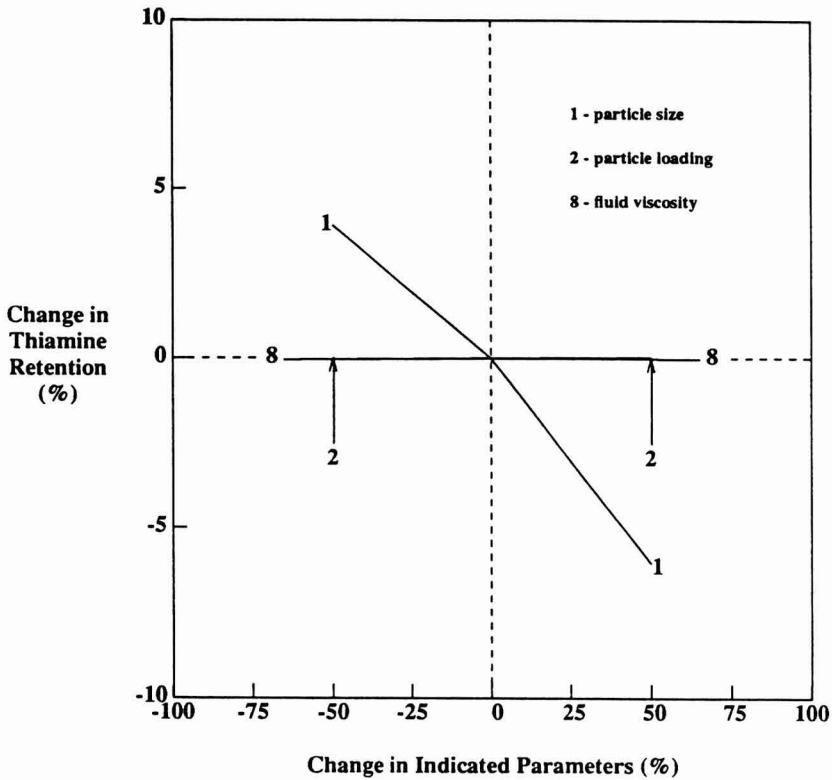


FIG. 4. PRODUCT PARAMETER SENSITIVITY TO THIAMINE RETENTION FOR SIMULATION "TOTAL"

tion. It is also observed that the responses are non-linear. This indicates that an optimum product condition may be found for each product although it does not appear in the range investigated in this study. This implies that product can be formulated in such a way that the thiamine retention can be maximized during the process.

Figure 6 presents change in thiamine retention with respect to the change in process parameters for simulation "Total". Changes in the thiamine retention are relatively small as compared to those of required holding tube length. Product flow rate has relatively large impact while velocity ratio in holding tube and rotor speed has relatively small impact on change in thiamine retention among other process parameters (Table 5). It is noticed that both increase and decrease in the most process parameters reduced thiamine retention indicating that an optimum process parameter combination can be found for each product. This was also reported by Chandarana (1988). Again, change in thermal conductivity of fluid has least effect on thiamine retention among all the parameters.

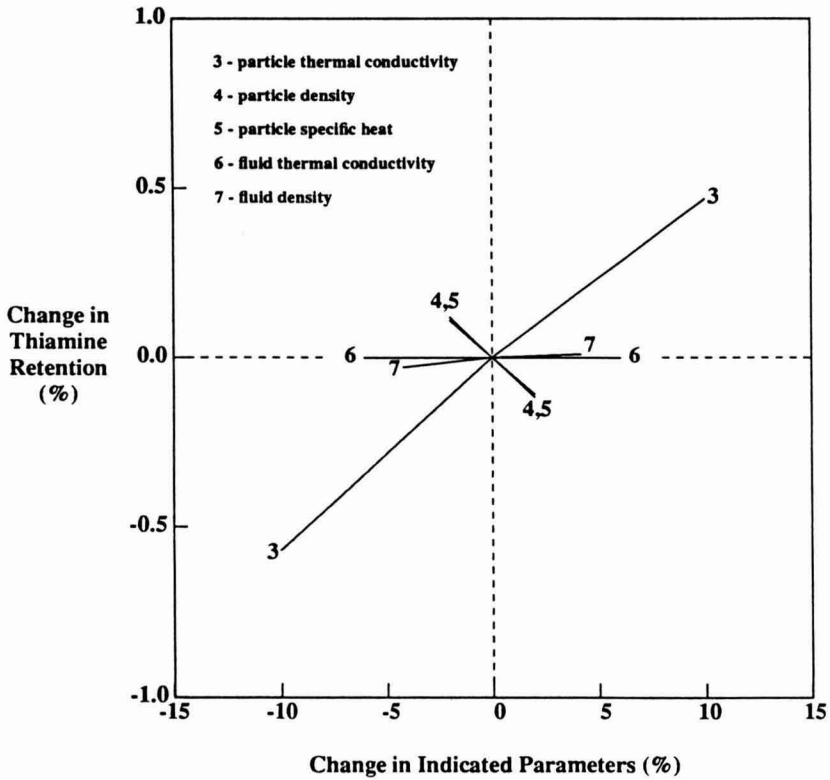


FIG. 5. PRODUCT PARAMETER SENSITIVITY TO THIAMINE RETENTION FOR SIMULATION "TOTAL"

Figures 7 and 9 present change in peroxidase retention with respect to the change in both product and process parameters for simulation "Total". It can be seen that parameter sensitivity to peroxidase retention is in similar direction of results with thiamine retention but magnitude with holding tube length required. Particle size largely affected the peroxidase retention up to 209% when it was decreased from 1.27 to 0.64 cm (side of a cube) while increase from 1.27 to 1.91 cm decreased the peroxidase retention by 81%. Next parameters that affected peroxidase retention considerably among product parameters are particle thermal conductivity, fluid density and particle loading whose effect on peroxidase retention with respect to their change was between 7 to 15%. Again, the fluid thermal conductivity has the least effect on peroxidase retention with its respective change (less than 1%).

Among process parameters, product flow rate has a considerable effect on peroxidase retention followed by particle surface convective heat transfer coefficient in holding tube, product initial temperature and velocity ratio in SSHE.

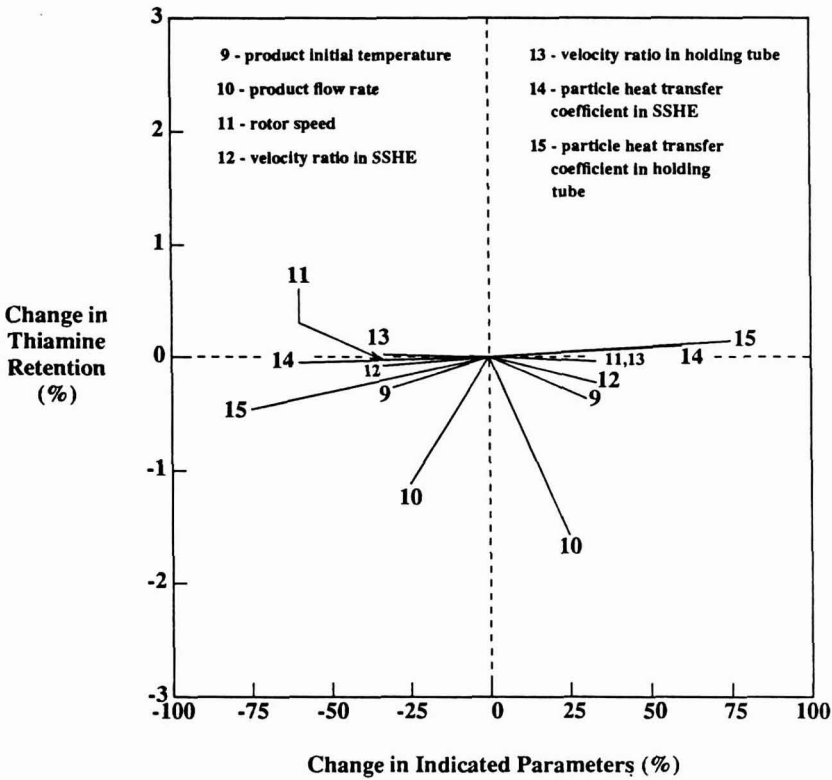


FIG. 6. PROCESS PARAMETER SENSITIVITY TO THIAMINE RETENTION FOR SIMULATION "TOTAL"

Again, it can be seen that the responses are non-linear which implies that there exists a maximum point of peroxidase retention depending on both product and process parameters. This indicates that the process can be optimized for each product by choosing appropriate parameters.

It is worthwhile to note that the properties such as the wall heat transfer coefficient, the particle convective heat transfer coefficient and the residence time distribution are influenced by the change in rheological properties of the carrier medium. However, their relationships are not widely available in the literature at present with an exception of the wall heat transfer coefficient. Therefore, full incorporation of those interactions in the simulation would not be possible. Nevertheless, in an attempt to investigate the effect of parameter interactions on the holding tube length required, thiamine and peroxidase retentions, all the product and process parameters were changed  $\pm 2\%$  from their respective reference value.

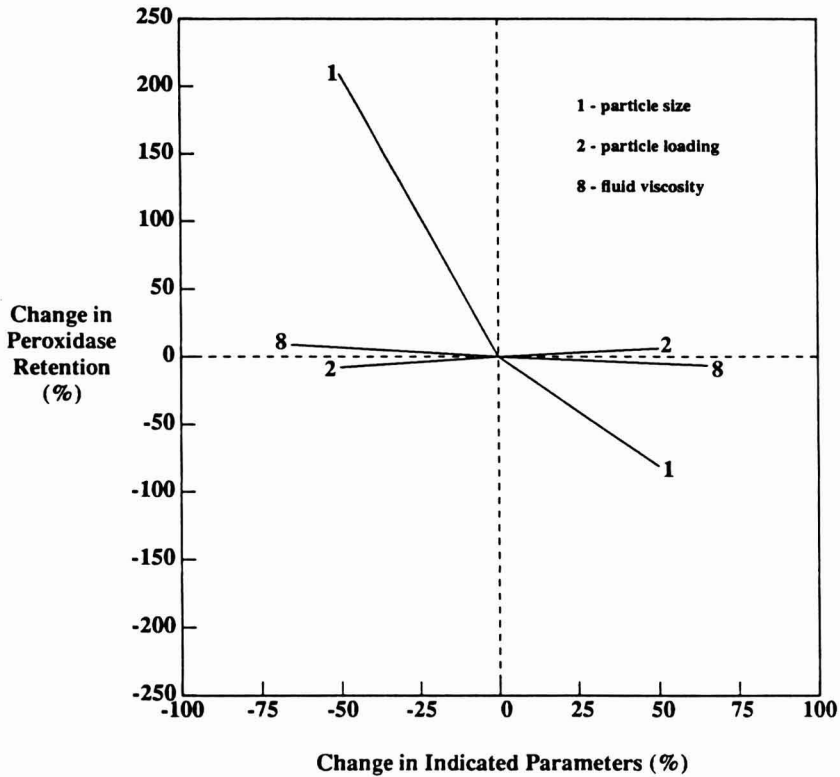


FIG. 7. PRODUCT PARAMETER SENSITIVITY TO PEROXIDASE RETENTION FOR SIMULATION "TOTAL"

The results indicated that 2% increase in all the input parameters resulted in 9% increase in required holding tube length while 2% decrease required 13% less holding tube length for the simulation "Total". Thiamine retention was, however, less affected by the change. Increase and decrease of 2% in all the input parameters affected the thiamine retention by less than 0.3% for the simulation "Total". In addition, peroxidase retention was estimated to be 9% lower when all the input parameters were 2% higher but 15% higher when they were 2% lower for the simulation "Total". This indicates that the errors in input parameters as small as 2% can result in as much as 15% deviation in model prediction. It is clear that these combined errors cause higher deviations as compared to that caused by single input error. For example, a 2% change in particle density or specific heat resulted in about 4 and 3% deviations in the prediction of required holding tube length, respectively for the simulation "Total". The same changes in particle density or specific heat in the prediction of



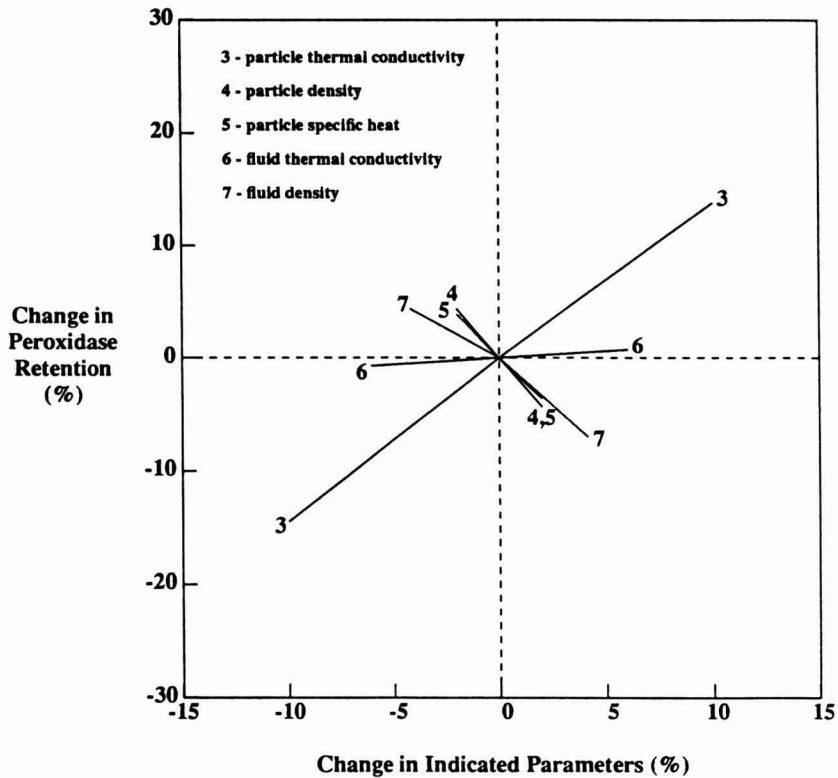


FIG. 8. PRODUCT PARAMETER SENSITIVITY TO PEROXIDASE RETENTION FOR SIMULATION "TOTAL"

thiamine and peroxidase retention resulted in about 0.1 and 4% deviations for the simulation "Total", respectively.

### Relative Sensitivity

Tables 4 through 6 summarize relative sensitivity coefficients for both product and process parameters as well as simulation types, namely, "Total", "F<sub>0</sub> Hold" and "Hold Only". The relative sensitivity coefficient is defined as the ratio of change in response to change in the input parameter. Upper value indicates its relative coefficient when parameter is subject to a decrease with respect to the reference while lower value indicates its relative coefficient when parameter is subject to an increase with respect to the reference. In addition, minus sign indicates direction of change in the response with respect to opposite change in the parameter.

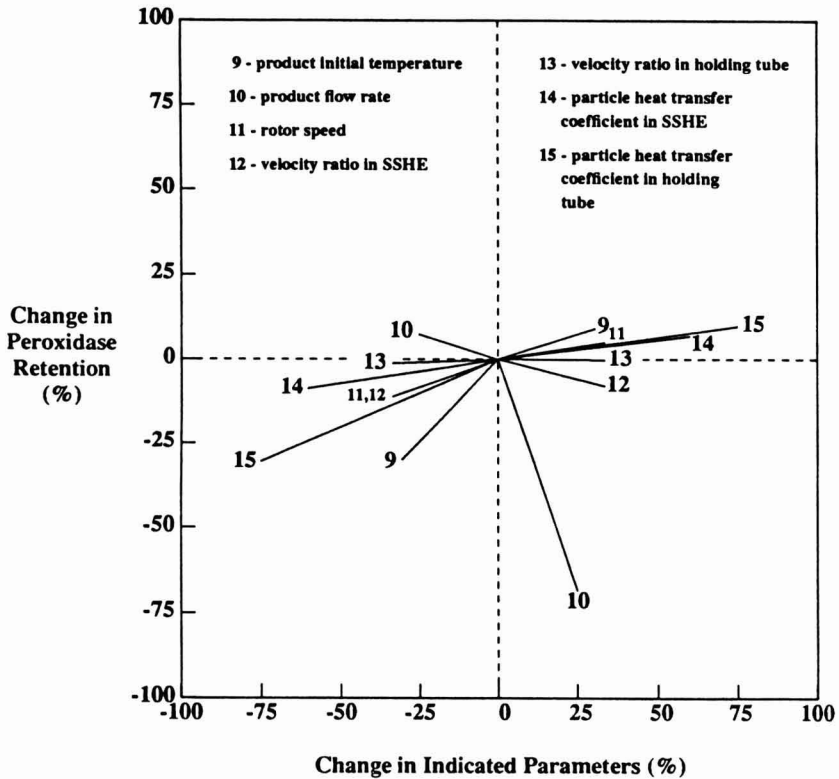


FIG. 9. PROCESS PARAMETER SENSITIVITY TO PEROXIDASE RETENTION FOR SIMULATION "TOTAL"

Table 4 presents relative sensitivity coefficients for holding tube length required. It indicates that there is no noticeable difference between simulation of "Total" and "F<sub>0</sub> Hold". This may be expected since only difference between two approaches is that "F<sub>0</sub> Hold" approach does not allow lethality credit in the SSHE while both allow thermal and lethality credits in the holding tube. There seems to be a smaller relative sensitivity coefficient when simulation "Hold Only" was used. It is, because changes in the required holding tube with respect to the same change in input parameters were small.

For the simulation "Total", fluid density, size, density and specific heat of particle seem to be the most critical parameters to affect the holding tube length required whereas particle loading and fluid viscosity seem to be the least critical parameters among the product parameters considered in this study. Among the process parameters, product flow rate is most likely to influence holding tube length required. Also product initial temperature, velocity ratios in holding tube

TABLE 4.  
RELATIVE SENSITIVITY COEFFICIENTS FOR  
HOLDING TUBE LENGTH REQUIRED

Parameter	Simulation Approach		
	Total	F <sub>0</sub> Hold	Hold Only
<b>PRODUCT</b>			
1. Particle size	1.57 <sup>a</sup>	1.43	0.68
	2.84 <sup>b</sup>	2.84	1.10
2. Particle loading	-0.27 <sup>c</sup>	-0.27	0.45
	-0.21	-0.20	1.83
3. Particle thermal conductivity	-1.01	-1.01	-0.84
	-0.84	-0.80	-0.39
4. Particle density	1.93	1.69	0.93
	1.96	2.96	0.93
5. Particle specific heat	1.47	1.24	0.58
	1.48	1.48	3.18
6. Fluid thermal conductivity	-0.16	-0.16	-0.17
	-0.16	-0.16	-0.12
7. Fluid density	1.69	1.69	0.95
	-3.01	2.60	3.10
8. Fluid viscosity	0.20	0.20	0.17
	0.14	0.14	0.13
<b>PROCESS</b>			
9. Product initial temperature	-1.88	-1.88	-3.60
	-1.34	-1.31	-1.18
10. Product flow rate	3.47	3.26	2.17
	8.35	8.35	9.05
11. Rotor speed	-0.48	-0.48	-0.47
	-0.24	-0.24	-0.21
12. Velocity ratio <sup>d</sup> in SSHE	-1.45	-1.45	-0.49
	-0.95	-0.91	-0.26
13. Velocity ratio in holding tube	-1.56	-1.56	-4.36
	-0.76	-0.76	-1.11
14. Particle surface convective heat transfer coefficient in SSHE	-0.69	-0.69	0.03
	-0.26	-0.25	-0.01
15. Particle surface convective heat transfer coefficient in holding tube	-0.42	-0.43	-1.02
	-0.10	-0.10	-0.15

<sup>a</sup> Upper value indicates its relative coefficient when parameter is subject to a decrease with respect to the reference.

<sup>b</sup> Lower value indicates its relative coefficient when parameter is subject to an increase with respect to the reference.

<sup>c</sup> Minus sign indicates direction of change in the response with respect to opposite change in the parameter.

<sup>d</sup> Velocity ratio =  $V_{avg}/V_{max}$

and SSHE seem to be the second most important parameters to influence holding tube length required. The least critical parameters that affect holding tube length required are particle surface convective heat transfer coefficient in holding tube and rotor speed.

TABLE 5.  
RELATIVE SENSITIVITY COEFFICIENTS FOR  
THIAMINE RETENTION

Parameter	Simulation Approach		
	Total	F <sub>0</sub> Hold	Hold Only
<b>PRODUCT</b>			
1. Particle size	-0.08 <sup>a</sup> -0.12 <sup>bc</sup>	-0.06 -0.12	-0.02 -0.08
2. Particle loading	0.00 0.00	0.00 0.00	-0.03 -0.11
3. Particle thermal conductivity	0.06 0.05	0.06 0.04	0.06 0.03
4. Particle density	-0.05 -0.06	-0.03 -0.06	-0.04 -0.03
5. Particle specific heat	-0.06 -0.06	-0.04 -0.06	-0.02 -0.18
6. Fluid thermal conductivity	0.00 0.00	0.00 0.00	0.01 0.00
7. Fluid density	0.01 0.00	0.01 -0.01	-0.02 -0.15
8. Fluid viscosity	0.00 0.00	0.00 0.00	-0.01 -0.01
<b>PROCESS</b>			
9. Product initial temperature	0.01 -0.01	0.01 -0.02	0.17 0.00
10. Product flow rate	0.04 -0.06	0.10 -0.06	0.17 -0.30
11. Rotor speed	0.00 0.00	0.00 0.00	0.02 0.01
12. Velocity ratio <sup>d</sup> in SSHE	0.00 -0.01	0.00 -0.01	0.00 -0.04
13. Velocity ratio in holding tube	0.00 0.00	0.00 0.00	0.11 0.03
14. Particle surface convective heat transfer coefficient in SSHE	0.00 0.00	0.00 0.00	-0.02 -0.01
15. Particle surface convective heat transfer coefficient in holding tube	0.01 0.00	0.01 0.00	0.03 0.00

<sup>a</sup> Upper value indicates its relative coefficient when parameter is subject to a decrease with respect to the reference.

<sup>b</sup> Lower value indicates its relative coefficient when parameter is subject to an increase with respect to the reference.

<sup>c</sup> Minus sign indicates direction of change in the response with respect to opposite change in the parameter.

<sup>d</sup> Velocity ratio =  $V_{avg}/V_{max}$

It is interesting to notice that the order of relative sensitivity coefficient of parameters is little different when simulations "Total" and "Hold Only" are compared (Table 4–6). Although the order of relative sensitivity coefficient of most of the product and process parameters remains almost the same, a noticeable

difference is found when velocity ratios in both SSHE and holding tube are considered. Velocity ratio in SSHE seems to be relatively more sensitive parameter to affect the holding tube length required for simulation "Total" than that for the simulation "Hold Only". In contrast, velocity ratio in holding tube becomes relatively more critical to influence the holding tube length required for simulation "Hold Only" than that for the simulation "Total". This is expected since simulation "Hold Only" considered the thermal and lethality accumulation only in the holding tube while simulation "Total" allowed thermal and lethality credits both in the SSHE and the holding tube. In other words, residence time distribution directly related by velocity ratio in SSHE did not affect the lethality accumulation when simulation "Hold Only" was considered to determine the holding tube length required. The velocity ratio in the holding tube became less important when simulation "Total" was considered probably because much of the lethality was accumulated before the particles entered the holding tube.

Relative sensitivity coefficients for thiamine retention are given in Table 5. Again, it indicates that there is no noticeable difference between simulation of "Total" and "F<sub>0</sub> Hold". In addition, the coefficients are relatively smaller than those in Table 4, which indicates that changes in destruction of thiamine with respect to the change in input parameters were less affected by both product and process parameters than those of destruction of *Clostridium sporogenes* (PA3679). Nevertheless, size, specific heat, density and thermal conductivity of the particles and the product flow rate and initial temperature seem to be the critical parameters in thiamine retention for the simulation "Total". Fluid viscosity, particle loading, particle surface convective heat transfer coefficient in SSHE and rotor speed are the least critical parameters to affect the thiamine retention for the simulation "Total". The effect of input parameters on the thiamine retention is similar to those of the holding tube length required in the simulations "Total" and "Hold Only".

Finally, relative sensitivity coefficients for peroxidase retention are summarized in Table 6. Generally, the coefficients are higher for simulation "Hold Only" while values for simulation "Total" and "F<sub>0</sub> Hold" are similar indicating that "Hold Only" approach appears to show more changes in peroxidase retention in the particles with respect to the same change in input parameters. This is because of excluding the thermal and lethality contributions of the SSHE while only including the thermal and lethality accumulated in the holding tube. For simulation "Total", size, density, specific heat of particle and fluid density seem to be the critical parameters among product parameters. Critical parameters among process parameters include product flow rate and product initial temperature. Fluid thermal conductivity, fluid viscosity, particle loading, particle thermal conductivity and velocity ratio in the holding tube are the least critical parameters to affect peroxidase retention for the simulation "Total". Again, the

TABLE 6.  
RELATIVE SENSITIVITY COEFFICIENTS FOR  
PEROXIDASE RETENTION

Parameter	Simulation Approach		
	Total	F <sub>0</sub> Hold	Hold Only
<b>PRODUCT</b>			
1. Particle size	-4.19 <sup>a</sup>	-2.96	-0.91
	-1.62 <sup>bc</sup>	-1.62	-1.44
2. Particle loading	0.16	0.16	-2.81
	0.13	0.10	-1.91
3. Particle thermal conductivity	1.45	1.45	2.19
	1.38	1.23	1.09
4. Particle density	-2.17	-1.45	-2.34
	-2.17	-2.17	-1.56
5. Particle specific heat	-1.94	-1.21	-1.56
	-1.82	-1.82	-8.60
6. Fluid thermal conductivity	0.12	0.12	0.52
	0.12	0.12	0.52
7. Fluid density	-1.04	-1.04	-2.60
	-1.68	-1.97	-7.84
8. Fluid viscosity	-0.14	-0.11	-0.57
	-0.10	-0.10	-0.34
<b>PROCESS</b>			
9. Product initial temperature	0.99	0.99	3.13
	0.30	0.19	2.67
10. Product flow rate	-0.30	0.81	0.62
	-2.74	-2.74	-3.94
11. Rotor speed	0.33	0.33	1.08
	0.15	0.15	0.70
12. Velocity ratio <sup>d</sup> in SSHE	0.33	0.33	0.37
	-0.24	-0.36	-0.75
13. Velocity ratio in holding tube	0.04	0.04	2.62
	-0.01	-0.01	2.25
14. Particle surface convective heat transfer coefficient in SSHE	0.15	0.15	-0.86
	0.12	0.09	-0.21
15. Particle surface convective heat transfer coefficient in holding tube	0.41	0.42	1.04
	0.13	0.12	0.31

<sup>a</sup> Upper value indicates its relative coefficient when parameter is subject to a decrease with respect to the reference.

<sup>b</sup> Lower value indicates its relative coefficient when parameter is subject to an increase with respect to the reference.

<sup>c</sup> Minus sign indicates direction of change in the response with respect to opposite change in the parameter.

<sup>d</sup> Velocity ratio =  $V_{avg}/V_{max}$

effect of input parameters on the peroxidase retention is similar to those of the holding tube length required in the simulations "Total" and "Hold Only".

Dignan *et al.* (1989), in what appeared to be the official position of the Food and Drug Administration (FDA), indicated that all credit for lethality be taken exclusively from the holding tube. However, FDA might accept the lethality

accumulated in the heater only when procedures and controls document and assure that delivery of lethality is consistent. Yet there are no reliable experimental methods to differentiate the lethality accumulated during heating from that accumulated during holding and cooling. Therefore, different types of simulation approaches offer a valuable tool to examine these aspects.

## CONCLUSIONS

The sensitivity analysis has demonstrated the relative importance of product and process parameters to predict holding tube length required, thiamine and peroxidase retention depending on simulation types, namely, "Total", "F<sub>0</sub> Hold" and "Hold Only". Basically no difference between the simulation "Total" and "F<sub>0</sub> Hold" was found in model prediction. Although the order of relative sensitivity of parameters remained almost the same, different magnitude of relative sensitivity coefficient existed among results from simulation types and the output variables considered in this study. It is because of different destruction characteristics among microorganism, thiamine and peroxidase as well as the simulation characteristics.

Among product parameters, particle size and particle thermal properties such as density and specific heat were sensitive to influence holding tube length required, thiamine and peroxidase retention (within the range investigated) while fluid properties such as fluid thermal conductivity and viscosity were less susceptible to affect the model prediction regardless of simulation types. In addition, product flow rate and product initial temperature seemed to be the most critical parameters within the range covered in this study while rotor speed seemed to be one of the least critical parameters among all the process parameters considered regardless of simulation types. If the velocity ratio (average velocity divided by the maximum velocity) in SSHE was more critical, the velocity ratio in holding tube seemed to be less critical parameter and vice versa depending on the simulation types due to the simulation characteristics.

This study provides insight into the possible margin of errors in the model predictions due to inaccurate estimates of the input parameters. The input parameters of the model can be divided into broad classes of sensitivity according to their specific application.

## NOMENCLATURE

### Symbols

- a = initial concentration of target factor (e.g., microorganism, nutrient or enzyme)  
A<sub>c</sub> = cooler incremental surface area, m<sup>2</sup>

- $A_{hd}$  = holding tube incremental surface area,  $m^2$   
 $A_{ht}$  = heat exchanger incremental length surface area,  $m^2$   
 $A_p$  = particle surface area,  $m^2$   
 $b$  = final concentration of target factor (e.g., microorganism, nutrient or enzyme)  
 $C_{p_f}$  = carrier fluid specific heat,  $kJ/kg \text{ } ^\circ C$   
 $C_{p_p}$  = particle specific heat,  $kJ/kg \text{ } ^\circ C$   
 $D$  = decimal reduction time, min  
 $D_{121.1^\circ C}$  = decimal reduction time at  $121.1 \text{ } ^\circ C$ , min  
 $D_{ref}$  = decimal reduction time at the reference temperature, min  
 $F$  = number of minutes required to destroy a given number of organisms at a given temperature, min  
 $F_s$  = integrated lethality, min  
 $h_p$  = particle surface heat transfer coefficient,  $W/m^2 \text{ } ^\circ C$   
 $k$  = thermal conductivity,  $W/m \text{ } ^\circ C$   
 $m_f$  = fluid mass flow rate,  $kg/h$   
 $N_p$  = number of particles in incremental length  
 $t$  = time, s  
 $T_c$  = environmental (ambient) temperature,  $^\circ C$   
 $T_f^t$  = fluid temperature at time "t",  $^\circ C$   
 $T_f^{t+1}$  = fluid temperature at time "t + 1",  $^\circ C$   
 $T_i$  = initial batch temperature,  $^\circ C$   
 $T_o$  = particle center temperature,  $^\circ C$   
 $T_p$  = particle temperature,  $^\circ C$   
 $T_{pm}$  = mean particle surface temperature,  $^\circ C$   
 $T_{ref}$  = reference temperature,  $^\circ C$   
 $T_s$  = steam temperature,  $^\circ C$   
 $T_w$  = cooling water temperature,  $^\circ C$   
 $U_c$  = cooler overall heat transfer coefficient,  $W/m^2 \text{ } ^\circ C$   
 $U_{hd}$  = holding tube overall heat transfer coefficient,  $W/m^2 \text{ } ^\circ C$   
 $U_{ht}$  = heat exchanger overall heat transfer coefficient,  $W/m^2 \text{ } ^\circ C$   
 $V_{avg}$  = average velocity,  $m/s$   
 $V_{max}$  = velocity of fastest particle,  $m/s$   
 $x, y, z$  = space coordinates  
 $z$  = number of  $^\circ C$  required for the thermal death time curve to traverse one logarithmic cycle,  $^\circ C$   
 $\rho$  = density,  $kg/m^3$

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