FNP

JOURNAL OF FOOD PROCESSING AND PRESERVATION

D.B. LUN
EDITOR

FOOD & NUTRITION PRESS, INC.

UME 11, NUMBER 2 JUNE 198

JOURNAL OF FOOD PROCESSING AND PRESERVATION

All articles for publication and inquiries regarding publication should be sent to Dr. D. B. Lund, University of Wisconsin, Department of Food Science, 1605 Linden Drive, Madison, Wisconsin 53706 USA. There are no page charges for publication in the *Journal of Food Processing and Preservation.*

All subscriptions and inquiries regarding subscriptions should be sent to Food & Nutrition Press, Inc., P.O. Box 71, Westport, Connecticut 06881 USA.

One volume of four issues will be published annually. The price for Volume 11 is $85.00 which includes postage to U.S., Canada, and Mexico. Subscriptions to other countries are $99.00 per year via surface mail, and $107.00 per year via airmail.

Subscriptions for individuals for their own personal use are $65.00 for Volume 11 which includes postage to U.S., Canada, and Mexico. Personal subscriptions to other countries are $79.00 per year via surface mail, and $87.00 per year via airmail. Subscriptions for individuals should be sent direct to the publisher and marked for personal use.

The *Journal of Food Processing and Preservation* is listed in *Current Contents/Agriculture, Biology & Environmental Sciences (CC/AB).*

The *Journal of Food Processing and Preservation* (ISSN: 0145-8892) is published quarterly by Food & Nutrition Press, Inc. — Office of Publication is 155 Post Road East, Westport, Connecticut 06881 USA.

Second class postage paid at Westport, CT 06881.

POSTMASTER: Send address changes to Food & Nutrition Press, Inc., P.O. Box 71, Westport, CT 06881.

JOURNAL OF FOOD PROCESSING AND PRESERVATION

Journal of FOOD PROCESSING and PRESERVATION

VOLUME 11
NUMBER 2

Editor: D. B. LUND

FOOD & NUTRITION PRESS, INC.
WESTPORT, CONNECTICUT 06880 USA

Food & Nutrition Press, Inc.
Westport, Connecticut 06880 USA

ISSN: 0145-8892

Printed in the United States of America

CONTENTS

CHANGES IN TENDERNESS DURING AGING OF VACUUM-PACKAGED BEEF

M. C. LANARI, A. E. BEVILACQUA and N. E. ZARITZKY

Centro de Investigación y Desarrollo en Criotecnologia de Alimentos CIDCA. Facultad de Ciencias Exactas Universidad Nacional de La Plata Calle 47 y 116 La Plata (1900). Pcia. Bs. As. ARGENTINA

Accepted for Publication September 29, 1986

ABSTRACT

The variation of tenderness during aging of vacuum-packaged beef at different storage temperatures (0, 4, 10 and 13°C) was determined. Tensile and Warner Bratzler (WB) shear measurements were used to assess the tenderness of raw and cooked beef (M. semitendinosus). Coefficients of variation corresponding to each method demonstrated the usefulness of the WB shear test with cooked beef. An exponential decay equation was used to quantify changes in beef tenderness during aging; rate constants and activation energies for each method were calculated. The model was adequate to represent the time dependence of different aging indicators: Biochemical Index of Myofibrillar Aging (BIMA) and Yield Point (YP).

INTRODUCTION

Aging or conditioning of meat is an important process from a commercial point of view as it improves tenderness. Postmortem tenderization has been studied extensively and has been related to structural changes in myofibrils and to changes in myofibrillar protein composition attributed to the action of endogenous proteolytic enzymes (Penny and Ferguson-Pryce 1979; Penny and Dransfield 1979; Penny 1980; Yates *et al.* 1983; Weber 1984).

Tenderness has been associated with the degree of myofibril fragmentation (Myofibril Fragmentation Index, MFI) after mechanical treatment (Olson *et al.*

Journal of Food Processing and Preservation 11 (1987) 95-109. All Rights Reserved.

1976; Olson and Parrish 1977; Culler*et al.* 1978; Parrish *et al.* 1981). Troponin T (TNT) degradation was also considered an indicator of proteolysis (MacBride and Parrish 1977; Parrish and Cheng 1979; Penny and Dransfield 1979; Penny and Ferguson-Pryce 1979; Yates *et al.* 1983). Many researchers have shown, in postmortem beef muscle, the appearance of a 30 K dalton component (MacBride and Parrish 1977; Olson *et al.* 1977) whose concentration increased during aging (Penny and Ferguson-Pryce 1979; Ouali *et al.* 1983a; Valin *et al.*, 1981 and Ouali 1984) showed that the sensitivity of the Mg-Ca enhanced myofibrillar ATPase activity to ionic strength is a good indicator of the degree of aging (Biochemical Index of Myofibrilar Aging, BIMA).

Meat tenderness has been assessed either by taste-panel sensory analysis or by different instrumental methods. The Warner Bratzler (WB) shear test is one of the techniques most commonly employed for testing meat texture. However, reported correlations between shear values and taste-panel determinations varied from high to low significant values (Szczesniak and Torgeson 1965a). Results from different authors (Bouton and Harris 1972a; Penfield and Meyer 1975) have indicated that peak shear-force values are more closely related to the myofibrillar component of toughness than to the connective-tissue factor (Bouton and Harris 1972b). Shear force correlates poorly with subjective assessments of tenderness when there are large differences in connective-tissue strength among samples (Bouton *et al.* 1973; Penfield and Meyer 1975). In an effort to predict taste-panel response to cooked meat, Stanley *et al.* (1971) studied the tensile properties of meat in raw tissues. Both raw and cooked meat were employed by Eino and Stanley (1973), Bouton and Harris (1972a, 1972b, 1972c) and Bouton *et al.* (1975) in their experiments.

Warner Bratzler and tensile tests were used in the analysis of postmortem changes in muscle to determine the effect of meat aging and its relation to tenderness (Bouton *et al.* 1977; Olson *et al*. 1976; Olson and Parrish 1977; MacBride and Parrish 1977). Locker and Wild (1981, 1982) reported that yield point (YP) can be considered a useful parameter for assessing meat tenderness, particularly in aging studies. Influence of storage temperature on the rate of tenderizing was studied by Penny and Dransfield (1979) and Dransfield *et al.* (1980a, b). Dransfield *et al.* (1980a) considered an exponential decay equation to characterize aging. Parameters of the kinetic model were estimated and their variations regarding animals, muscles, storage temperature and post-slaughter treatments such as electrical stimulation were studied (Dransfield *et al.* 1980a; George *et al.*1980).

Because of the importance of refrigerated beef exports in Argentine economy, a detailed analysis of beef texture is necessary to improve the quality and acceptance of the product. The purposes of this study were: (1) to analyze changes in tenderness during aging of Argentine beef, vacuum-packaged in low-permeability films (EVA-SARAN-EVA), using two different mechanical methods:

tensile and WB shear tests for both raw and cooked meat; (2) to evaluate the parameters of the kinetic model, and (3) to determine aging times at different storage temperatures.

MATERIALS AND METHODS

Samples were obtained from semitendinosus (ST) muscles removed from 39 commercial steer carcasses from a local slaughter-house, classified as U2 grade A according to the Argentine National Meat Board Classification (carcass weight of 220 kg) with a postmortem time of 24 h at 4 °C. Muscle pH ranged from 5.4 to 5.8 measured with an insertion pH meat electrode INGOLD LOT 405-M4 (Switzerland).

Muscles were cut transversely to the fiber axis in sections 6 cm thick for tensile test and 2 cm thick for WB shear test.

Strips for tensile determination had a cross-sectional area of 1 cm^2 and a length of 6 cm in the fiber direction; samples for WB shear test were 1 cm in diameter and 2 cm in length. From each section a minimum of 8 reference samples (tenderness values corresponding to 24 h after slaughter were obtained) the other samples were vacuum packaged in a low permeability film and stored at 0, 4, 10 and 13 °C for different times to measure tenderness changes.

Vacuum Packaging

Samples were packaged under vacuum using a composite EVA/SARAN/EVA, coextruded film, 60μ thickness (trade name Super Cryovac, DAREX SAIC, GRACE; water vapor permeability WVP = 7.2 g/m^2 day at 30 °C and RH = 78%; oxygen transmission rate OTR = 37.5 cm^3/m^2 atm day at 25 °C and RH = 75%) and a single chamber equipment (Minidual, model MW 4980, Schcolnik SAIC, Bs. As.) at 4.5 mm Hg and heat sealing.

Cooking Methods

Samples wrapped in EVA-SARAN-EVA were immersed in a water-bath at 83 °C, heated until the temperature at the center reached 78 °C and then cooled in a water bath at 23 °C for 30 min. Thermocouples Cu-Co (Omega Engineering, Stamford, Conn.) connected to a Data Logger model 2240-C (John Fluke M. Fg. Co., Mountlake Terrace, WA) were used for temperature measurements.

Tenderness Measurements

An INSTRON Universal Testing Machine was used to measure mechanical properties with the following operating parameters: for the WB shear test crosshead

and chart speeds were 10 cm/min each; for the tensile method chart speed was 4 cm/min and crosshead speed 5 cm/min. In both methods the force-deformation curve was recorded at different storage periods, tenderness values were measured as the peak force in raw and cooked beef; a minimum of 8 replicates were performed on each muscle section.

Statistical Methods

Coefficients of variation (C_V) of the mechanical techniques used in the present study were determined. Analysis of variance (ANOVA) was used to ascertain treatment effects and to evaluate standard errors.

RESULTS AND DISCUSSION

Tenderness values corresponding to samples obtained from muscle sections 24 h after slaughter were used to calculate the coefficients of variation (C_V) of each method, in raw and cooked beef (Table 1). Standard deviation of the mean peak force within slices (Box *et al.* 1978) was considered as the experimental error of the method. The lowest C_V value was observed in WB shear test in cooked beef. The decrease in C_V, when the methods were used with cooked beef, can be attributed to partial solubility of collagen produced by cooking (Szczesniak and Torgeson 1965).

TABLE 1
COEFFICIENTS OF VARIATION (C_v) OF EACH METHOD APPLIED TO RAW AND COOKED ST MUSCLE

Method	Ø	C_v %
Tensile test-raw meat	74	20.45
Tensile test-cooked meat	101	11.51
WB test-raw meat	55	15.11
WB test-cooked meat	100	7.51

ϕ = number of tested samples
$C_v\%$ = percent coefficient of variation = $\frac{\text{standard deviation}}{\text{mean value}} \times 100$.

Rate of Tenderizing

The relationships between average peak force (WB and tensile test) and storage time at 0 and 4 °C for different animals are shown in Fig. 1 and 2; similar curves were obtained at 10 and 13 °C.

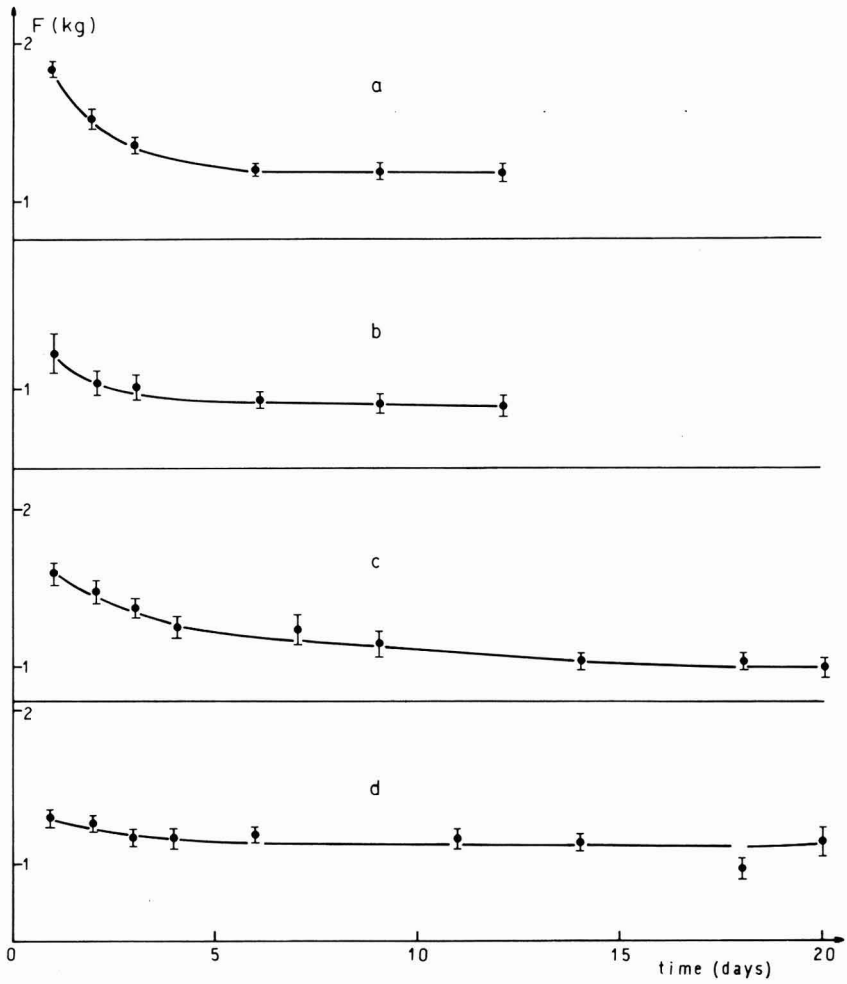

FIG. 1. TIME COURSE OF TENDERNESS DURING AGING OF ST BEEF MUSCLES MEASURED WITH THE TENSILE TEST

Each point represents the mean of N animals.

Cooked muscles stored at: (a) 4 °C, N = 2; (c) 0 °C, N = 4. Raw muscles stored at: (b) 4 °C, N = 3; (d) 0 °C, N = 2. Vertical bars represent standard deviation of mean values.

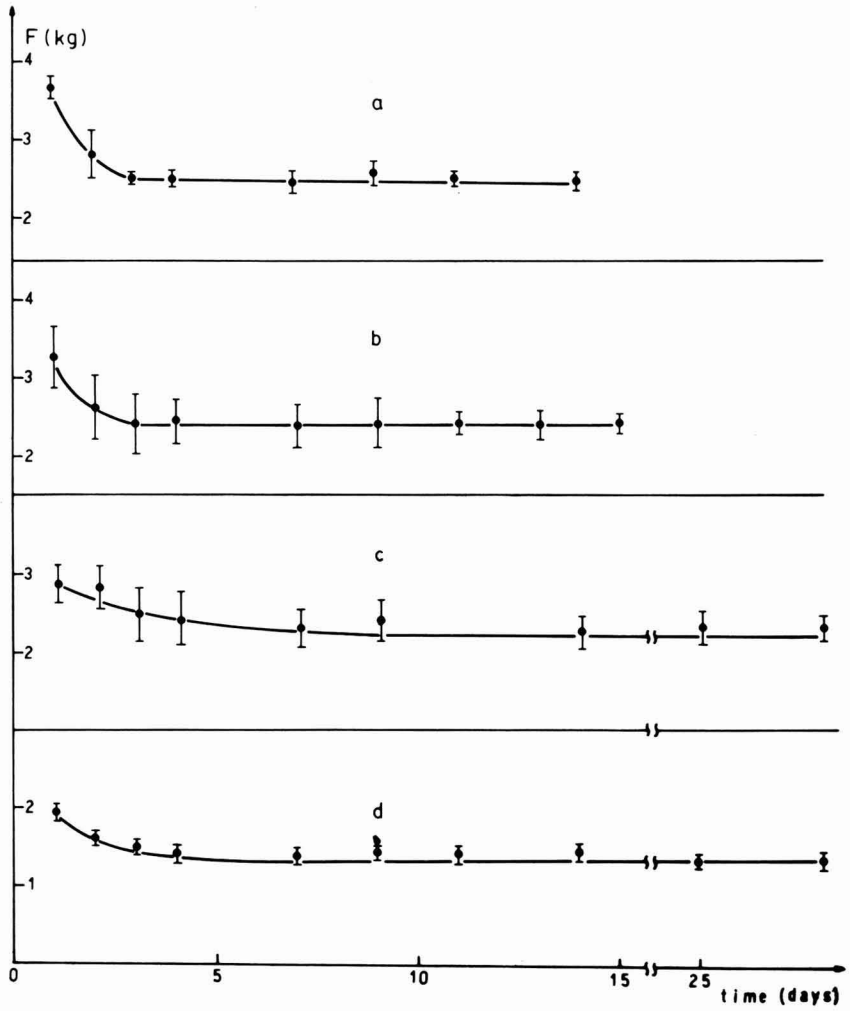

FIG. 2. TIME COURSE OF TENDERNESS DURING AGING OF ST BEEF MUSCLES MEASURED WITH THE WB SHEAR TEST

Each point represents the mean of N animals.

Cooked muscles stored at: (a) 4 °C, N = 3; (c) 0 °C, N = 4; Raw muscles stored at: (b) 4 °C, N =2; (d) 0 °C, N = 3. Vertical bars represent standard deviation of mean values.

A first order kinetic equation was considered for the analysis of the tenderness-storage time relationship:

$$dF/dt = -kF \qquad \text{with } F = F_\infty \text{ at } t = t_\infty$$

$$\text{thus} \quad \ln (F/F_\infty) = k (t_\infty - t)$$

where F and F_∞ are force values (kg) at time t and at the end of the aging process (t_∞) respectively; k is the rate constant ($days^{-1}$).

Influence of animals on F/F_∞ and ln (F/F_∞) values was not significant ($P > 0.05$), therefore samples from animals corresponding to each storage temperature were pooled and common rate constants were calculated.

From the plot ln (F/F_∞) vs. $(t_\infty - t)$ (Fig. 3 and 4), slopes were calculated by the least square regression method obtaining the rate constant k ($days^{-1}$) for each technique and storage temperature (Table 2).

Model fitness was verified by the "lack of fit test" ($P < 0.05$) (Himmelblau 1970). Results of aging time (t_∞) of raw and cooked beef measured by both methods during storage at 0, 4, 10, 13 °C are also shown in Table 2.

Activation Energy

Activation-energy values of the aging process (E_a) and the corresponding standard deviations were calculated for each technique in raw and cooked beef (Table

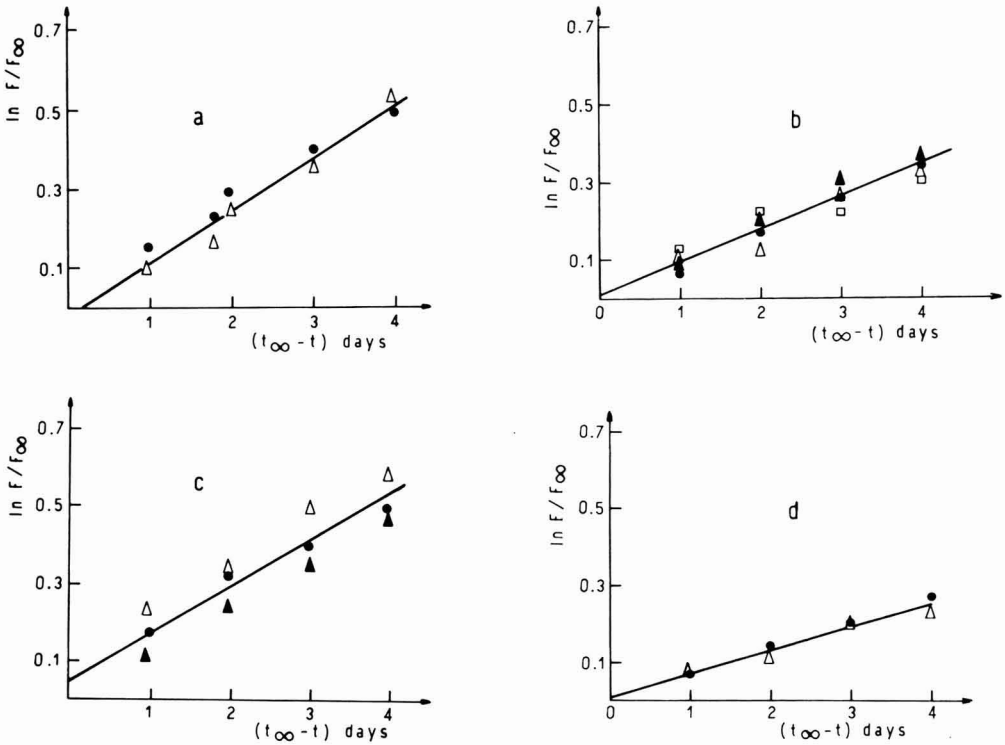

FIG. 3. RELATIONSHIP BETWEEN TENDERNESS (MEASURED WITH THE TENSILE TEST) AND AGING TIME USING THE FIRST ORDER KINETIC MODEL
Cooked beef stored at: (a) 4 °C, (b) 0 °C. Raw beef stored at: (c) 4 °C, (d) 0 °C. Each symbol corresponds to a different animal.

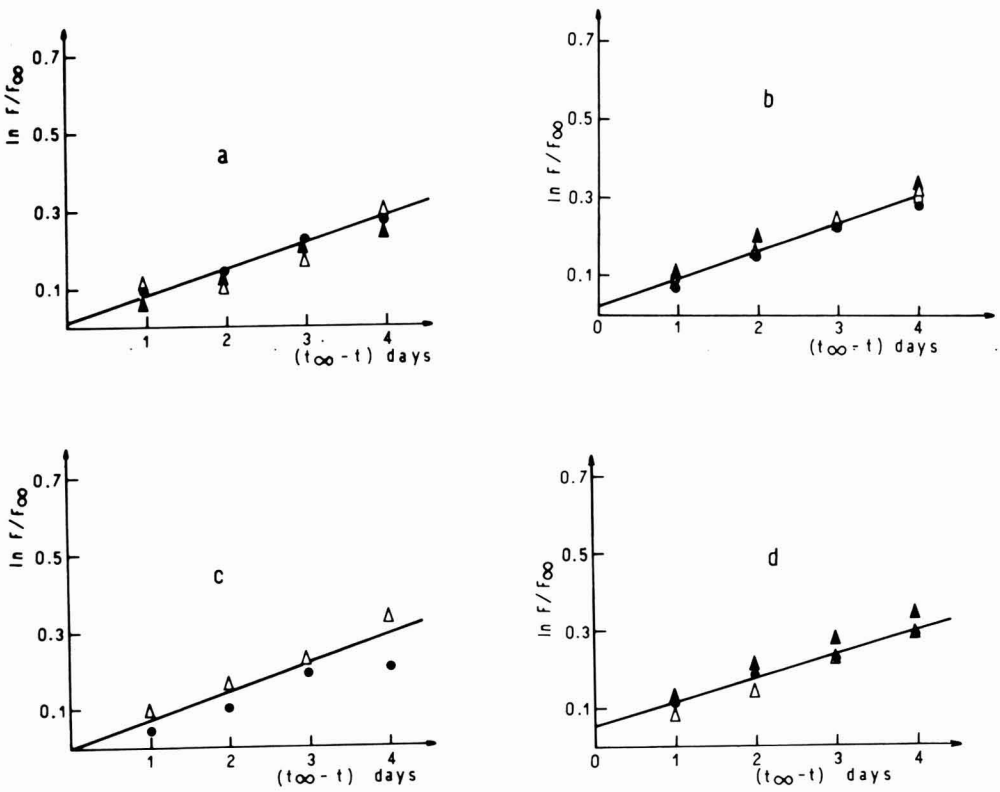

FIG. 4. RELATIONSHIP BETWEEN TENDERNESS (MEASURED WITH THE WARNER-BRATZLER SHEAR TEST) AND AGING TIME USING THE FIRST ORDER KINETIC MODEL

Cooked beef stored at (a) 4 °C, (b) 0 °C. Raw beef stored at: (c) 4 °C, (d) 0 °C. Each symbol corresponds to a different animal.

3) assuming an Arrhenius-type temperature dependence of the rate constants. A typical plot of ln k vs $\frac{1}{T}$ ($°K^{-1}$) corresponding to tensile data is shown in Fig. 5. The values agree with those reported by Davey and Gilbert (1976) (61.6 kJ/mol), Penny and Dransfield (1979) (63 kJ/mol) and Dransfield *et al.* (1980a) (76 kJ/mol, standard deviation (s) = 13 kJ/mol) using compression techniques in cooked beef.

Yates *et al.* (1983) suggested that myosin degradation could be an important factor in the postmortem increase of tenderness, and the loss of troponin T was considered as an indicator of proteolysis. The energy of activation for the loss of troponin T was determined by Penny and Ferguson-Pryce (1979) in raw meat homogenates (E_a = 37 kJ/mol) and by Penny and Dransfield (1979) in cooked

TABLE 2
EFFECT OF TEMPERATURE ON RATE CONSTANTS (K) AND AGING TIME (T_∞)

T(°C)	WB test						Tensile test					
	Raw meat			Cooked meat			Raw meat			Cooked meat		
	N	k	t_∞	N	k	t_∞	N	k	t_∞	N	k	t_∞
0	3	0.068 (0.007)	7	4	0.050 (0.005)	7	2	0.065 (0.005)	9	4	0.083 (0.004)	7
4	2	0.067 (0.015)	3.5	3	0.063 (0.006)	3.5	3	0.110 (0.017)	4	2	0.150 (0.014)	6
10	2	0.107 (0.008)	3	2	0.110 (0.015)	3	2	0.150 (0.014)	3	2	0.210 (0.019)	3
13	2	0.146 (0.060)	2	2	0.178 (0.028)	2	2	0.208 (0.028)	2	2	0.270 (0.025)	2

Standard errors of the rate constants are given in parentheses. N = number of animals used in each experiment.
k = rate constant ($days^{-1}$); t_∞ = aging time (days).

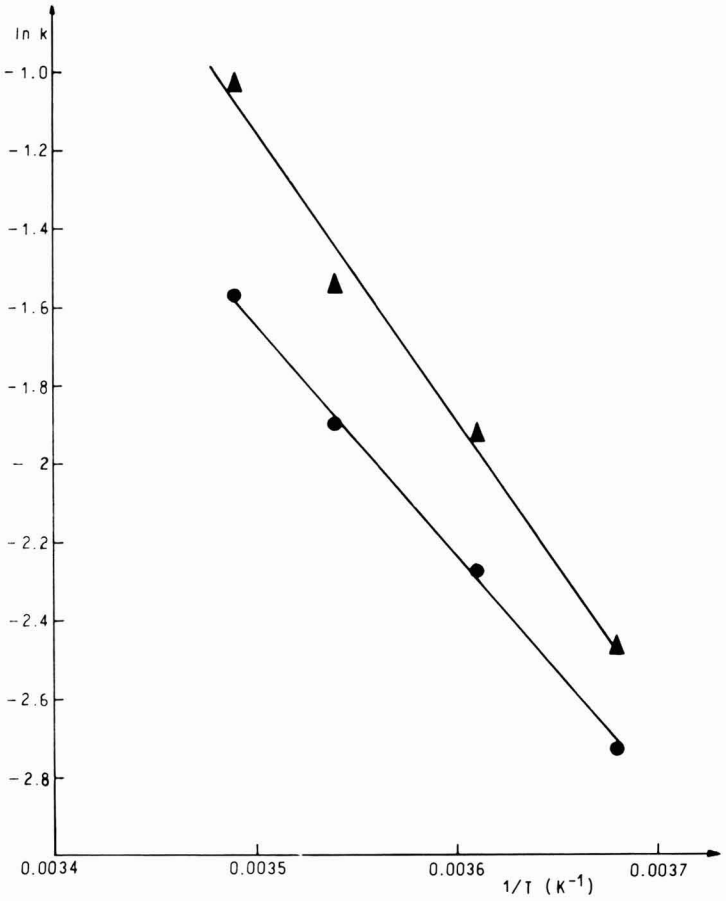

FIG. 5. EFFECT OF TEMPERATURE ON RATE CONSTANTS
Tenderness was measured using the tensile test in cooked (▲) and raw (●) Semitendinosus muscle.

beef (E_a = 73 kJ/mol). Activation-energy values for beef aging obtained in the present study are within the range of the reported data for the loss of troponin T.

Other Aging Indicators

Reported experimental data of Biochemical Index of Myofibrillar Aging (BIMA) (Ouali 1984) and Yield Point (YP) (Locker and Wild 1982) during aging were analyzed applying the exponential decay equation proposed in this work (Fig. 6a and 6b). $BIMA_\infty$ and YP_∞ represented the values of BIMA and YP at the end of the aging process (t_∞).

TABLE 3
ENERGY OF ACTIVATION FOR BEEF AGING DETERMINED BY TENSILE AND WB TESTS

Method		Activation Energy (kJ/mol)
Tensile test	raw beef	47.10 (6.87)
	cooked beef	65.97 (7.99)
WB test	raw beef	45.47 (9.71)
	cooked beef	62.11 (8.35)

Standard deviation of the activation energy values are given in parentheses.

Correlation coefficients were highly significant in both cases. For BIMA (Fig. 6a) $r = 0.997$ ($P < 0.001$) and for YP (Fig. 6b) $r = 0.998$ ($P < 0.005$).

Aging Time

Significant differences among recommended aging times were observed in the literature. Stanley (1976) proposed about 10 to 14 days at 5 °C; Effenberger and Schotte (1972) 5 to 6 days at 0 °C; MacDougall (1971) reviewed the literature and showed that the aging process occurred during the first 14 days at 0.5 °C to −2 °C and that no remarkable changes were observed during the following weeks. Larmond *et al.* (1969) compared aging at 1 °C during 2, 9, and 16 days and noticed that samples stored for 9 days were more tender than those stored for 2 days; no significant differences were observed between samples stored for 16 and 9 days. Dransfield *et al.* (1980b) recommended 14 days at a temperature of 2-4 °C and George *et al.* (1980), 7 days at 1 °C for nonstimulated beef.

Aging times at low temperatures obtained from the present study were 7 to 9 days at 0 °C and 3 to 6 days at 4 °C. These results are in agreement with scanning and transmission electron microscopy observations by Katsaras *et al.* (1984). These authors found that from the sixth day postmortem at 2 °C, intermyofibrillar cohesion was weakened by the partial collapse of transverse structures (T tubules

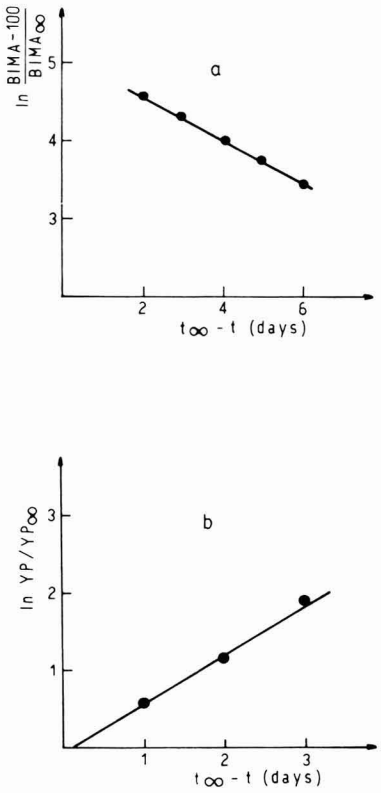

FIG. 6. RELATIONSHIP BETWEEN DIFFERENT AGING INDICATORS AND TIME USING THE PROPOSED KINETICS

(a) Biochemical Index of Myofibrillar Aging (BIMA), data from Ouali (1984); (b) Yield Point (YP), data from Locker and Wild (1982).

and Z discs), connection points between actin filaments and Z lines were destroyed, and collagen fibrils and basal membrane were partially broken down.

Aging periods are determined not only by meat-tenderness changes but also by microbial growth; establishing 10^5 CFU/cm^2 (CFU = colony forming units) as a limit of microbial counts in aged beef, Zamora and Zaritzky (1985) recommended maximum vacuum aging periods of 14 days at 0 °C and 6 days at 4 °C when the pH is lower than 6.0 and initial cell density is approximately 10^4 CFU/cm^2. Considering that initial microbial counts of the samples used in the present study were in the order of 10^4 CFU/cm^2 it can be concluded that during the studied aging periods levels of contamination did not exceed 10^5 CFU/cm^2, leading to a product suitable for direct marketing or subsequent freezing process.

CONCLUSIONS

Results showed that the proposed exponential decay equation can be used effectively to quantify changes in tenderness of raw and cooked meat. The model was also adequate to represent the time dependence of the aging indicators Biochemical Index of Myofibrillar Aging (BIMA) and Yield Point (YP). Aging time assessed at 4 °C was considerably lower than the value reported by Stanley (1976) and Dransfield *et al.* (1980a). Thus, products with lower microbial counts can be obtained and faster marketing procedures are possible. At 0 °C the estimated value of aging time was similar to that proposed by Effenberger and Schotte (1972) and George *et al.* (1980).

Coefficient of variation (C_V) of each instrumental method in the present study showed the convenience of the WB shear test in cooked meat. This technique is also preferable to the tensile test because samples can be readly cut, and more of them can be evaluated for tenderness.

REFERENCES

BOUTON, P. E., FISCHER, A. L., HARRIS, P. V. and BAXTER, R. L. 1973. A comparison of the effect of some post-slaughter treatments on the tenderness of beef. J. Food Technol. *8,* 39-49.

BOUTON, P. E., FORD, A. L., HARRIS, P. V., MAC FARLANE, J. J. and O'SHEA, J. M. 1977. Pressure-heat treatment of post rigor muscle effect on tenderness. J. Food Sci. *42,* 132-135.

BOUTON, P. E. and HARRIS, P. V. 1972a. The effect of cooking temperature and time on some mechanical properties of meat. J. Food Sci. *37,* 140-144.

BOUTON, P. E. and HARRIS, P. V. 1972b. A comparison of some objective methods to assess meat tenderness. J. Food Sci. *37,* 218-221.

BOUTON, P. E. and HARRIS, P. V. 1972c. The effect of some post-slaughter treatments on the mechanical properties of bovine and ovine muscles. J. Food Sci. *37,* 539-542.

BOUTON, P. E., HARRIS, P. V. and SHORTHOSE, W. R. 1975. Changes in shear parameters of meat associated with structural changes produced by aging, cooking and myofibrillar contraction state. J. Food Sci. *40,* 1122-1126.

BOX, G. P., HUNTER, W. H. and HUNTER, J. S. 1978. *Statistics for Experimenters.* pp. 67-68, J. Wiley & Sons, New York.

CULLER, R. D., PARRISH, Jr. F. C., SMITH, G. C. and CROSS, H. R. 1978. Relationship of myofibrillar fragmentation index to certain chemical, physical and sensory characteristic of bovine longissimus muscle. J. Food Sci. *43,* 1177-1180.

DAVEY, C. L. and GILBERT, K. V. 1976. The temperature coefficient of beef aging. J. Sci. Fd. Agric. *27*, 244-250.

DRANSFIELD, E., JONES, R. C. D. and MACFIE, H. J. H. 1980a. Quantifying changes in tenderness during storage of beef. Meat Sci. *5*, 131-137.

DRANSFIELD, E., JONES, R. C. D. and MACFIE, H. J. H. 1980b. Tenderizing in M. Longissimus dorsi of beef, veal, rabbit, lamb and pork. Meat Sci. *5*, 139-147.

EFFENBERGER, G. and SCHOTTE, K. 1972. Verpackung von Fleisch und Fleischwaren. Verlag der Rheinhessischen Druckwerkstätte Alzey.

EINO, M. F. and STANLEY, D. W. 1973. Catheptic activity, texture properties and surface ultrastructure of postmortem beef muscle. J. Food Sci. *38*, 45-50.

GEORGE, A. R., BENDALL, J. R. and JONES, R. C. D. 1980. The tenderizing effect of electrical stimulation of beef carcasses. Meat Sci. *4*, 51-68.

HIMMELBLAU, D. H. 1970. *Process Analysis by Statistical Methods.* pp. 117-118, J. Wiley & Sons, New York.

KATSARAS, K., TETZLAFF, G. and BUDRAS, K. D. 1984. Morphological changes in beef musculature during aging. Fleischwirtschaft *64* (10), 1222-1225.

LARMOND, E., PETROSOVITS, A. and HILL, P. 1969. Application of multiple paired comparisons in studying the effects of aging and finish on beef tenderness. Can. J. Animal Sci. *49*, 51-55.

LOCKER, R. H. and WILD, D. J. C. 1981. Yield point in raw beef muscle, the effects of aging, rigor temperature and stretch. Meat Sci., *7*, 93-107.

LOCKER, R. H. and WILD, D. J. C. 1982. A machine for measuring yield point in raw meat. J. Texture Studies *13*, 71-82.

MacBRIDE, M. A. and PARRISH Jr., F. C. 1977. The 30 K dalton component of tender bovine longissimus muscle. J. Food Sci. *42*, 1627-1629.

MacDOUGALL, D. B. 1971. The effect of time and storage temperature on consumer quality. 17th European Meeting of Meat Research Workers. Bristol p. 8.1.

OLSON, D. G. and PARRISH Jr., F. C. 1977. Relationship of myofibrillar fragmentation index to measures of beefsteak tenderness. J. Food Sci. *42*, 506-509.

OLSON, D. G., PARRISH Jr., F. C., DAYTON, W. R. and GOLL, D. E. 1977. Effect of postmortem storage and calcium activated factor on the myofibrillar proteins of bovine skeletal muscle. J. Food Sci. *42*, 117-120.

OLSON, D. G., PARRISH Jr., F. C. and STROMER, M. A. 1976. Myofibrillar fragmentation index and shear resistance of three bovine muscle during postmortem storage. J. Food Sci. *41*, 1036-1041.

OUALI, A. 1984. Sensitivity of ionic strength of Mg-Ca enhanced ATPase activity as an index of myofibrillar aging in beef. Meat Sci. *11*, 79-88.

OUALI, A., OBLED, A., COTTIN, P., MERDACI, N., DUCASTAING, A. and VALIN, C. 1983. Comparative effects of postmortem storage and low calcium requiring neutral proteinase on bovine and rabbit myofibrillar proteins. J. Sci. Fd. Agric. *34*, 466-470.

PARRISH, Jr., F. C. and CHENG, C. S. 1979. Heat-induced changes in myofibrillar proteins of bovine longissimus muscle. J. Food Sci. *44*, 22-24.

PARRISH, Jr., F. C., SELVIG, C. J., CULLER, R. D. AND ZEEZE, M. G. 1981. Calcium concentration and the 30 K dalton component of tough and tender bovine longissimus muscle. J. Food Sci. *46*, 308-311.

PENFIELD, M. P. and MEYER, B. H. 1975. Changes in tenderness and collagen of beef semitendinosus muscle heated at two rates. J. Food Sci. *40*, 154-156.

PENNY, I. F. 1980. The Enzimology of conditioning. In *Developments in Meat Science*, p. 11, Appl. Sci. Publ. Ltd., London.

PENNY, I. F. and DRANSFIELD, E. 1979. Relationship between toughness and Troponin T in conditioned beef. Meat Sci. *3*, 135-141.

PENNY, I. F. and FERGUSON-PRYCE, R. 1979. Measurement of autolysis on beef muscle homogenates. Meat Sci., *3*, 121-134.

STANLEY, D. W. 1976. The texture meat and its measurement. In *Rheology and Texture in Food Quality*, (J. M. de Man, P. W. Voisey, V. F. Rasper and D. W. Stanley, eds.) p. 413-417.

STANLEY, D. W., PEARSON, G. P. and COXWORTH, V. E. 1971. Evaluation of certain physical properties using the Universal Testing Machine. J. Food Sci. *36*, 256-260.

SZCZESNIAK, A. S. and TORGESON, K. W. 1965. Methods of meat texture measurement viewed from the background of factors affecting tenderness. In *Advances in Food Research*, Vol. 14, (C. V. Chschester, E. M. Mrak, and G. F. Stewart,)(eds.) p. 33 & 68. Academic Press, New York.

VALIN, C., TOURALLE, C., OUALI, A. and LACOURT, A. 1981. Influence de la stimulation electrique sur la maturation et les qualites organoleptiques de la viande de boeuf. Sci. Aliments. *1*, 467-476.

WEBER, A. 1984. Aging of bovine muscle: Desmin degradation observed via Enzyme Linked Immuno Sorbent Assay (ELISA). Proc. 30th. Meet. Meat Res. Workers. No 30, 3: 20, pp. 135-136.

YATES, L. D., DUTSON, T. R., CALDWELL, J. S. and CARPENTER, Z. L. 1983. Effect of temperature and pH on the postmortem degradation of myofibrillar proteins. Meat Sci. *9*, 157-179.

ZAMORA, M. C. and ZARITZKY, N. E. 1985. Modeling of microbial growth in refrigerated packaged beef. J. Food Sci. *50*, 1003-1006, 1013.

SURFACE ACTIVE PROPERTIES AND CLEANING EFFICACY OF MATERIALS DERIVED FROM LACTITOL

C. G. HILL, JR.[1] R. HEDINSDOTTIR[2] and C. H. AMUNDSON[3]

University of Wisconsin-Madison
Madison, Wisconsin 53706

Revised for Publication September 15, 1986

ABSTRACT

Six lactitol esters were produced by direct esterification of lactitol and propoxylated lactitol with long chain fatty acids: lactitol palmitate, lactitol stearate, an ester of lactitol and a 50:50 mixture of the aforementioned fatty acids (lactitol palmitate/stearate) as well as the three esters formed by reaction of propoxylated lactitol with the aforementioned fatty acids (propoxylated lactitol palmitate, propoxylated lactitol stearate and propoxylated lactitol palmitrate/stearate). These products were tested for surface activity as reflected in such properties as surface tension, lime soap dispersion, detergency, and emulsification power. All esters exhibited substantial surface activity. They proved to be effective lime soap dispersing agents and detergents for cotton fabrics. The propoxylated lactitol esters showed lime soap dispersion and detergency properties superior to those of the conventional lactitol esters. The stability of emulsions formed with all these products exceeded that of the sorbitan monolaurate, a commercially available emulsifier and stabilizer.

INTRODUCTION

Of the 2.8 billion pounds of whey solids produced annually in the United States as a byproduct of cheese manufacture, only approximately 50% is utilized. The remainder is disposed of as waste (Morr 1984). Because these solids contain

[1]Department of Chemical Engineering
[2]Department of Food Science
[3]Department of Food Science and Agricultural Engineering

Journal of Food Processing and Preservation 11 (1987) 111-134. All Rights Reserved.

primarily carbohydrates and proteins, they are characterized by a high biological oxygen demand (BOD) and thus constitute a significant waste disposal or treatment problem. At the same time, however, the nutrient content of the whey solids presents a significant opportunity for enhanced utilization. Hence a number of research efforts have, in recent years, focused on the problems of recovery of whey proteins for use in food industry applications (Kosikowski 1979); McDonough 1977; Morr 1984; Slack 1984; Slack *et al.* 1986abc) and on the utilization of the lactose which constitutes the major solid component of whey.

Many of these efforts have focused on the use of ultrafiltration as a vehicle for fractionating whey. Ultrafiltration yields two process streams (1) the retentate, a concentrated protein solution containing some lactose and salts, and (2) the permeate which contains no proteins but retains its original concentrations of lactose and ash. The permeate from ultrafiltration has a high lactose content and its BOD is not far from that of the original whey. Thus it remains a major utilization or disposal problem (Caughlin *et al.* 1978). Even though recent years have seen improved utilization of whey proteins and considerable nutritional values being saved from disposal, the major component of whey solids (lactose) is still disposed of as waste. This situation has provided the incentive for a number of research efforts aimed at improving the utilization of lactose. While substantial markets for lactose utilization exist within the food and pharmaceutical industries, the intrinsic properties of lactose (low solubility, low sweetening power) and the fact that substantial portions of the human population suffer from a lactose-intolerance condition limit its market potential in these areas.

It would be highly advantageous if its market potential could be expanded via chemical modification. Previous efforts utilizing this approach include both hydrolysis of lactose (to glucose and galactose) (Caughlin *et al.* 1978; Scott 1985; Scott *et al.* 1984, 1985; Hill *et al.* 1986; Peterson *et al.* 1986) and its use as a monomer in the production of polyols (Viswanathan *et al.* 1984).

The major purpose of this research was to prepare surface active materials via chemical alterations of lactose. Lactitol derived from lactose was reacted with long chain fatty acids to form lactitol esters. The resulting products were tested for surface active properties, viz, surface tension, emulsifying capability, lime soap dispersion power, and detergency.

Scholnick *et al.* (1974) formed lactose esters by a reaction between lactose and highly reactive fatty acid chlorides dissolved in N-methyl-2-pyrrolidinone. Crude yields of 88-95% were obtained. The products were mainly monoesters, which are the desired products. These lactose esters possessed good detergency and emulsification properties. Scholnick and his coworkers later reported the production of lactitol esters (1975) and oxyalkylated lactitol esters (1977). These products were formed in mutual solvents. The lactitol derivatives showed surface active properties comparable to those of their corresponding lactose derivatives and were readily biodegradable. Van Velthuijsen (1979) has reported

a solvent free method for preparing fatty esters of lactitol and some detergency and emulsification data for lactitol palmitate.

In the present research we sought to prepare lactose based surfactants without recourse to the use of toxic solvents in the production procedure. It was also highly desirable to avoid the use of expensive raw materials, such as fatty acid halides. Because of the alkali and heat sensitivity of lactose, it was not possible to apply any of the solvent free methods that have previously been used for the production of sucrose esters (Feuge *et al.* 1970; Osipow and Rosenblatt 1967; Parker *et al.* 1977). The method of choice was first to reduce lactose to form lactitol and then to use lactitol as the hydrophilic moiety of a nonionic surface active agent.

MATERIALS AND TECHNIQUES USED IN PREPARATION THEREOF

Six different samples of lactitol esters were propared. Synthesis conditions are listed in Table 1, together with shorthand codes for the several surfactants.

Lactitol and Propoxylated Lactitol

Lactitol was either prepared in our laboratory as anhydrous lactitol using the method of Scholnick *et al.* (1975) or obtained from the Express Dairy U. K., Limited (Middlesex, United Kingdom) as lactitol monohydrate. Propylene oxide was obtained from Union Carbide, Linde Division (Somerset, New Jersey)

Propylene oxide was reacted with lactitol following the method described by Viswanathan *et al.* (1984) for whey permeate propoxylation. In a typical run 0.1 mole of anhydrous lactitol or lactitol monohydrate and 1 mole of propylene oxide were placed in a 300 mL pressure reactor obtained from Parr Instrument Company (Moline, Illinois). 0.5 g of potassium hydroxide was added as a catalyst. The reactor was sealed and the mixture heated to 120°C with stirring. At this temperature, the pressure in the reactor was about 120 psi. As the reaction progressed and the propylene oxide was consumed, the pressure decreased. When the pressure inside the reactor reached atmospheric pressure, the reaction was considered complete. The product was a very viscous, yellowish syrup.

Esters

Palmitic acid and stearic acid were obtained from Aldrich Chemical Co. (Milwaukee, Wisconsin). Lactitol esters were made by direct esterification of these fatty acids with lactitol or propoxylated lactitol. Sodium or potassium soaps of the fatty acids were used as catalysts and emulsifiers. The reaction was carried out under reduced pressure at temperatures of 145-160°C. The procedure employed was that developed by van Velthuijsen (1979) except that, in most cases, lower temperatures were used. The reduced temperatures led to products of lighter

TABLE 1
RAW MATERIALS FOR PREPARATION OF SURFACTANTS

Sample	(Code)	moles lactitol hydrate	moles anhydrous lactitol	moles stearic acid	moles palmitic acid	moles KOH	moles NaOH	reaction Time, h	reaction temperature [°C]
lactitol stearate	(S)	0.1		0.095			0.025	10	145
lactitol palmitate	(P)	0.1			0.095		0.025	9	145
lactitol palmitate/ stearate	(P/S)	0.1		0.048	0.048		0.025	10	145-150
propoxylated lactitol stearate	(PS)		0.1	0.1		0.03		8	155
propoxylated lactitol palmitate	(PP)		0.1		0.1	0.03		8.5	158
propoxylated lactitol palmitate/ stearate	(PP/S)	0.1		.048	0.048	0.09	0.016	10	148

color. In a typical example 0.1 mole of anhydrous lactitol or lactitol monohydrate was dissoved in a small amount of water (10-15 mL) in a three-necked, 500 mL, round bottom flask. To the lactitol solution was added 0.095 mole of fatty acid (0.025 moles to form the sodium soap and 0.07 moles to react with the lactitol and form lactitol fatty acid esters). The lactitol/fatty acid ratio was 1:0.7. The flask was placed in a temperature controlled oil bath and attached to a Dean/Stark trap, a stirrer, and a nitrogen gas source. The contents of the flask were well-stirred at all times. The temperature of the oil bath was raised until the fatty acid was molten. Then the water was slowly distilled off under reduced pressure while the temperature was faised further to about 100 °C. When the mixture was water free, 1 g (0.025 mole) of NaOH dissolved in as little water as possible was added to the flask to form the sodium soap. (If too much water is added to the flask with the NaOH, the soap may form lumps which can be difficult to eliminate.) The water was again distilled off under reduced pressure and the temperature was raised to 145 °C. The reaction mixture was then maintained at 145 °C and a reduced pressure of 20-80 mm Hg under good stirring for 9-10 h. A small stream of nitrogen gas was continuously passed over the reaction mixture. The final product was a brown viscous liquid which hardened into a solid when cooled to room temperature. The solid could then be easily ground to a fine powder.

In the case of propoxylated lactitol, the polyol product of the propoxylation step (0.1 mole) was piaced in a three-necked flask. Then the procedure described for the lactitol ester production was followed, except that less alkali metal was added since some was already present from the propoxylation step. The final product was a brown liquid which upon cooling formed a very viscous, pastelike material.

METHODS FOR CHARACTERIZATION OF SURFACE ACTIVE PROPERTIES AND CLEANING EFFICACY

All characterization experiments were performed on the crude products, without any purification.

Surface Tension

Surface tension curves for aqueous solutions of the surfactant samples were obtained using the DuNouy ring method (Sosis 1975). A Cenco^R-DuNouy^R Interfacial Tensiometer was utilized (Central Scientific Co., Chicago, Illinois).

Emulsification Power

Emulsion stability was determined as follows. A solution of the surfactant sample (0.2% w/v) was prepared in deionized water. The pH of the solution was ad-

justed to 7.0 with 0.1N HCl or NaOH. A 100 mL aliquot of the surfactant solution was placed in a Waring Blendor. Methylene blue indicator (0.5 mL of 0.1% solution) was added to facilitate detection of separation. During mixing, 100 mL of additive free soy oil (Wesson) was added to the blender at a rate of 3 mL/s. The emulsion was mixed for one minute at a low speed. The emulsion was then transferred to a 100 mL graduated cylinder and held at room temperature. The time of separation of the aqueous phase was observed. The emulsion stability was taken as the time required for 10% separation of either phase. All tests were performed in triplicate.

Lime Soap Dispersion

Lime soap dispersion was tested using the method of Borghetty and Bergman (1950). The test is based on the fact that calcium oleate precipitates when a solution of sodium oleate is added to a hard water solution.

Five mL of 0.5% sodium oleate solution is mixed with 10 mL of hard water solution, an appropriate amount of 0.25% surfactant solution, and enough distilled water to make a total volume of 30 mL. The appropriate amount of detergent solution is found by trial and error. The hard water solution employed had a total hardness of 1000 ppm, calculated as $CaCO_3$. Sixty percent of the hardness was attributed to the presence of calcium ions and 40% to magnesium ions. The test measures the amount of surfactant needed to completely disperse the insoluble calcium oleate.

Sodium oleate was obtained from Aldrich Chemical Company (Milwaukee, Wisconsin), calcium chloride from Amend Drug and Chemical Company (Irvington, New Jersey), and magnesium chloride from Columbus Chemical Industries, Inc. (Columbus, Wisconsin).

Detergency

Detergency was determined as the difference in reflectance (ΔR) of standard soiled testcloths before and after they were washed in a solution of the surfactant. The five different testcloths listed in Table 2 were utilized.

The samples were tested at two water hardnesses, 50 ppm and 300 ppm. The hard water solution of 300 ppm hardness (calculated as $CaCO_3$) contained a Ca/Mg ratio of 2:1. The 50 ppm solution had a Ca/Mg ratio of 4:1 (Anonymous).

Two reference surfactants were used for the detergency measurements.

(1) Triton X-100, a registered trademark of Rohm and Haas Co., obtained from Aldrich Chemical Co. [Milwaukee, Wisconsin].
(2) Sorbitan monolaurate obtained from BASF Wyandotte Corp. [Parsippany, New Jersey].

TABLE 2
TEST FABRICS FOR DETERGENCY STUDIES

Number	Description	Manufacturer
1	Soiled cotton, style 405	Testfabrics, Inc. (Middlesex, New Jersey)
2	Soiled Dacron 54W/cotton 65/35 with durable press finish, style 7406 WRL	Testfabrics, Inc. (Middlesex, New Jersey)
3	WFK-20C polyester/cotton 65/35 with standard soil	Waescherei Forschungs Institute of Krefeld (West Germany)
4	WFK-30C polyester with standard soil	Waescherei Forschungs Institute of Krefeld (West Germany)
5	EMPA (117) Dacron 54W/cotton 65/35 Soil Test Cloth: Blood-Milk-Ink	Swiss Federal Testing Station

Testfabrics, Inc. represents the WFK-products and the EMPA-products in the United States.

The washing tests were carried out in a Type LDH-EF Launder-Ometer manufactured by Atlas Electric Devices Co. [Chicago, Illinois]. The Launder-Ometer Single Wash Technic, described by Harris (1954a) was utilized.

Into each ½ liter Launder-Ometer jar were placed 200 mL of test solution and ten 0.25 in. stainless steel balls. The solutions were preheated to the desired temperature. Then two 3 × 3 in. pieces of soiled testcloth were put in each jar. The jars were sealed, transferred to the Launder-Ometer drum and rotated for 30 min. at 40 rpm. In most cases each test solution was tested in triplicate. Thus the results represent an average of six cloth swatches for each sample. After the washing cycle the test swatches were rinsed in distilled water at 100°F. The swatches were then dried and kept overnight at room conditions.

Reflectance measurements were made before and after washing with a Model D-1 Color-Eye from Instrument Development Laboratories [Attleboro, Massachusetts]. The brightness of the testcloth compared to a white standard was measured using the green filter of the instrument. Results are expressed as increase in reflectance after washing (ΔR).

RESULTS AND DISCUSSION

The lactitol esters that were produced in this study were either light colored powders (conventional lactitol esters) or brown pastes (the propoxylated lactitol esters). All samples formed cloudy solutions in water. Some heat and stirring were needed to dissolve the powders, but the propoxylated derivatives dissolved more readily.

All samples were tested for the following surface active properties: surface tension, lime soap dispersion power, emulsifying capability, and detergency. The results of each aspect of this study are treated in turn.

Surface Tension Results

Surface tension curves for the surfactants produced in this study are shown in Fig. 1. All surfactants showed surface tension curves similar to those expected for surface active agents (Becher 1965).

Emulsification Stability Tests

Emulsion stability is expressed as the time required for a given degree of separation of the aqueous phase of the emulsion. For all samples in the present study, the extent of separation of the emulsion was found to approximate a linear function of time. Typical data are shown in Fig. 2. In all cases except that of the propoxylated lactitol stearate, the emulsion stability was taken as the time necessary to bring about the increase in separation from 5% to 15%. Separation was so rapid in the case of the noted exception that it was necessary to employ the region from 10% to 20% as a measure of (in)stability in that case. Since the straight lines through the data did not all pass through the origin, this approach circumvents the physical difficulties in trying to assess the degree of separation at time zero.

The use of the straight line gives an average value, rather than the exact time for the separation of the first 10% of the aqueous phase (see Fig. 2). Sorbitan monolaurate, a commercially available emulsifier and stabilizer was used as a reference material. The results are summarized in Table 3. These results indicate that there is considerable variation in emulsion stability from one ester to another. The propoxylated products do not show a superior performance here. The stabilities of the several emulsions seem to be affected primarily by the fatty acid constituent of the ester. The palmitates give emulsions with vastly better stability than the stearates and the palmitate/stearates give emulsions of intermediate stability. All lactitol derivatives investigated showed much better ability to stabilize emulsions than the reference material. These results are in agreement with those of Scholnick *et al.* (1974, 1975) who reported that the use of lactose- and lactitol palmitates yielded more stable emulsions than the corresponding stearates. Van Velthuijsen (1979) has reported emulsification data for lactitol palmitate and sorbitan monolaurate. His results indicate that lactitol palmitate is a more powerful emulsifying agent than sorbitan monolaurate.

Lime Soap Dispersion Results

The lime soap dispersion test measures the ability of surfactants to disperse insoluble metallic soaps (Borghetty and Bergman 1950). The amount of dispers-

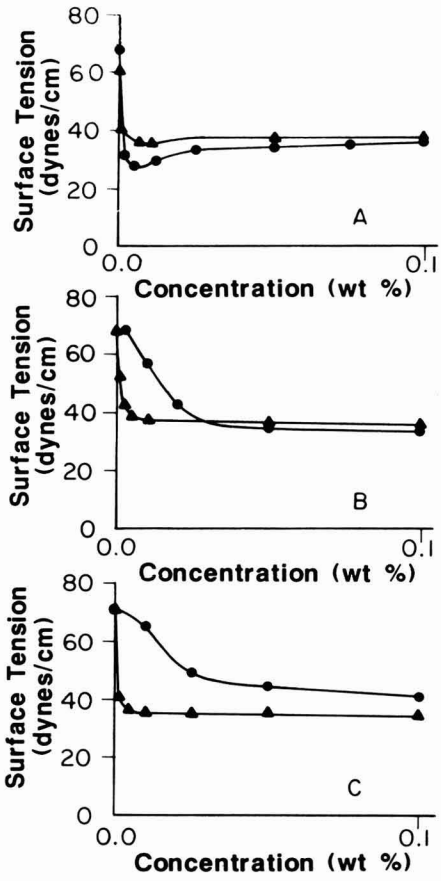

FIG. 1. SURFACE TENSION VERSUS CONCENTRATION CURVES

a) lactitol palmitates.

● Lactitol palmitate
▲ Propoxylated lactitol palmitate

b) Lactitol stearates.

● Lactitol stearate
▲ Propoxylated lactitol stearate

c) Lactitol palmitate/stearates

● Lactitol palmitate/stearate
▲ Propoxylated lactitol palmit

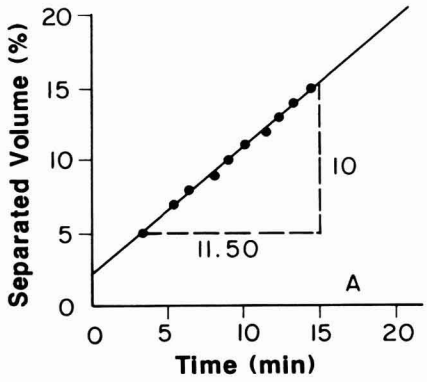

FIG. 2A. STABILITY OF OIL/WATER EMULSIONS FORMED USING LACTITOL PALMITATE/STEARATE

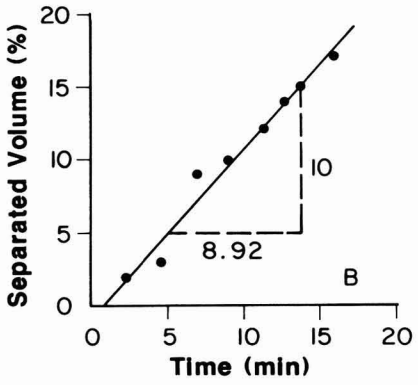

FIG. 2B. STABILITY OF OIL/WATER EMULSIONS FORMED USING PROPOXYLATED LACTITOL PALMITRATE/STEARATE

ing agent is expressed as a percentage of the weight of sodium oleate employed in the test.

$$\% \text{ lime soap disperison requirement} = \frac{\text{grams of dispersing agent to disperse lime soap}}{\text{weight of sodium oleate}} \times 100$$

Effective agents for this purpose have dispersion numbers in the range of 10 to 40, while materials which disperse the solution moderately have dispersion numbers between 40 and 80 (Harris 1954b). The lime soap dispersion powers of the various lactitol derivatives are listed in Table 4.

TABLE 3
EMULSION STABILITY

Sample	Time [min] required for 10% separation of aqueous phase
Lactitol palmitate	10.5
Propoxylated lactitol palmitate	23.3
Lactitol stearate	6.9
Propoxylated lactitol stearate	2.8
Lactitol palmitate/stearate	11.5
Propoxylated lactitol palmitate/stearate	8.9
Sorbitan monolaurate	1.1

TABLE 4
LIME SOAP DISPERSING POWDER (LSDP) CALCULATED AS PERCENT OF SODIUM OLEATE.

Sample	% LSDP
Lactitol palmitate	32
Lactitol stearate	42
Lactitol palmitate/stearate	32
Propoxylated lactitol palmitate	11
Propoxylated lactitol stearate	16
Propoxylated lactitol palmitate/stearate	10
Triton X-100	4

Both the lactitol esters and the propoxylated lactitol esters are effective lime soap dispersion agents. However, the propoxylated derivatives show much better lime soap dispersing power than the conventional lactitol esters. All samples, however, are inferior to the Triton X-100 used as a reference sample. These results are consistent with those of Scholnick *et al.* (1974, 1975, 1977) although the results for the conventional lactitol esters are marginally inferior to their results for lactose esters and lactitol esters. However, it has to be kept in mind that the results published by Scholnick *et al.* were obtained with purified products. On the other hand, the present results were obtained with the crude reaction products in the absence of purification.

Detergency Results

The detergency studies involved three phases: (1) Studies of the effect of surfactant concentration at 130°F. (2) Studies of the effect of chemical composition at constant temperature (130°F) and constant concentration [0.1% (w/v)]. (3) Studies of the effect of washing temperature (100°F versus 130°F).

In phases 1 and 2 the effects of using washwater of different hardness levels (50 ppm and 300 ppm, calculated as $CaCO_3$) were also ascertained.

In each experiment reference surfactants (Triton X-100 and sorbitan monolaurate) were utilized for purposes of comparison. Results are expressed as the increase in reflectance of the test fabric after washing.

The t-test for comparing means of two samples was employed to test the significance of the differences between results under two different experimental conditions (Chatfield 1970). A one-tailed t-test was used to determine whether the mean x of a sample of size n and standard deviation p was significantly greater than the mean y of a sample of size m and standard deviation q.

$$t = (x - y)/[s\ \{(1/n) + (1/m)\}^{1/2}]$$

$$s = [\{(n - 1)p^2 + (m - 1)q^2\}/\{n + m - 2\}]^{1/2}$$

The value of t was compared to the critical value for $n + m - 2$ degrees of freedom at the desired level of significance, usually 99%.

The results of each of the three phases of the detergency studies are treated in turn:

Effects of Surfactant Concentration and Water Hardness

To assess the effect of surfactant concentration on detergency, lactitol stearate was tested at five levels (w/v): 0.25%, 0.20%, 0.15%, 0.10%, and 0.05%. The fabric samples tested were numbers 1-3: a cotton, a 65/35 Dacron/cotton blend, and a 65/35 polyester/cotton blend. The results for the cotton samples (#1) (see Fig. 3) indicate that the lactitol sterate is a good detergent for this material.

For soiled cotton (sample #1) in the water with a hardness of 50 ppm, the solutions with concentrations in the range 0.05-0.20% (w/v) performed in a manner such that they could not be statistically distinguished from one another (at the 99% confidence level) or from the 0.1% Triton X-100 solution. The performance of the 0.25% sample was superior to that of all other solutions. Solutions containing Triton X-100 and lactitol stearate (all concentrations) performed significantly better than either water alone or a 0.10% solution of sorbitan monolaurate. In hard water (300 ppm), the 0.25% lactitol stearate solution does not show detergency superior to those of solutions which contain lower concentrations of this compound. At this water hardness, all concentrations of lactitol stearate and

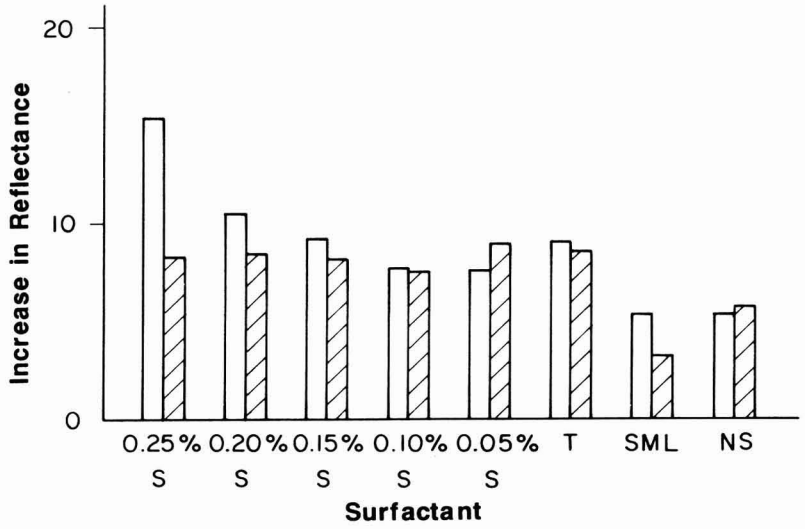

FIG. 3. INCREASE IN REFLECTANCE (ΔR) OF COTTON 405 FABRIC AFTER WASHING AT 130°F IN SOLUTIONS CONTAINING FIVE DIFFERENT CONCENTRATIONS OF LACTITOL STEARATE

S = lactitol stearate
T = 0.1% Triton X-100
SML = 0.1% sorbitan monolaurate
NS = no surfactant

☐ Soft water (50 ppm)

☑ Hard water (300 ppm)

the 0.1% Triton X-100 solution appear to perform equally well. Moreover, these solutions have significantly better detergency than the water alone and than the 0.10% solution of sorbitan monolaurate. Statistically significant differences in performance at the 50 ppm hardness level versus the 300 ppm hardness level were observed only for the 0.25% and 0.20% lactitol stearate solutions. The other solutions performed equally well at the two water hardness levels studied.

For the synthetic fiber/cotton blends containing either Dacron or polyester (samples 2 and 3), the various lactitol stearate solutions exhibited less efficacious detergency characteristics than they did for cotton alone. Significant differences in detergency between the different concentration levels of lactitol stearate were not observed for the Dacron/cotton blend (see Fig. 4). An exception was the performance of the 0.25% lacitol stearate solution versus that of the 0.05% lactitol stearate solution in hard (300 ppm) water. Solutions of lactitol stearate at all concentration levels performed significantly better than either water alone or the

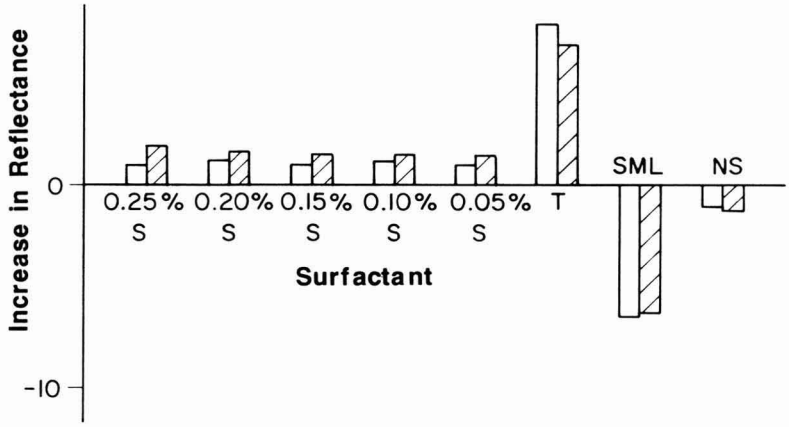

FIG. 4. INCREASE IN REFLECTANCE (ΔR) OF DACRON 54W/COTTON 65/35 FABRIC FROM TESTFABRICS AFTER WASHING AT 130°F IN SOLUTIONS CONTAINING FIVE DIFFERENT CONCENRATIONS OF LACTITOL STEARATE

S = lactitol stearate
T = 0.1% Triton X-100
SML = 0.1% sorbitan monolaurate
NS = no surfactant

☐ Soft water (50 ppm)

☑ Hard water (300 ppm)

0.10% solution of sorbitan monolaurate. However, the 0.1% Triton X-100 solution performed vastly better than all other surfactant solutions. Sorbitan monolaurate and water alone gave negative results with this fabric, i.e. the reflectance of the fabric was lower after washing than before. This phenomenon can be explained in terms of an increase in the particle size of the soil material during the washing cycle. If the soil particles are not removed from the fabric, they may adhere to the fibers in a different manner than prior to washing. In such a case, the fabric may seem darker after washing. Even though the lactitol stearate solutions performed much better than water or sorbitan monolaurate on this fabric, the improvement is so small that lactitol stearate cannot be considered a suitable detergent for use with this fabric. In all cases, comparable results were obtained at both levels of water hardness (50 and 300 ppm).

For the polyester/cotton blend (sample #3), the results obtained were quite comparable to those obtained with the Dacron/cotton blend. Solutions of Triton X-100 gave very good results but solutions of lactitol stearate were relatively ineffective. However, the latter performed significantly better than solutions containing

either no surfactant or sorbitan monolaurate. The performance at all concentration levels of lactitol stearate was similar; effectiveness decreased only slightly with decreasing concentration. No significant differences between performance in soft and hard water were observed.

The results of this series of experiments on the effects of variations in lactitol stearate concentration and water hardness may be summarized as follows: (1) A variation of the concentration of lactitol stearate from 0.05% to 0.20% has only a slight effect on the soil removal. (2) Water hardness has little effect on the detergency of lactitol stearate solutions, except at the highest concentrations (0.25% and 0.20%). (3) Lactitol stearate performs well on cotton fabrics. Its performance is comparable to that of Trion X-100 and is markedly better than that of solutions of sorbitan monolaurate or water alone. On the other hand, lactitol stearate shows limited ability to clean the synthetic fiber/cotton blends tested. Nonetheless, its performance is significantly better than that of either solutions of sorbitan monolaurate or solutions containing no surfactant.

The first of these results is consistent with the results of the surface tension experiments which indicate that surfactant concentrations of 0.05% (w/v) are all that are required to reach the asymptotic limit in a surface tension versus concentration curve. Under such circumstances substantial numbers of surfactant micelles will exist in solution. This hypothesis and the results of the other surface tension studies formed the basis for our choice of 0.1% solutions of surfactants as the level at which we would study the effects of variation in chemical structure on the detergency characteristics of the several lactitol esters.

Effects of Chemical Composition and Water Hardness

In this phase of the detergency study, all products were tested at the same concentration, 0.1% (w/v). Triton X-100 and sorbitan monolaurate were used as reference materials, also at concentrations of 0.1%. Studies were carried out using both hard (300 ppm) and soft (50 ppm) water. The test fabrics included cotton, polyester, and a Dacron/cotton blend (sample numbers 1, 4, and 5).

The results of the studies employing the cotton test samples are depicted in Fig. 5. All of the surfactants performed better in soft water than in hard water. Statistically significant differences (99% confidence level) were observed for all products except lactitol stearate. In 50 ppm water 0.1% solutions of all products performed significantly better than either a solution of sorbitan monolaurate or water alone. The differences were smaller when 300 ppm water was employed.

It is interesting to note that in general the propoxylated lactitol products performed better than the conventional lactitol esters. At a water hardness of 50 ppm the performance levels of the propoxylated products are markedly better than those of the conventional lactitol esters. Statistically significant differences (at the 99% confidence level) are observed when propoxylated palmitate and stearate are com-

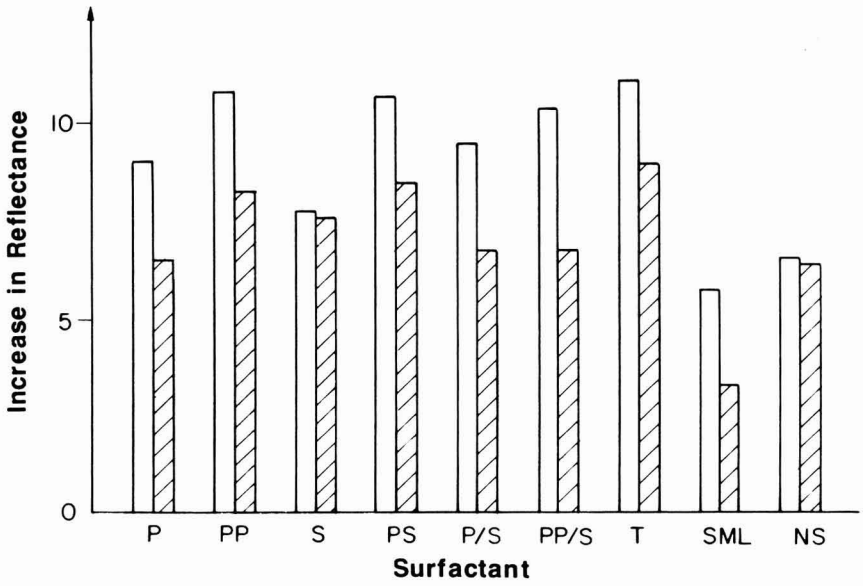

FIG. 5. INCREASE IN REFLECTANCE (ΔR) OF COTTON 405 FABRIC AFTER WASHING AT 130°F IN 0.1% SOLUTIONS (W/V) OF LACTITOL ESTERS

P = 0.1% lactitol palmitate
PP = 0.1% propoxylated lactitol palmitate
S = 0.1% lactitol stearate
PS = 0.1% propoxylated lactitol stearate
P/S = 0.1% lactitol palmitrate/stearate
PP/S = 0.1% propoxylated lactitol plamitrate/stearate
T = 0.1% Triton X-100
SML = 0.1% Sorbitan monolaurate
NS = no surfactant

☐ Soft water (50 ppm)

☑ Hard water (300 ppm)

pared to their conventional counterparts. In 50 ppm water the performance of these products is quite comparable to that of Triton X-100 and is much better than that of either a solution of sorbitan monolaurate or water alone.

In soft water conventional lactitol esters are slightly inferior to the Triton X-100, but all are significantly better than no surfactant. The difference is statistically significant at the 99% level for the palmitate and the palmitate/stearate, but only at the 95% level for the stearate.

The propoxylated derivatives seem to be somewhat more affected by hard water than their conventional analogs. Hence differences in performance between the

propoxylated and conventional lactitol esters become less obvious in 300 ppm water.

The results of the experiments using WFK-30C polyester (sample #4) as the test fabric are presented in Fig. 6. It is readily apparent that both the conventional and the propoxylated lactitol esters possess only a small fraction of the detergency of Triton X-100 for this fabric. However, all solutions of these esters perform significantly better than water alone. For this fabric the differences in performance between soft and hard water solutions are minimal.

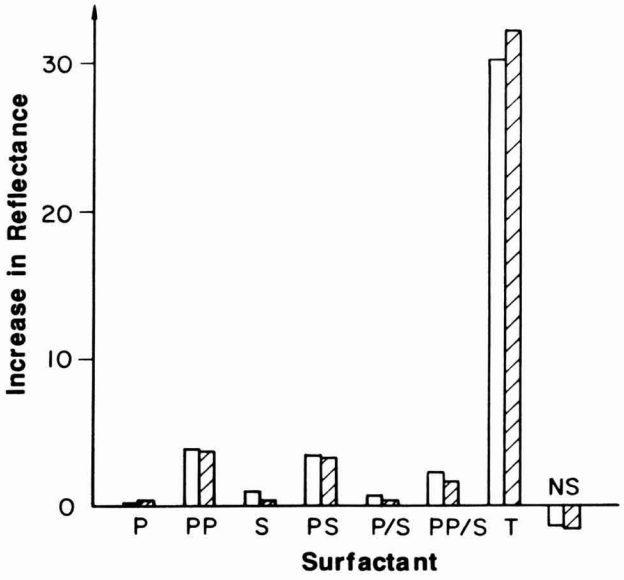

FIG. 6. INCREASE IN REFLECTANCE (ΔR) OF WFK-30C POLYESTER FABRIC AFTER WASHING AT 130°F IN 0.1% SOLUTIONS (W/V) OF LACTITOL ESTERS

P = 0.1% lactitol palmitate
PP = 0.1% propoxylated lactitol palmitate
S = 0.1% lactitol stearate
PS = 0.1% propoxylated lactitol stearate
P/S = 0.1% lactitol palmitrate/stearate
PP/S = 0.1% propoxylated lactitol palmitrate/stearate
T = 0.1% Triton X-100
NS = no surfactant

☐ Soft water (50 ppm)

▨ Hard water (300 ppm)

For this fabric as well as for the cottons the propoxylated lactitol esters give markedly better results than the conventional lactitol esters. The differences are statistically significant (at the 99% confidence level) in both soft and hard water. However, the performance of these products is not good enough to qualify any of them as a good detergent for polyester fabrics.

The results for the EMPA (117) Dacron 54W/cotton fabric (sample #5) are presented in Fig. 7. These results indicate that all samples and standards perform similarly on this fabric. This is in marked contrast with the results obtained with the various lactitol stearate solutions for the Dacron 54W/cotton sample obtained from Testfabrics (see Fig. 4). On that test fabric, Triton X-100 performed fairly well and the several lactitol stearate solutions gave results which were significantly better than those obtained with no surfactant or with sorbiton monolaurate.

The difference between the two test fabrics lies in the amount and content of the soil applied. The soiled EMPA fabric is much darker than the Dacron/cotton

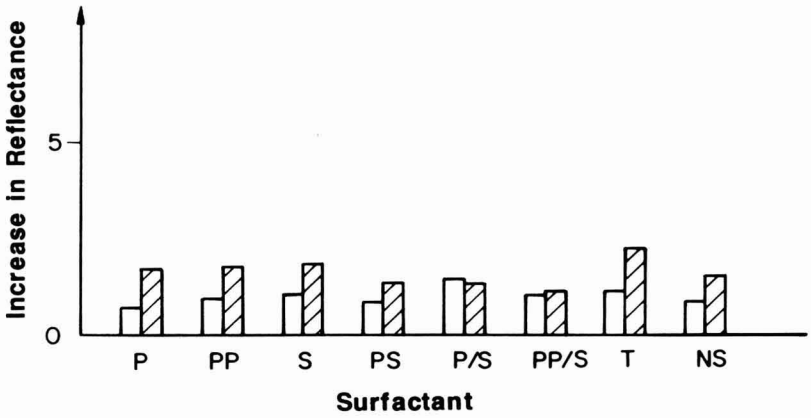

FIG. 7. INCREASE IS REFLECTANCE (ΔR) OF EMPA (117) DACRON 54W/COTTON FABRIC AFTER WASHING AT 130°F IN 0.1% SOLUTIONS (W/V) OF LACTITOL ESTERS

P = 0.1% lactitol palmitate
PP = 0.1% propoxylated lactitol palmitate
S = 0.1% lactitol stearate
PS = 0.1% propoxylated lactitol stearate
P/S = 0.1% lactitol palmitrate/stearate
PP/S = 0.1% propoxylated lactitol palmitrate/stearate
T = 0.1% Triton X-100
NS = no surfactant

☐ Soft water (50 ppm)

☑ Hard water (300 ppm)

fabric manufactured by Testfabrics Inc. (sample #2). When the fabric is very dark, the changes in the reflectance may not correspond very well to the amount of soil removed. In such cases one can remove a considerable amount of soil without seeing much change in the brightness of the fabric. Therefore, the EMPA fabric is not a very good representative test sample for home laundry conditions. The reason for its use in the latter portion of this detergency study was the unavailability of the Dacron 54W/cotton fabric that was used earlier. It is, therefore, difficult to predict what the performance of our products would be on Dacron/cotton blends if they were not as heavily soiled. The results shown in Fig. 7 indicate, however, that neither the lactitol esters nor the reference materials would be effective detergents for heavily soiled Dacron/cotton fabrics.

The results of this phase of the detergency study indicate that for both cotton and polyester fabrics, the propoxylated lactitol esters exhibit better detergency properties than do the conventional lactitol esters. The propoxylated derivatives are more affected by water hardness than are the conventional esters. All the lactitol esters studied exhibited substantial detergency for cotton fabrics. On the other hand, the esters exhibited limited ability to clean either polyester or Dacron fabrics. Consequently, most applications of these detergents would require that they be used in concert with another active component which would be effective for synthetic fibers. This conclusion is especially applicable to the conventional lactitol esters. All the products are markedly better than either water alone or solutions of sorbitan monolaurate.

These conclusions are consistent with the results of Scholnick *et al.* (1974, 1975, 1977) relative to various lactose and lactitol esters.

Effect of Temperature

Three test fabrics were washed both at 100°F and 130°F in water alone and in 0.1% solutions of both lactitol esters and Triton X-100. Water hardness was 50 ppm.

Results for cotton 405 (sample 1) are shown in Fig. 8. All products and standards appear to perform somewhat better at the higher temperature on this fabric. In most cases the difference between the two temperatures is statistically significant at the 99% confidence level. The exceptions are lactitol palmitate which gives a significant difference at the 95% level and lactitol stearate which does not show a significant difference.

The performance of solutions of the propoxylated lactitol esters is slightly more influenced by temperature than is that of solutions of the conventional esters. The significant difference that was observed at 130F for the performance of the propoxylated esters relative to the conventional esters on cotton does not appear at 100°F. At 100°Fall the solutions of the lactitol esters exhibited a performance

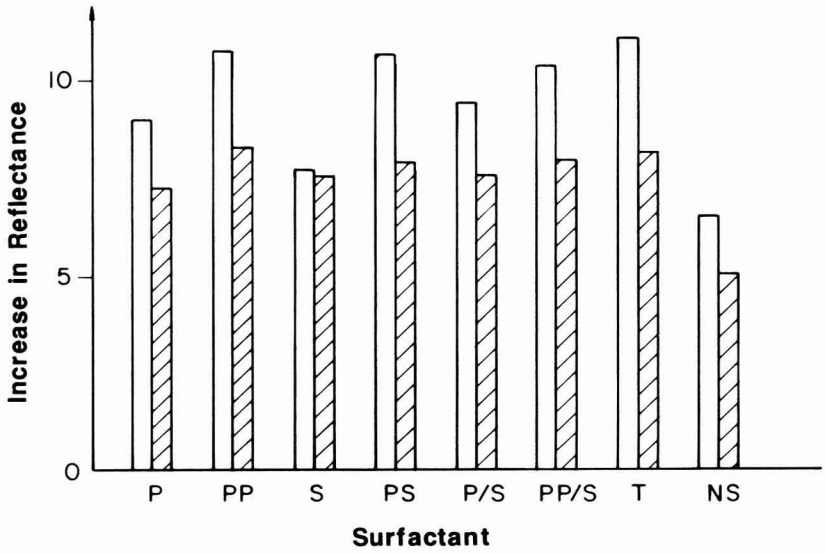

FIG. 8. INCREASE IN REFLECTANCE (ΔR) OF COTTON 405 FABRIC AFTER WASHING IN 0.1% SOLUTIONS (W/V) OF LACTITOL ESTERS IN SOFT WATER (50 PPM) AT TWO DIFFERENT TEMPERATURES

P = 0.1% lactitol palmitate
PP = 0.1% propoxylated lactitol palmitate
S = 0.1% lactitol stearate
PS =0.1% propoxylated lactitol stearate
P/S = 0.1% lactitol palmitrate/stearate
PP/S = 0.1% propoxylated lactitol palmitrate/stearate
T = 0.1% Triton X-100
NS = no surfactant

☐ 130°F

☑ 100°F

which was comparable to that of Triton X-100 and which was markedly better than that of solution containing no surfactant.

The effect of temperature on the detergency properties of the lactitol esters was also studied for the WFK-30C polyester fabric (sample 4) as Fig. 9 indicates. The performance of the propoxylated esters is significantly better at 130°F than at 100°F. The propoxylated lactitol esters also perform significantly better at 100°F than the corresponding conventional lactitol esters. The performance of Triton X-100 on this fabric is vastly better at both temperatures than is that of any of the lactitol esters.

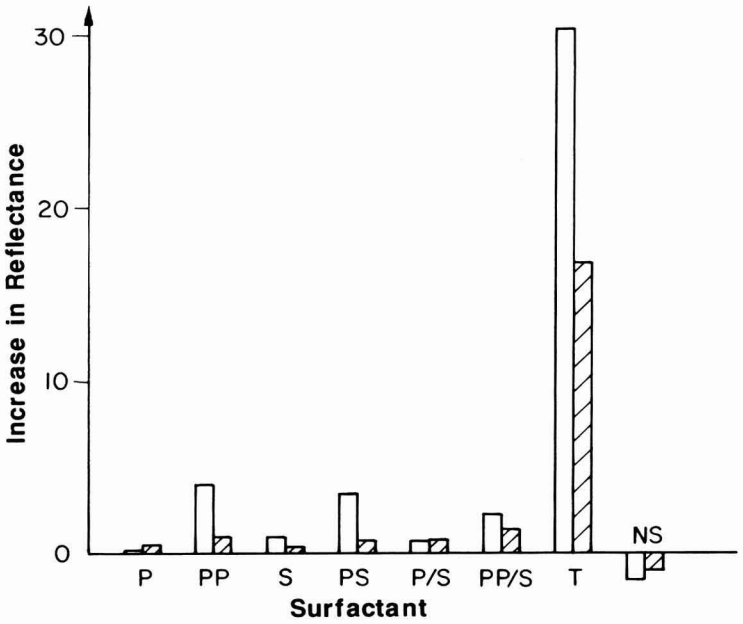

FIG. 9. INCREASE IN REFLECTANCE ($_DR$) OF WFK-30C POLYESTER FABRIC AFTER WASHING IN 0.1% SOLUTIONS (W/V) OF LACTITOL ESTERS IN SOFT WATER (50 PPM) AT TWO DIFFERENT TEMPERATURES

P = 0.1% lactitol palmitate
PP = 0.1% propoxylated lactitol palmitate
S = 0.1% lactitol stearate
PS = 0.1% propoxylated lactitol stearate
P/S = 0.1% lactitol palmitrate/stearate
PP/S = 0.1% propoxylated lactitol palmitrate/stearate
T = 0.1% Triton X-100
NS = no surfactant

☐ 130°F

☑ 100°F

Results for the EMPA (117) Dacron 54W/cotton fabric are depicted in Fig. 10. These results are different from all others in this phase of the detergency study in the sense that higher temperature seems to have an adverse effect on soil removal. However, all values are very low and it is difficult to draw any meaningful conclusions concerning the relative detergency properties of the various surfactant solutions for this fabric.

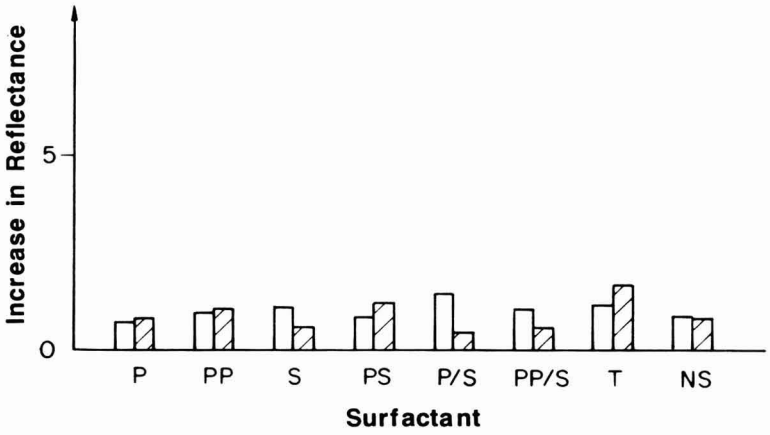

FIG. 10. INCREASE IN REFLECTANCE (ΔR) OF EMPA (117) DACRON 54W/COTTON FABRIC AFTER WASHING IN 0.1% SOLUTIONS (W/V) OF LACTITOL ESTERS IN SOFT WATER (50 PPM) AT TWO DIFFERENT TEMPERATURES

P = 0.1% lactitol palmitate
PP = 0.1% propoxylated lactitol palmitate
S = 0.1% lactitol stearate
PS = 0.1% propoxylated lactitol stearate
P/S = 0.1% lactitol palmitrate/stearate
PP/S = 0.1% propoxylated lactitol palmitrate/stearate
T = 0.1% Triton X-100
NS = no surfactant

☐ 130°F

☑ 100°F

CONCLUSIONS

The following surface active properties of conventional and propoxylated lactitol esters (palmitates, stearates, and a 50/50 mixture thereof) were determined: surface tension, lime soap dispersion, detergency and emulsification power. All esters exhibited substantial surface activity. They were effective lime soap dispersing agents and detergents for cotton fabrics. The propoxylated lactitol esters showed lime soap dispersion and detergency properties superior to those of the conventional lactitol esters. The stability of emulsions formed with all these products exceeded that of the sorbitan monolaurate, a commercially available emulsifier and stabilizer.

ACKNOWLEDGMENTS

This work was supported in part by the College of Agricultural and Life Sciences, University of Wisconsin-Madison, the College of Engineering, University of Wisconsin-Madison, the Whey Products Institute and the Walter V. Price Cheese Research Institute. The authors also wish to thank Express Dairy U. K. Ltd. for providing lactitol monohydrate. Helpful discussions concerning the detergency study were held between Professor Manfred Wertz (School of Family Resources and Consumer Sciences) and Ragnheidur Hedinsdottir.

REFERENCES

ANONYMOUS, ASTM D4265-83. Standard guide for evaluating stain removal performance in home laundering.

BECHER, P. 1965. *Emulsions: Theory and Practice*. Second edition, p. 24-25. Reinhold Publishing Corp., New York.

BORGHETTY, H. C. and BERGMAN, C. A. 1950. Synthetic detergents in the soap industry. J. Am. Oil Chem. Soc. *27*, 88-90.

COUGHLIN, R. W., CHARLES, M. and JULKOWSKI, K. 1978. Experimental results from a pilot plant for converting acid whey to potentially useful food products. AIChE Symposium Series Vol. 74, No. 172, 40-46.

CHATFIELD, C. 1970. *Statistics for Technology*, p. 144-146 and p. 336. Penguin Books.

FEUGE, R. O., ZERINGUE, H. J., JR., WEISS, T. J. and BROWN, M. 1970. Preparation of sucrose esters by interesterification. J. Am. Oil Chem. Soc. *47*, 56-60.

HARRIS, J. C. 1954a. *Detergency Evaluation and Testing*, p. 97. Wiley-Interscience, New York.

HARRIS, J. C. 1954b. *Detergency Evaluation and Testing*, p. 27-36. Wiley-Interscience, New York.

HILL, C. G., JR., AMUNDSON, C. H., SCOTT, T. C. and PETERSON, R. S. 1986. Evaluation of immobilized lactose for processing ultrafiltered whey permeate. Paper presented at the 1986 Cheese Research and Technology Conference.

KOSIKOWSKI, F. V. 1979. Our industry today: whey utilization and whey products. J. Dairy Sci. *62*, 1149-60.

McDONOUGH, F. E. 1977. Whey solids utilization and salvage systems. Cult. Dairy Prod. J. *12*(1), 8-11.

MORR, C. V. 1984. Production and use of milk proteins in food. Food Technol. *38*(7), 39.

NICKERSON, T. A. 1978. Why use lactose and its derivatives in food? Food Technol. *32*(1), 40-2, 44, 46.

OSIPOW, L. I. and ROSENBLATT, W. 1967. Micro-emulsion process for the preparation of sucrose esters. J. Am. Oil Chem. Soc. *44*, 307.

PARKER, K. J., JAMES, K. and HURFORD, J. 1977. Sucrose ester surfactants — a solventless process and the products thereof. In *Sucrochemistry* (J. L. Hickson, ed.). ACS Symp. Series *41*, 97-114. Am. Chem Soc., Washington, D. C.

PETERSON, R. S., HILL, C. G., JR. and AMUNDSON, C. H. 1986. Lactose hydrolysis by immobilized beta-galactosidase in a capillary bed reactor. Paper presented at the June 1986 meeting of the Institute of Food Technologists.

SCHOLNICK, F., BEN-ET, G., SUCHARSKI, M. K., MAURER, E. W. and LINFIELD, W. M. 1975. Lactose-derived surfactants II: fatty esters of lactitol. J. Am. Oil Chem. Soc. *52*, 256-8.

SCHOLNICK, F. and LINFIELD, W. M. 1977. Lactose-derived surfactants III: fatty esters of oxyalkylated lactitol. J. Am. Oil Chem. Soc. *54*, 430-2.

SCHOLNICK, F., SUCHARSKI, M. K. and LINFIELD, W. M. 1974. Lactose-derived surfactants I: fatty esters of lactose. J. Am. Oil Chem. Soc. *51*, 8-11.

SCOTT, T. C. 1985. Hydrolysis of lactose in permeate from the ultrafiltration of cottage cheese whey using immobilized beta-galactosidase. Ph.D. thesis. University of Wisconsin-Madison.

SCOTT, T. C., HILL, C. G., JR. and AMUNDSON, C. H. 1984. Elucidation of the activity decay characteristics of immobilized β-galactosidase using an integral reactor scheme. *Biotechnol. and Bioeng. Symp.* No. 14, 321-39.

SCOTT, T. C., HILL, C. G., JR. and AMUNDSON, C. H. 1985. Determination of the steady-state behavior of immobilized beta-galactosidase utilizing an integral reactor scheme. *Biotechnol. and Bioeng. Symp.* No. 15, 431-45.

SLACK, A. W. 1984. Functional properties of β-lactoglobulin and α-lactalbumin enriched fractions manufactured from cheese whey. Ph.D. thesis. University of Wisconson-Madison.

SLACK, A. W., AMUNDSON, C. H. and HILL, C. G., JR. 1986a. Production of enriched β-lactoglobulin and α-lactalbumin whey protein fractions. J. Food Proc. Pres. *10*, 19-30.

SLACK, A. W., AMUNDSON, C. H. and HILL, C. G., JR. 1986b. Nitrogen solubilities of β-lactoglobulin and α-lactalbumin enriched fractions derived from ultrafiltered cheese whey retentates. J. Food Proc. Pres. *10*, 31-46.

SLACK, A. W., AMUNDSON, C. H. and HILL, C. G., JR. 1986c. Foaming and emulsifying characteristics of fractionated whey protein. J. Food Proc. Pres. *10*, 81-88.

VAN VELTHUIJSEN, J. A. 1979. Food additives derived from lactose: lactitol and lactitol palmitate. J. Agric. Food Chem. *27*, 680-6.

VISWANATHAN, T., BURRINGTON, D. and RICHARDSON, T. 1984. Preparation of rigid, low-density, flame-retardant polyurethane foams from whey permeate. J. Chem. Tech. Biotechnol. *34B*, 52-6.

PREDICTION OF TEMPERATURE IN FROZEN FOODS EXPOSED TO SOLAR RADIATION

K. D. DOLAN[1] and R. PAUL SINGH[2]

University of California
Davis, California 95616

and
D. R. HELDMAN[3]

Campbell Soup Co.
Camden, New Jersey

Accepted for Publication October 22, 1986

ABSTRACT

Simultaneous convective and radiative boundary conditions were incorporated into a one-dimensional three time level implicit finite difference scheme in rectangular coordinates. The scheme was used to calculate temperature profiles and temperature histories in a packaged frozen food exposed to the sun. The solution was verified by thawing slabs of a standard test material (Karlsruhe test substance) and orange juice in different environments. Procedures were developed to measure the temperature distribution and to estimate the radiation properties of the package surface. The maximum thickness of the slabs was 38 mm. The agreement between experimental and predicted food surface and center temperature profiles was determined by corresponding R^2 values ranging from 0.872 to 0.998. The computer program was also used to predict temperature profiles in a 100 × 92 × 92-cm pallet of frozen food exposed to different environmental conditions. Conditions were simulated by varying initial product temperature, product thermal properties, air resistance between the product and the package, external heat transfer coefficient, ambient temperature, and radiation surface properties of the carton.

[1]Author Dolan is Graduate Research Assistant. Department of Agricultural Engineering.;
[2]Author Singh is Professor, Department of Agricultural Engineering and Department of Food Science and Technology. Enquires regarding this paper should be directed to author Singh.
[3]Author Heldman is currently at National Food Processors Association, 1401 New York Ave., N. W., Washington, D. C.

Journal of Food Processing and Preservation 11 (1987) 135-158. All Rights Reserved.

INTRODUCTION

Quality of frozen foods is affected by time and temperature during storage (Van Arsdel 1969, and Singh *et al.* 1984). Undesirable changes in various quality attributes can result from an increase in food temperature. Fluctuations in temperature below the initial freezing point can cause these changes; in many cases partial thawing may occur, but is an extreme example.

During packaging and transport of frozen foods in industry, a variety of conditions may exist. Ambient temperatures may vary from −30 °C during freezing, to 40 °C immediately after, to 5 °C during transport. Dagerskog (1974) presented a typical handling cycle of frozen food shipments.

In this paper, the effect of the most abusive conditions encountered by frozen foods were analyzed, with particular attention given to possible solar exposure. Often the primary reason that frozen food cartons may receive solar exposure is lack of shade structures and storage space at a production facility.

A potential problem exists if the frozen food remains exposed to the sun for too long. Food processors may understandably be willing to risk some quality loss in a food layer along the surface of the pallet in order to maintain desired production rates. If the effect of solar exposure on the food were known, one could know the loss in quality, the amount of exposure tolerable, and the preventive methods needed. Since the only effect of solar exposure considered in this study was an increase in heat flux to the food, the same analysis should apply to the storage of food in conditions without the sun (e.g. within a refrigerated truck). Therefore, the quality loss in the food could be described as a lumped effect of different storage conditions.

OBJECTIVES

To investigate solar exposure as a factor influencing quality loss in frozen foods during the distribution chain, the following objectives were established:

(1) to develop a methodology to obtain the temperature distribution in a package layer and in a frozen food with specified boundary conditions;
(2) to modify a finite difference scheme and use it to predict the temperature profile in a packaged frozen food exposed to solar radiation;
(3) to simulate a pallet of frozen food in typical ambient conditions, and determine the relative importance of the variables influencing heating of the food.

REVIEW OF LITERATURE

Plank (1913) and Charm (1978) developed methods to predict temperature in frozen foods using two basic assumptions, the removal of latent heat at a specific

temperature, and a constant thermal conductivity throughout the frozen region. The result was the prediction of a moving phase-change boundary. However, in a food system, latent heat is transferred over a range of temperatures, a phenomenon simulated by using thermal conductivity k(T) and apparent specific heat, C(T) = dH/dT. A number of studies, including the following, have shown the advantage of using these continuously-varying thermal properties instead of a moving phase-change boundary.

Dagerskog (1974) used a three-dimensional explicit finite difference scheme (enthalpy transformation) to calculate the temperature distribution and history in a pallet load of food during handling in the distribution chain. The variable thermal properties were combined into new variables by integral transformations to obtain a simplified form of the heat conduction equation. In his computations radiative heat transfer was not considered.

Hayakawa *et al.* (1983a, 1983b) simulated the effect of moisture loss and radiative heat exchange with a finite element model simulating two-dimensional freezing in an anisotropic food. Although the agreement between experimental and predicted values was excellent in the conditions chosen, radiation did not significantly influence the freezing rate.

Scott and Heldman (1984) used a modified Crank-Nicolson finite difference scheme without iteration to predict temperatures and quality distribution histories in strawberries. The model simulated one-dimensional heat transfer, including the effect of a packaging layer on the surface. Again, radiation was an insignificant variable.

Cleland and Earle (1977a, 1977b, 1979a, 1979b, 1984a, 1984b) used a three time level finite difference scheme to predict temperatures in slabs, cylinders, spheres, and bricks during freezing. The boundary conditions were specified temperature and convection coefficient.

Most food freezing/thawing studies have used convection or specified surface temperature as the predominant "driving source" of heat flux. In this paper, convection and radiation were simulated simultaneously, the latter being the overriding source of heat flux.

THEORETICAL DEVELOPMENT

Differential Equation

A three time level finite difference computer program was developed to predict the transient one-dimensional temperature profile in rectangular coordinates in a homogeneous solid with temperature-varying thermal properties. The equation to be solved was:

$$C(T)\frac{\partial T}{\partial t} = \frac{\partial}{\partial x}\left(k(T)\frac{\partial T}{\partial x}\right) \qquad (1)$$

with appropriate boundary conditions.

Referring to Fig. 1, the boundary of the solid was the food surface (x = 0). The convective boundary layer, the package thickness and the air layer between the food surface and package was treated as a combined heat resistance. The x coordinate of the package surface was given the value "−a" (Fig. 1), since the actual coordinate varies with the situation.

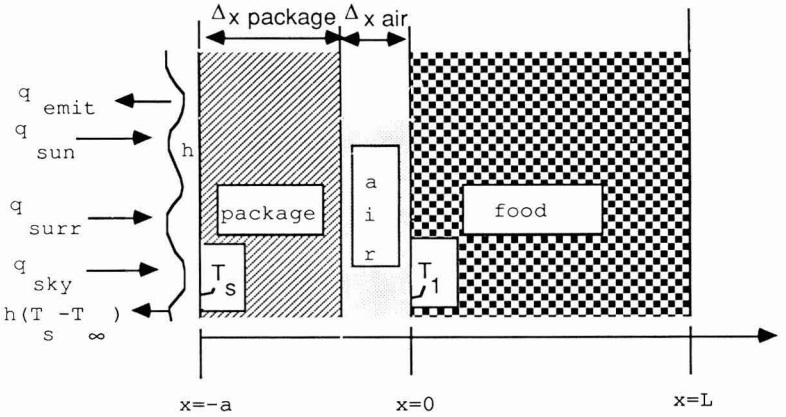

FIG. 1. COORDINATE SYSTEM TO ANALYZE ONE-DIMENSIONAL HEAT TRANSFER IN A PALLET OF FROZEN FOOD

For equations of the type (1), Bonacina and Comini (1971, 1973) and Bonacina *et al.* (1973, 1974) proposed a three time level implicit finite difference scheme developed by Lees (1966) as follows:

$$C(T_i^n)\frac{(T_i^{n+1} - T_i^{n-1})}{2\Delta t} =$$

$$\frac{1}{3\Delta x}\left\{k_{i+1/2}^n\left[\frac{(T_{i+1}^{n+1} - T_i^{n+1})}{\Delta x} + \frac{(T_{i+1}^n - T_i^n)}{\Delta x} + \frac{(T_{i+1}^{n-1} - T_i^{n-1})}{\Delta x}\right]\right.$$

$$\left. - k_{i-1/2}^n\left[\frac{(T_i^{n+1} - T_{i-1}^{n+1})}{\Delta x} + \frac{(T_i^n - T_{i-1}^n)}{\Delta x} + \frac{(T_i^{n-1} - T_{i-1}^{n-1})}{\Delta x}\right]\right\} \quad (2)$$

where

$$k_{i-1/2}^n = k((T_i^n + T_{i-1}^n)/2) \quad (3)$$

$$k_{i+1/2}^n = k((T_{i+1}^n + T_i^n)/2) \quad (4)$$

$$T_i^{n-1} = (T_i^{n-1} + T_i^n + T_i^{n+1})/3 \quad (5)$$

Equation (5) was a modification necessary to eliminate stable oscillations for boundary conditions other than a specified temperature (Cleland and Earle 1977b).

Equations (2) through (5), the "modified" Lees scheme, and boundary Eq. (11), described later, were used in this paper. The scheme is linear and requires no iteration. The computational element for the scheme is shown in Fig. 2.

The scheme was started by assuming stationary initial conditions at $t \leqslant 0$, thereby assigning the initial temperature to T at the 0 and -1 time levels, as suggested by Comini and Bonacina (1974) and Cleland (1985).

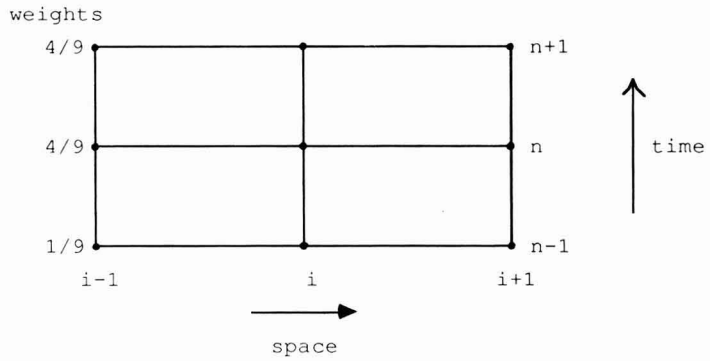

FIG. 2. COMPUTATIONAL SCHEME USED IN THE FINITE DIFFERENCE PROGRAM

Boundary Conditions

The boundary conditions used in this study are a combination of the third and second kind (Newton's Law of Cooling and a prescribed flux, respectively), and one radiative (nonlinear) kind (Fig. 1). In addition, there are two layers of thermal resistance, the corrugated board and an air layer, or "contact resistance", between the board and food.

Experimental trials involving thawing showed that temperature fluctuations on the top of the corrugated board were matched by identical changes at the bottom of the board within 40 s. Therefore, a linear temperature profile across the board at any time was assumed. Essentially this assumption implies that the board stored no heat, but rather conducted all of it, unlike the food. The temperature at the board surface could be predicted by knowing only q into the food and the total resistance between the environment and the food surface. For simplicity, it was assumed that the air layer between the food and the board is equal to zero. Referring to Fig. 1, the heat balance at the board surface node is:

$$q = q_{surr} + q_{sun} + q_{sky} - q_{emit} - h(T_s^n - T_\infty^{n+1}) \qquad \text{heat flux in} \qquad (6)$$

$$q = \frac{-k_{board}}{\Delta x_{board}} (T_1^{n+1} - T_s^n) \qquad \text{heat flux out} \qquad (7)$$

To avoid solving a set of nonlinear equations, T_s was taken at the last known time level, n. The error introduced in q_{emit} is not significant because T_s is an

absolute temperature (Chau 1984). The value of $q_{emit} = \sigma\epsilon_s T_s^4$ was considered a function of T_s^n (the last known time level). The values of q_{surr}, q_{sun}, and q_{sky} were assumed constant over the time considered (< 4 h).

Three separate terms, $q_{surr} + q_{sky} - q_{emit}$, were used instead of the usual radiative boundary condition $\sigma\epsilon(T_\infty^4 - T_s^4)$. The reason was that the latter expression assumes ϵ_∞ is equal to ϵ_s. In this paper ϵ_∞ (emissivity of the sky) and ϵ_s (emissivity of the board surface) ranged independently from 0.75 to 0.86, and q_{surr} was estimated. In addition, the board was assumed to absorb all atmospheric radiation.

Using Eq. (6) and (7), the net heat flux from the environment to the food was solved for in terms of T_∞ and T_1

$$q = U\,[(T_\infty^{n+1} - T_1^{n+1}) + c/h] \tag{8}$$

where

$$c = q_{surr} + q_{sun} + q_{sky} - q_{emit} \tag{9}$$

and the overall heat transfer coefficient is

$$U = \frac{1}{\frac{1}{h} + \sum\left(\frac{\Delta x}{k}\right)} \tag{10}$$

where $\sum\left(\frac{\Delta x}{k}\right)$ is the sum of all conductive thermal resistances between the environment and the food surface.

Using Eq. (8) and the Lee's scheme, the boundary condition equation at the food surface is:

$$\frac{-2\,\Delta t}{3(\Delta x)^2}\,(U\,\Delta x)\,T_\infty^{n+1} + \left[\frac{C(T_1^n)}{2} + \frac{2\,\Delta t}{3(\Delta x)^2}\,(U\,\Delta x + k_{1+1/2}^n)\right] T_1^{n+1}$$

$$\frac{-2\,\Delta t}{3(\Delta x)^2}\,k_{1+1/2}^n\,T_2^{n+1} = \frac{2\,\Delta t}{3(\Delta x)^2}\left\{k_{1+1/2}^n\,[(T_2^n - T_1^n) + (T_2^{n-1} - T_1^{n-1})]\right.$$

$$\left. -U\,\Delta x\,[T_1^n - T_\infty^n) + (T_1^{n-1} - T_\infty^{n-1}) - 3c/h]\right\} + \frac{C(T_1^n)}{2}\,T_1^{n-1} \tag{11}$$

After all temperatures within the food were calculated at time $n+1$, the board surface temperature was calculated as:

$$T_s^{n+1} = q\sum\left(\frac{\Delta x}{k}\right) + T_1^{n+1} \tag{12}$$

where q was calculated from Eq. (8).

A heat balance was used to prevent "jumping" of the latent heat peak, as discussed by Cleland and Earle (1977b). The difference between the initial and final enthalpy of the product was calculated. This value was compared to the heat gained at the boundary during the same time. If the heat balance disagreed by more than 4.0% of unity, the time step was decreased until the criterion was met.

MATERIALS AND METHODS

Experiments

To verify the computer model, thawing experiments were conducted starting with the simplest case and progressively adding one more variable for each new experiment. Consequently, the causes of error were more easily identified and minimized at each of the steps. The Karlsruhe Test Substance, known as Tylose, was used as a food substitute. The experimental progression was as follows:

Trial 1. The frozen Tylose surface temperature was specified as a function of time, thereby eliminating error associated with convection or package layers.

Trial 2. The frozen Tylose was exposed to air only, requiring determination of a heat transfer coefficient, h.

Trial 3. The frozen Tylose was covered with insulation and exposed to air only, requiring determination of h and the thermal properties of the insulation.

Trial 4. The frozen Tylose was covered with insulation and exposed to the sun, requiring determination of h and the thermal and radiative properties of the package. Three experiments were necessary to isolate sources of error.

Trial 4a. An insulation of known thermal conductivity was used.

Trial 4b. Industrial corrugated board was used as insulation in this trial.

Trial 4c. The corrugated board was used as insulation on frozen orange juice.

Only Trial 1 was not conducted in this study. The experimental data for this trial were plotted by Comini *et al.* (1974).

Additional details of the above trials will be given later in this paper.

The thermal conductivity values used for Tylose were those measured experimentally by Bonacina and Comini (1971). The apparent specific heat values used for Tylose were calculated numerically by Cleland and Earle (1984a) from enthalpy values measured experimentally by Reidel (1960). A new sample of Tylose

was prepared when the moisture content (wet basis) of the current sample varied more than 1.0% from 77%, as measured by drying the sample in an oven at 105 °C.

Common Experimental Design

The following design was common to Trials 2-4c. The Tylose was formed into a slab 100 mm × 72 mm with a depth from 21 mm to 38 mm, depending on the experiment. Three to four 30-gauge copper-constantan thermocouples were placed horizontally at different depths within the Tylose. The measurement error was ±1 °C.

Heat transfer coefficients were determined using a lumped-capacity analysis. A 200 × 50 × 50-mm aluminum slab in a polyurethane container, to provide geometry similar to that of the Tylose, was placed in the environment of the Tylose. An exponential regression of the temperature-time data yielded the h values. The data fitting was done over two ranges, one for slab temperatures less than 5 °C, and the other for those greater than 5 °C. The "h" at lower temperatures was expected to be greater because of the added latent heat transferred from the condensing air vapor to the slab or sample (Suzuki and Singh 1984).

The C and k values for orange juice were obtained from Table 1. The initial freezing point of orange juice was found using an osmometer (Advanced Instruments, Model 3W11).

TABLE 1
ORANGE JUICE, $T_f = -1.2$ °C

Temperature (°C)	Thermal conductivity (W/m K)	Temperature (°C)	Volumetric Specific heat ($J/m^3K \times 10^{-6}$)
-40.0	2.40[d]	-40.0	1.9[e]
-11.0	1.93	-15.0	3.3
-10.0	1.89	-11.0	4.7
-7.0	1.76	-10.0	5.4
-5.0	1.61	-9.0	6.2
-4.0	1.49	-8.0	7.4
-3.0	1.32	-7.0	9.1
-2.0	1.05	-6.0	11.8
-1.8	0.97	-5.0	16.3
-1.5	0.82	-4.0	24.5
-1.3	0.68	-3.0	42.6
-1.2	0.59	-2.0	95.6
20.0	0.59	-1.8	119.
		-1.6	153.
		-1.5	174.
		-1.4	157.
		-1.3	133.
		-1.2	104.
		-1.0	32.7
		-0.5	3.58
		20.0	3.58

[d]calculated by the method of Heldman and Gorby (1974)
[e]calculated as the slope of the volumetric enthalpy vs. temperature curve; method of calculating enthalpy is described in Heldman and Singh (1981)

The model food and the orange juice samples were thawed in a polyurethane container shown in Fig. 3. To verify one-dimensional heat transfer, temperatures were measured at a 23-mm depth at three different lateral locations. The agreement between these temperature values was within 0.5 °C during 140 min.

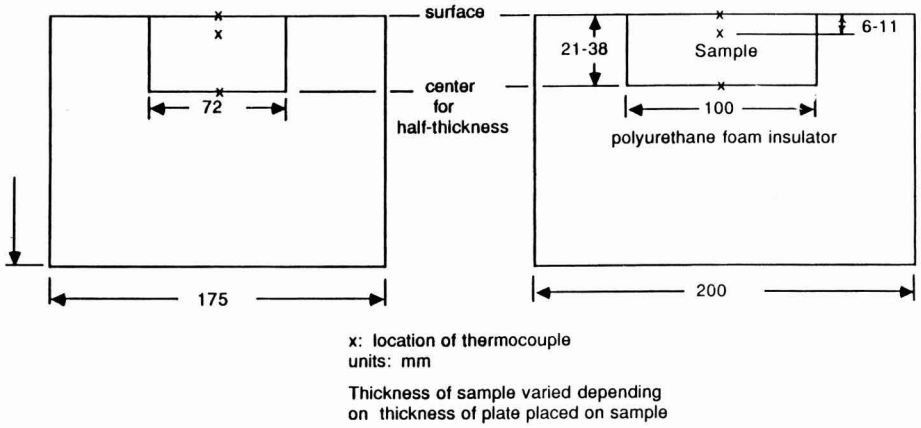

FIG. 3. INSULATING CONTAINER COVERING FIVE SIDES OF SAMPLE

Temperatures were simultaneously recorded and plotted every 20 or 40 s using an automatic Data Acquisition System (Hewlett Packard). Experiments not requiring solar radiation were conducted in an air-conditioning unit providing constant temperature and relative humidity (Aminco-aire, Model 4-5591). Direct solar and diffuse radiation were measured with a pyranometer (LI-COR Model LI-185B).

Individual Experimental Design

The following is the procedure for the six thawing experiments summarized at the beginning of this section.

Trial 1. Comini *et al.* (1974) simulated the first kind of boundary condition by varying the surface temperature of a 38-mm-thick slab as a function of time in freezing and thawing experiments of Tylose. Using a three time level finite difference scheme, they found an error less than 3% of the total temperature change between their experimental and predicted center temperatures. The same varying surface temperature was made an input to the computer program developed in this paper. The predicted center temperature was compared to Bonacina and Comini's experimental curve.

Trial 2. The next simple case was the convection boundary condition. The containers holding the aluminum slab and a 38-mm-thick slab of Tylose were equili-

brated to −34 °C before being placed in an air-conditioning unit providing constant temperature and relative humidity (Aminco-Aire, American Instrument Co.). Temperatures within the Tylose and within the aluminum were recorded at 20-s intervals.

Trial 3. Since the value of thermal conductivity of corrugated board was less reliable than that of other materials, a 103 × 72 × 3-mm plate of polycarbonate (Tuffac, Rohm and Haas Co., Philadelphia, PA, k = 0.193 W/m K) was used. To measure the difference in temperature across the plate, a 30-gauge copper-constantan thermocouple was secured with a piece of tape to the center of each of the two surfaces of the plate.

The 23-mm-thick Tylose sample was wrapped in aluminum foil and frozen to −37 °C. The polycarbonate temperature was brought to that of the air, to simulate a container at ambient conditions into which a frozen food was packaged. The plate surface and edges were fit closely to the surface of the Tylose and against the polyurethane, respectively. The intent of this design was to prevent heat transfer from the plate edges to the center, in compliance with the assumption of one-dimensional heat transfer.

Both containers were placed in the air-conditioning unit at constant temperature and relative humidity. Temperatures within the Tylose and aluminum slabs were recorded at 20-s intervals.

Trial 4a. A 103 × 72 × 4.8-mm plexiglas plate (Rohm and Haas Co., Philadelphia, PA, k = 0.22 W/m K) was spray-painted on one surface with a paint of known emissivity and absorptivity (3M Brand ECP-2200, Non-selective Solar Absorber Coating, emissivity = 0.86, solar absorptivity = 0.96 at 24 °C). Thermocouples were attached as described in Trial 3. Instead of placing the Tylose and aluminum containers in the air conditioning unit, the former (with a 21-mm-thick sample) was set in direct sunlight and the latter was set 1 m away in the shade. Temperatures within the Tylose and aluminum were recorded at 40s interval.

Trial 4b. Experiments were conducted using a 3.5-mm thick corrugated board (Owens-Illinois, Forest Products Division, Tracy, CA) from industrial containers. The corrugated board surface exposed to the sun was painted like the plexiglas in Trial 4a. The two 30-gauge thermocouples were secured under the first layer of paper on either side of the board. A drop of a highly conductive silicone grease was placed at the junction of the thermocouple and the paper. The Tylose slab was 22 mm thick.

Thermal Conductivity of Board

The thermal conductivity of the board was approximated by the method of Cleland and Earle (1976, 1977b) and Cleland (1985), which uses an equation

from Goodman (1964). The concept was to place a board between a body of low thermal conductivity and a constant temperature source, and calculate the overall thermal resistance from the temperature-time plot at the body surface. In this case, the body was Tylose and the source was an aluminum pan in circulating water.

Radiation Properties of Board Surface

The emissivity and solar absorptivity were estimated by placing two 10 × 10-cm corrugated boards, one painted and one unpainted, side by side, facing the sun. The temperature on both surfaces of each board was recorded over 70 min to insure steady state conditions. An infrared thermometer (Heat-Spy Automatic Infrared Thermometer, Wahl Corp., Los Angeles, CA) with a correction for emissivity was used to approximate the emissivity of the unpainted board.

The solar absorptivity was estimated by formulating a heat balance at the surface of each board. The difference between the two steady-state heat flows was attributed to the difference between the values of the solar flux times the solar absorptivity.

Trial 4c. To test the prediction for an actual food product, a 24-mm thick slab of frozen concentrated orange juice beneath unpainted corrugated board was allowed to thaw in the sun. The procedure was identical to that in Trial 4b, except the concentrated juice was poured into an aluminum foil lining placed in the polyurethane container, and the board surface against the slab was waxed. When the juice had frozen, the slab was removed while the center thermocouple was set in place. The intermediate thermocouple was placed in a hole made by penetrating the side of the slab with a 1-mm diameter copper wire. The surface thermocouple was frozen to the surface with drops of orange juice.

Computer Simulation

Pallet loads of frozen orange juice and strawberries exposed to the sun were simulated using the computer program. The k and C values for strawberries (discussed later) were also given by Cleland and Earle (1984a). Figures 4 and 5 are the schematic of the pallet load and the wall cross-section simulated by the program. The front view of the pallet shows a package interface both at and 11.5 cm from the food surface. The stacking pattern, dimensions, and environmental conditions were those found in a frozen food plant during the summer in the San Joaquin Valley in California. All other inputs, except the last two in the following, were taken directly from experimental data of this paper.

Time step, dt = 120 s; space increment, dx = 0.005 m; half-thickness of the pallet, L = 0.46 m; board emissivity = 0.75; board absorptivity = 0.47; T_∞ = 40 °C; h = 18 W/m² K; solar flux = 980 W/m²; contact resistance = 1/50 m² K/W; corrugated board resistance = 1/13.7 m² K/W; initial freezing point for orange juice −1.2 °C, strawberries −0.5 °C; estimated: package resistance

= (0.0005 m)/(0.095 W/m K) = 1/190 m² K/W; air layer resistance = (0.0014 m)/(0.029 W/m K) = 1/21 m² K/W.

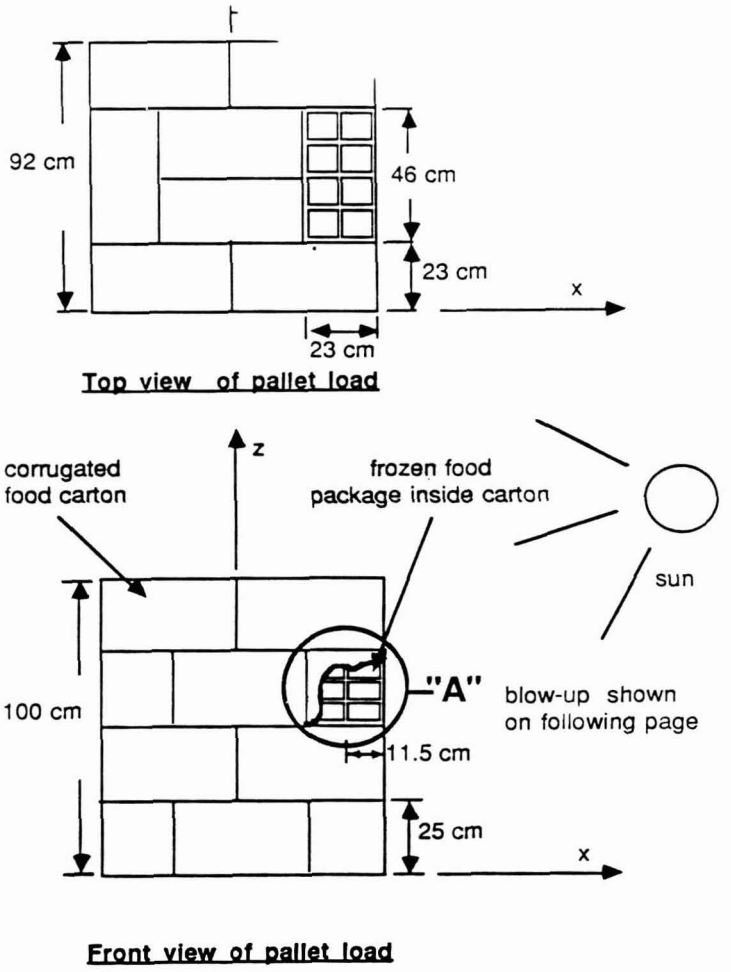

FIG. 4. SCHEMATIC OF STACKED FROZEN FOOD CARTONS EXPOSED TO SOLAR RADIATION

Independent Variables

The inputs to the computer model were obtained using the following procedure. The variables measured directly were L, T_0, T_∞, h, solar flux, and k_{board}. The values of α and ϵ were known from published data for the paint or were estimated for the board surface. Atmospheric radiation was found from charts of Bliss (1961) as a function of dewpoint and dry-bulb temperatures. Trials 2-4c were used to

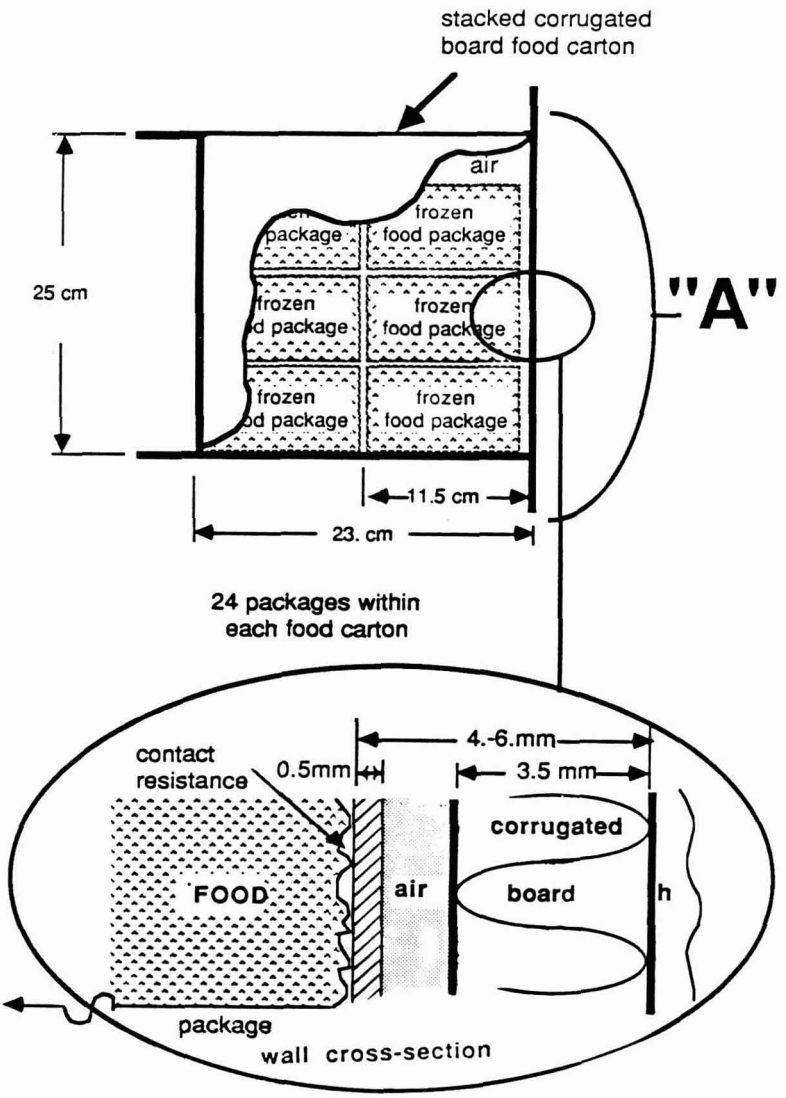

FIG. 5. SCHEMATIC OF FROZEN FOOD PACKAGES WITHIN THE CARTON IN FIG. 4 AND AN ENLARGED VIEW OF CARTON WALL CROSS-SECTION

determine the thermal resistance of the air layer under the board. The temperature difference between the board top and bottom, and between the bottom surface of the board and the food surface were found at different times. At five to ten values of time, the flux through the board ($= -(k\Delta T/\Delta x)_{board}$) was equated to the flux through the air layer ($= (\Delta T/\text{resistance})_{\text{air layer}}$), and the average resistance

was solved for. Surrounding radiation was derived by equating the net radiative and convective flux at the board surface to the flux through the board.

RESULTS AND DISCUSSION

Experiments

Trial 1. The results of the computer program used in this study followed within 0.5 °C the predicted thawing curve of Bonacini and Comini's (1974) which was within 3% of the total temperature change.

Trial 2-4c. The R^2 values for the Trials 2-4c are presented in Table 2. Table 2 shows that the accuracy of the computer scheme was not affected by the progression of complexity in the experiments. The R^2 values for the food surface temperatures were within 6% of each other, and those for the center were within 14%. Furthermore, the R^2 values did not uniformly decrease from trial 2 through trial 4c.

Trials 4b and 4c were chosen as representative results.

TABLE 2
VALUES OF R^2 FROM A LINEAR REGRESSION ON PAIRS OF EXPERIMENTAL AND PREDICTED THAWING CURVES IN THE TRIALS 2-4c.
(Number of temperatures sampled = 20, each taken at 10-min intervals.)

	Values of R^2			
	Location of temperature in slab sample			
Trials	board surface	food surface (x=0)	between surface and center	center
2	---	0.997	0.992 (x=8.5 mm)	0.989 (x=34. mm)
3	---	0.994	---	0.915 (x=25. mm)
4a	0.946	0.946	---	0.872 (x=21. mm)
4b	0.728	0.988	---	0.907 (x=22. mm)
4c	0.761	0.971	---	0.998 (x=24. mm)

"---" indicates the analysis was not applicable or was not performed.

Trial 4b. Results for thawing Tylose under a corrugated board (trial 4b) are shown in Fig. 6. The thermal conductivity of the cardboard was calculated as 0.048 W/m K. This value was within 7% of values obtained by Cleland (1985)

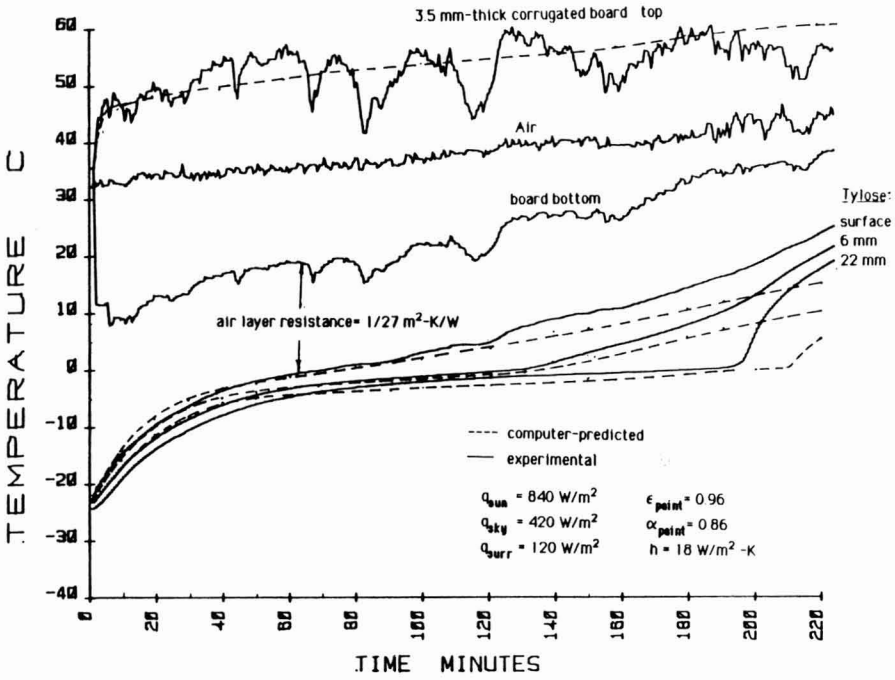

FIG. 6. THAWING CURVE OF A TYLOSE SLAB (L = 22 mm) COVERED BY A PAINTED CORRUGATED BOARD, EXPOSED TO SOLAR RADIATION (Trial 4b)

for other similar boards. The thermal resistance of the board was $(x/k)_{board}$ = 0.0035/0.048 = 1/13.7 m² K/W.

The heat balance at the board surface is shown in Fig. 7. The value of 120 W/m² assumed for radiation from surrounding bodies was reasonable, because a blackbody at 30 °C radiated 480 W/m².

The agreement between predicted and experimental center temperatures was within 1.0 °C through 180 min. As shown in Fig. 6, the predicted center temperature was late in leaving the plateau (at 210 min rather than 195 min) because the other temperatures were predicted low. The center temperature leaves the plateau after the latent heat has been removed. Therefore, the accuracy of this point is a measure of the accuracy of all temperatures in the slab.

The surface temperature was predicted within 0.5 °C up to 80 min. The likely causes for error were the value of h, placement of the thermocouple, and effects of contraction of the food as phase change occurred.

The agreement between values for the temperature at 6 mm was within 1 °C at times < 150 min. (Fig. 6). At longer times, the error in the surface temperature was propagated to this point. However, the rise out of the thawing plateau at

6 mm is predicted within 5 min of the actual rise at time = 130 min, indicating the enthalpy changes in the finite difference scheme was accurate.

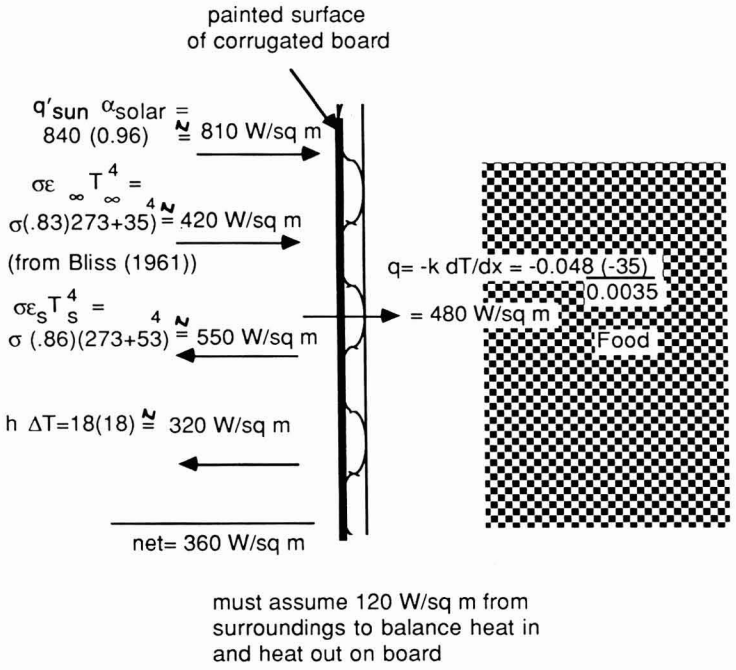

FIG. 7. HEAT BALANCE FOR PAINTED CORRUGATED BOARD COVERING TYLOSE AND EXPOSED TO SOLAR RADIATION (Trial 4b)

Trial 4c. The results for the thawing of frozen orange juice under a corrugated board (Trial 4c) are shown in Fig. 8. The estimated values of solar absorptivity (0.47) and longwave emissivity (0.75), derived from the steady-state experiment using the infrared thermometer, were used in the heat balance.

The accuracy of the predicted solution was comparable to that for Trial 1, as shown by the R^2 values of 0.971 and 0.998 for the food surface and center temperature profiles, respectively (Table 2).

Computer Simulation

Table 3 summarizes the eight simulations showing the effects of six parameters on the temperature profiles: initial product temperature, air layer resistance, h, environmental temperature T_∞, reduced board surface absorptivity α and increased emissivity ϵ, and product thermal properties. An upper limit of $-5\,°C$ was chosen as a criterion to compare temperature profiles.

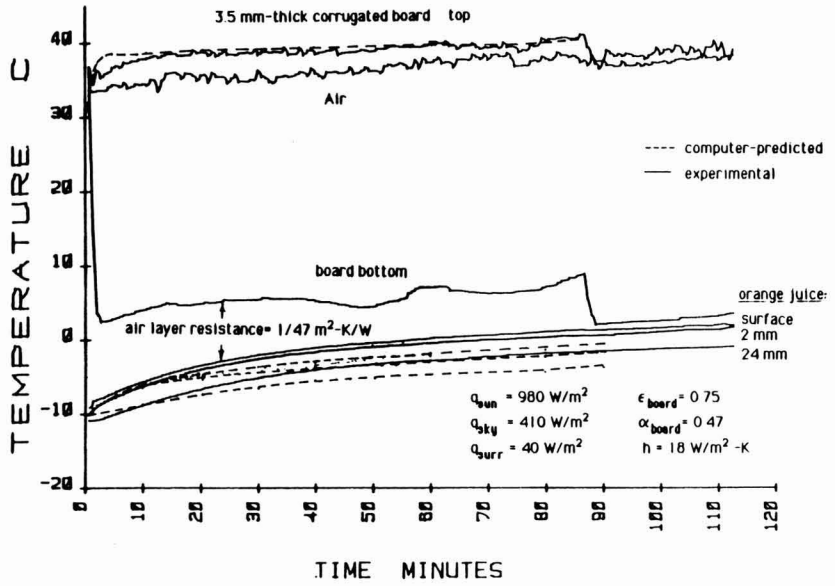

FIG. 8. THAWING CURVE OF AN ORANGE JUICE SLAB (L = 24 mm) COVERED BY AN UNPAINTED CORRUGATED BOARD, EXPOSED TO SOLAR RADIATION (Trial 4c)

The results shown in Table 3 have the following practical applications. In the hot summer day described, Table 3 shows that the surface of a pallet of frozen strawberries would warm almost twice as fast as a pallet of orange juice with identical geometry and conditions (the time for the surface initially at −18 to reach −5 °C was 61 and 117 min, respectively). By freezing a pallet of orange juice to −25 °C instead of −12 °C initially, the time for the surface to reach −5 °C would be lengthened from 48 min. to 227 min. The use of a covering of low solar absorptivity on the same pallet initially at −18 °C would extend the time the surface was less than −5 °C from 117 min. to 193 min.

Table 3 implies that the wind conditions have less influence than the other variables on the thawing time for a pallet. The time periods required for the surface initially at −18 °C to rise to −5 °C with h = 18 and h = 8 W/m² K were 117 and 99 min, respectively. One must consider the ambient temperature when estimating the thawing time. For an ambient temperature of 30 °C rather than 40 °C, Table 3 shows the orange juice surface initially at −18 °C would remain less than −5 °C for 145 min, as opposed to 117 min. This type of table readily presents the relative influence of variables affecting heating of a frozen food pallet.

The following is a more detailed discussion of the effect of the three most influential variables.

TABLE 3
COMPUTER-PREDICTED TIME TO REACH −5°C
IN A STACKED FROZEN FOOD

Trial #	Initial Temp (°C)	Air layer thickness (mm)	h (W/m^2K)	T_∞ (°C)	α, ε	Product	Time (min.) Distance from food surface into pallet 0 cm	1.0 cm
1	-12	1.4	18	40	.47,.75	Orange juice (OJ)	48	105
2	-18	1.4	18	40	.47,.75	OJ	117	200
3	-18	0.2	18	40	.47,.75	OJ	73	143
4	-18	1.4	8	40	.47,.75	OJ	99	176
5	-18	1.4	18	30	.47,.75	OJ	145	236
6	-18	1.4	18	30	.154,.9[a]	OJ	193	296
7	-18	1.4	18	40	.47,.75	Strawberries	61	100
8	-25	1.4	18	40	.47,.75	OJ	227	337

[a]simulates 0.05 mm Titanox on black paint (Chapman, 1974, Table A.13). For example, this substance could be on a cloth cover placed over the pallet.

Effect of Initial Product Temperature

Figure 9 presents the temperature profiles at two times for three different initial temperatures of frozen orange juice. If the rate of change in quality of the food as a function of temperature were known analytically, the program could be used to predict the quality at any time or location. Comparing profiles at identical times, the range of temperatures was less as time increased, i.e. as the temperatures approached the initial freezing point. For example, at time = 20 min, the food surface temperatures (from highest to lowest T_0) were −7.4, −12.0, and −17.8°C. At time = 120 min, the same locations were at −2.6, −5.0, and −9.2°C (Fig. 9).

The distance the heat diffused into the pallet was less than the container width (23 cm) for time $\leqslant$ 120 min. (Fig. 9). The heat diffusion distance was greater at lower initial temperatures (22 and 14 cm for −25 and −12°C, respectively).

Effect of External Convection Coefficient

The product with h = 8 W/m^2 K heated more rapidly than that with h = 18 W/m^2 K (Table 3), but the difference was less than 0.8°C. The effect of h upon

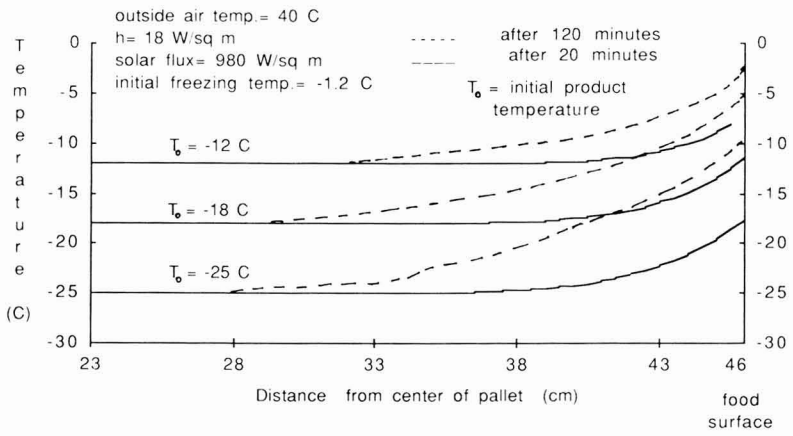

FIG. 9. SIMULATED TEMPERATURE DISTRIBUTION AFTER 20 MIN AND AFTER 120 MIN IN STACKED FROZEN ORANGE JUICE EXPOSED TO THE SUN
(Schematic shown in Fig. 4 and 5).

the thawing depends on whether the pallet is exposed to the sun. When the pallet is exposed to the sun, the board surface temperature rises above that of ambient conditions. Therefore, a higher h will cause more heat loss from the board surface, resulting in slower heating of the food. Conversely, when the pallet is not exposed to the sun, the board surface temperature is below that of ambient conditions. A higher h in this case causes more rapid heating of the food.

Over a range including h values for virtually any ambient condition ($h = 1$ to $h = 70$ W/m^2 K) the difference in profiles was < 1 °C, implying h is an insignificant variable.

Effect of Product Thermal Properties

The influence of product thermal properties on temperature distribution is presented in Fig. 10, where pallet loads of orange juice and strawberries were simulated in identical conditions. The temperature profile in the strawberries was up to 3 °C greater than that in the orange juice during 120 min. In addition, at 120 min, the heat had diffused through the width of the strawberry container (23 cm), whereas the diffusion in the orange juice was through 18 cm. The reason for the more rapid thawing of the strawberries was its lower values of apparent specific heat compared to the orange juice, over the frozen region. The removal of equal enthalpy ($= \int C\, dT$) from each food resulted in a greater increase of temperature for the strawberries.

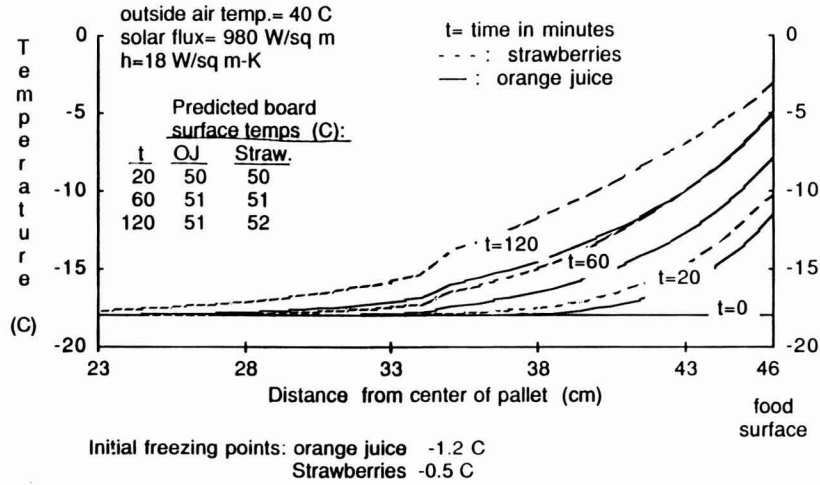

FIG. 10. SIMULATED TEMPERATURE DISTRIBUTION AFTER 20, 60, AND 120 MIN IN STACKED FROZEN ORANGE JUICE AND STRAWBERRIES EXPOSED TO THE SUN
(Schematic shown in Fig. 4 and 5).

The board surface temperatures (T_s) at x = 46.5 cm were not plotted because the temperature scale would be too compressed to compare profiles in the food. The values of T_s are shown in a table on the left side of Fig. 10.

In summary, of the six parameters investigated, the thermal properties and the initial temperature of the product had the greatest effect upon the temperature distribution within the pallet. The air layer between the package and the container influenced the temperature profile primarily within 5 cm of the surface (10% of the half-thickness). The external heat transfer coefficient, within the ranges studied, produced the least difference in temperature profiles.

CONCLUSIONS

(1) The addition of convection, a package layer, and radiation as boundary conditions did not affect the accuracy of predicted food temperatures by more than 5%.

(2) The temperature profile in a packaged frozen food exposed to solar radiation was predicted to be highly dependent upon the thermal properties and the initial temperature of the food, and insensitive to the heat transfer coefficient at the exposed carton surface.

(3) The simulation results showed that the food surface temperature of a pallet of strawberries initially at −18°C reached −5°C in 61 min under ambient conditions of 40°C and in full sun.

LIST OF SYMBOLS

A	area perpendicular to heat flow, m^2
a	distance from food surface to package surface, m
C	volumetric apparent specific heat, $J/(m^3\ K)$
c	defined in Eq. (9), W/m^2
h	convective heat transfer coefficient, $W/(m^2\ K)$
k	thermal conductivity, W/m K
$k_{i+1/2}(T)$	$k((T_i + T_{i+1})/2)$, W/m K
$k_{i-1/2}(T)$	$k((T_{i-1} + T_i)/2)$, W/m K
L	slab half-thickness, m
q	net heat flux, W/m^2
q'	incident heat flux W/m^2 (Fig. 7)
q_{sun}	absorbed solar radiation $= q'_{sun}\alpha_{solar}$, W/m^2
q_{sky}	absorbed atmospheric radiation $= \sigma\epsilon_\infty T_\infty^4 = \sigma T_{sky}^4$, W/m^2
q_{emit}	absorbed surface radiation $= \sigma\epsilon_s T_s^4$, W/m^2
q_{surr}	absorbed radiation from surroundings other than atmosphere, W/m^2
R^2	statistical measure of the agreement between two sets of values
T	temperature, K
T_1	food surface or boundary temperature, K
T_∞	ambient temperature, K
T_f	initial freezing point temperature, K
T_0	initial temperature, K
T_s	surface temperature, K
T_{sky}	mean radiant sky temperature, K
t	time, s
U	overall heat transfer coefficient, $W/(m^2\ K)$
V	volume, m^3
x	distance, m

Superscripts:

n	time coordinate

Subscripts:

0	initial value at time = 0
1	food surface or boundary
∞	ambient, or "a value at $x = \infty$"
air layer	layer of air between board and food surface
board	corrugated board layer
emit	emitted low-temperature radiation

i	space coordinate
package	stiff paper package covering food surface
s	surface
sky	atmosphere, specifically radiation properties of CO_2 and water vapor
sun	sun
surr	surroundings at ambient temperature
t	the value at arbitrary time t

Greek symbols:

α	absorptivity (usually superscripted), dimensionless
Δt	$T_s - T_\infty$
ϵ_∞	apparent sky emissivity, dimensionless
ϵ_s	surface emissivity, dimensionless
σ	Stefan-Boltzmann constant, 5.669×10^{-8} W/m^2 K^4
ρ	density of the product, kg/m^3

REFERENCES

BLISS, R. W. Jr. 1961. Atmospheric radiation near the surface of the ground: a summary for engineers. Solar Energy Vol. *5*, 103-120.

BONACINA, C., COMINI, G., FASANO, A. and PRIMICERIO, M. 1973. Numerical solution of phase-change problems. Int. J. Heat Mass Transfer *16*, 1825.

BONACINA, C., COMINI, G., FASANO, A. and PRIMICERIO, M. 1974. On the estimation of thermophysical properties in nonlinear heat-conduction problems. Int. J. Heat Mass Transfer *17*, 861.

BONACINA, C. and COMINI, G. 1971. On a numerical method for the solution of the unsteady state heat conduction equation with temperature dependent parameters. Proc. XIII Int. Congr. Refrig. Vol. *2*, 329.

BONACINA, C. and COMINI, G. 1973. On the solution of the nonlinear heat conduction equations by numerical methods. Int. J. Heat Mass Transfer *16*, 581.

CHAU, K. V. 1984. Simulation of heat and mass transfer in products with internal heat generation and transpiration. ASAE Paper No. 84-6513.

CLELAND, A. C. 1985. Personal communication.

CLELAND, A. C. and EARLE, R. L. 1976. A new method for prediction of surface heat transfer coefficients in freezing. Bull. I.I.A., Annexe-1, 361.

CLELAND, A. C. and EARLE, R. L. 1977a. A comparison of analytical and numerical methods of predicting the freezing times of foods. J. Food Sci. *42*(5), 1390.

CLELAND, A. C. and EARLE, R. L. 1977b. The third kind of boundary condition in numerical freezing calculations. Int. J. Heat Mass Transfer *20*, 1029.

CLELAND, A. C. and EARLE, R. L. 1979a. A comparison of methods for predicting the freezing times of cylindrical and spherical foodstuffs. J. Food Sci. *44*, 958.

CLELAND, A. C. and EARLE, R. L. 1979b. Prediction of freezing times for foods in rectangular packages. J. Food Sci. *44*, 964.

CLELAND, A. C. and EARLE, R. L. 1984a. Assessment of freezing time prediction methods. J. Food Sci. *49*, 1034.

CLELAND, A. C. and EARLE, R. L. 1984b. Freezing time predictions for different final product temperatures. J. Food Sci. *49*, 1230.

COMINI, G. and BONACINA, C. 1974. Application of computer codes to phase-change problems in food engineering. I.I.R. Meeting Commissions B1, C1, C2. Bressanone, 17-20 Sept.

COMINI, G., BONACINA, C. and BARINA, S. 1974. Thermal properties of foodstuffs. Bull. Int. Inst. Refrig., Annexe-3, 163.

COMINI, G., DEL GUIDICE, S., LEWIS, R. W. and ZIENKIEWICZ, O. C. 1974. Finite element solution of nonlinear heat conduction problems with special reference to phase change. Int. J. Num. Meth. Engng. *8*, 613.

DAGERSKOG, M. 1974. Development of a computer program to simulate temperature distribution and time-temperature exposure of packed frozen food during handling in the distribution chain. Swedish Institute of Food Preservation Research (SIK).

DOLAN, K. D. 1985. Prediction of temperature in stacked frozen foods. Master's Thesis, University of California, Davis.

GOODMAN, T. R. 1964. Application of Integral Methods to Transient Nonlinear Heat Transfer, Adv. Heat Trans. *1*, 59. Cited by Cleland and Earle, 1976. A new method for prediction of surface heat transfer coefficients in freezing. Bull. I.I.R., Annexe-1, 361.

HAYAKAWA, K., NONINO, C. and SUCCAR, J. 1983a. Two dimensional heat conduction in food undergoing freezing: predicting freezing time of rectangular or finitely cylindrical food. J. Food Sci. *18*, 1841.

HAYAKAWA, K., NONINO, C., SUCCAR, J., COMINI, G. and DEL GIUDICE, S. 1983b. Two dimensional heat conduction in food undergoing freezing: development of computerized model. J. Food Sci. *48*, 1849.

HELDMAN, D. R. and GORBY, D. P. 1974. Prediction of thermal conductivity in frozen foods. ASAE Paper No. 74-6017.

HELDMAN, D. R. and SINGH, R. P. 1981 *Food Process Engineering*, 2nd ed. The AVI Publishing Co., Westport, CT.

LEES, M. 1966. A linear three-level difference scheme for quasilinear parabolic equations. Math. Comput. *20*, 516.

PLANK, R. Z. 1913. Ges. Kalte-Ind. *20*, 109 (in German). Cited by Ede, A. J. 1949. The calculation of the freezing and thawing of foodstuffs. Mod. Refrig. *52*, 52. Cited by Heldman, D. R. and Singh, R. P. 1981. *Food Process Engineering*, 2nd ed. The AVI Publishing Co., Westport, CT.

RIEDEL, L. 1960. A test substance for freezing experiments. Kaltetechnik. Karlsruher. *12*, 222-225.

SCOTT, E. P. and HELDMAN, D. R. 1984. Prediction of frozen food quality during fluctuating temperature storage. ASAE Paper No. 84-6570.

SINGH, R. P., WELLS, J. H., DOLAN, K. D., GONNET, E. J. and MUNOZ, A. M. 1984. Critical evaluation of time-temperature indicators for monitoring quality changes in stored subsistence. Report prepared for United States Army Natick Research & Development Center. Natick, MA. October.

SUZUKI, K. and SINGH, R. P. 1985. A computer-aided calculation of temperature history during thawing of foods. Trans. of ASAE, *28*(4), 1330-1335.

VAN ARSDEL, W. B., COPLY, M. J. and OLSON, R. L. 1969. *Quality and Stability of Frozen Foods*. Wiley-Interscience, New York.

EFFECT OF TUMBLING SOLUTIONS CONTAINING ETHANOL ON TENDERNESS AND YIELD OF BROILER BREASTS[1]

JAMES L. HEATH[2] and SANDRA L. OWENS

Department of Poultry Science
University of Maryland
College Park, MD 20742

Accepted for Publication October 22, 1986

ABSTRACT

There is increasing interest in adding alcohol-containing ingredients to marinades used in preparing chicken meat for the fast food market. Since there is a lack of publised information on this subject, studies were conducted to determine the effect of tumbling broiler breasts in solutions containing alcohol on tenderness and yield.

Tumbling broiler breasts in ethanol (ETOH) caused an initial toughening that was overcome by increasing the length of time they were tumbled and by increasing holding time after tumbling. This tenderizing effect was enhanced at the higher levels of ETOH. Moisture content of the tissue was larger when 4% ETOH was used (study 1) but neither length of tumbling time nor holding time had any affect. Drip loss data showed no trends important to this study. Breasts tumbled in 4% ETOH in study 1 had smaller cooking losses and cooking losses decreased as holding time increased. Proximate composition of the tumbling solutions was not affected by the various levels of alcohol in the tumbling solutions.

INTRODUCTION

The portion of the fast food — convenience food industry that markets poultry meat products is very competitive and they must develop new and unique prod-

[1]Scientific Article No. *A4461* Contribution No. *7453* of the Maryland Agriculture Experiment Station (Department of Poultry Science).

[2]To whom correspondence should be sent.

Journal of Food Processing and Preservation 11 (1987) 159-169.

ucts that are tender and can be produced with maximum yield. These companies have become aggressive in their product development operations and often use short term marinade exposure and tumbling in their product preparations. Because of success in other muscle food products, a lot of interest has been directed toward the use of alcohol-containing ingredients in marinade recipes.

Wine is often used in marinade recipes to impart a particular flavor and to tenderize meat products. Lind *et al.* (1971) used a wine-vinegar solution to marinade beef rounds for a 48-h period and found it increased tenderness and juiciness. Tenderization is a result of the low pH of the marinade and wine has a pH range of 2.9-3.0 (Wenham and Locker 1976). This agreed with the work of Kotula and Heath (1986) in their studies of precook tumbling of hot-boned broiler breasts in acetic acid solutions. They found that as acetic acid concentration increased and pH decreased, shear values decreased. No evidence was found in the literature that indicated alcohol in the marinade affected tenderness or yield of chicken products.

The fast food industry uses tumbling in their precook treatments to increase the rate of penetration of flavor and tenderizing conponents into the tissue. Chen (1980) found that marinating increased the final yields of fried chicken parts when compared to controls that were not marinated. He also found that penetration of the marinade into chicken parts during 4 h of still (not tumbled) marination can be equaled by 10 min exposure to the marinade during tumbling. Babji *et al.* (1982) found that short term tumbling and salting did not affect the chemical composition of turkey breast muscle. Tumbling had no affect on moisture content or water holding capacity although cooking losses were lower and pH was higher in tumbled carcasses when compared to the nontumbled treatment. A marinade was not used during tumbling in these studies.

The effects of alcohol on chicken tissue have not been studied in relation to short term marindade exposure and tumbling. The objective of this research was to determine if short term exposure to alcohol in the tumbling solution has an effect on tenderness and yield of broiler breasts.

MATERIALS AND METHODS

Sample Preparation

Broiler chickens were reared to 52 days of age using University of Maryland management techniques and were fed commercially formulated and mixed broiler rations. The birds were exsanguinated, defeathered and eviscerated in the Department of Poultry Science's pilot facility. With the exception of those used in study 4 where cold shortening was induced, carcasses were chilled during the period of time allowed for resolution of rigor mortis. Breasts were removed from each carcass, split into left and right halves at the keel (sternum) and the intact muscles

were cut from the bone. Deboned breasts were randomly assigned to treatments and the various studies were conducted as described.

Study 1

Treatment groups of 10 broiler breasts were tumbled in either 2L of water or aqueous solutions containing 1, 2 or 4% ethanol (ETOH) for either 2, 4 or 8 min. The alcohol solutions were made with 95% ETOH. Tumbling was accomplished at room temperature (22-24C) using a Mar-2 tumbler (Diversified Equipment Corp., Columbus, Ohio) operating at 44 revolutions per minute. Breasts were weighed, tumbled and placed on a wire mesh screen and held for 15 min. This provided a uniform period of time for the samples to drip before they were reweighed. Tumbling loss was the difference in weight before tumbling and after the breasts were allowed to drip.

The breasts were placed on ridged, aluminum trays, covered with aluminum foil and cooked at 177C in a Market Forge forced air oven to an internal temperature of 83C. The samples were allowed to cool to room temperature before the post cook weight was taken and then they were sliced. Cooking loss was the difference in weight before cooking and after cooling. Two 3 mm thick slices were removed from the side of the pectoralis superficialis that was next to the skin before deboning and the second slice was saved for shear analysis (Wise and Stadelman 1959; Goodwin *et al.* 1962).

A 3x3 cm sample was cut from the second slice using a template and weighed. These samples were removed from the same area of each muscle so that the fibers were orientated in the same direction. Samples were positioned in a Kramer Shear Cell (Model -500-412D- 2444) so that the muscle fibers were at a 90 degree angle to the long axis of the shear blade surface and in the same location in the cell each time. Resistance to shear was determined with an Instron Universal Testing Instrument (Model -1130) using the 100 kg load cell and the samples were sheared at room temperature (22-24C). Percent water was determined after cooking using procedures described in AOAC, 1984.

Study 2

Three trials (n = 8) were conducted in which breasts were tumbled for 10 min in 2L of either water or aqueous solutions containing 2 and 4% ETOH. They were removed from the tumbler and held for 10, 30 or 50 min prior to cooking to determine if alcohol would have an effect if it remained in the raw tissue for longer periods of time. The breasts were placed on a wire mesh screen during the holding period so that the tumbling solution would drip away and not soak the tissue. Tumbling loss was the weight lost during tumbling and the holding period. After the completion of tumbling, triplicate samples of the tumbling solution were removed after the solution was thoroughly mixed and amounts of water,

protein, ether extract and ash were determined by methods in AOAC (1984). Shear values, H_2O, and cooking losses were determined as described in study 1. The 10 min tumbling time was used to make sure the effects of tumbling found in study 1 would be realized in this study.

Study 3

Broiler breasts were tumbled for 10 min in 2L of water or aqueous solutions containing .2, .4, .8 and 1.6% ETOH, removed from the tumbler and held for 10, 30 or 50 min before cooking. Two trials with the same experimental design were conducted and in each n = 8. All other procedures and parameters measured were as described in study 2.

Study 4

This study was conducted the same as study 3 with the following exceptions. Immediately after the carcasses were eviscerated they were washed, sealed in individual plastic bags and placed in frozen storage until they were thawed in preparation for treatment. The carcasses were thawed in the plastic bags in a refrigerator overnight. The breasts were removed from the thawed carcasses, deboned and used as needed in the study.

Statistical Analysis

The studies were a randomly assigned, factorial arrangement of treatments that allowed the parameters to be compared by Analysis of Variance. Significantly different means were separated and identified by use of the Student-Newman-Kuels (SNK) test. Statistical procedures were those of Sokal and Rohlf (1972) and Steel and Torrie (1960).

RESULTS AND DISCUSSION

Study 1

Tumbling breasts in water for longer than 2 min decreased ($P < .05$) shear values (Table 1). Addition of 1% ETOH to the tumbling solution increased ($P < .05$) shear values when the samples were tumbled for 4 and 8 min and compared to those tumbled in water without added ETOH for the same periods of time. Tumbling for 8 min in 2% and for 4 and 8 min in 4% ETOH produced breasts with shear values that were not ($P > .05$) different from those tumbled for the respective times in water. These data show an effect due to tumbling and ETOH content of the tumbling solution. ETOH caused toughening of the tissue resulting in larger shear values when 1% ETOH was used but appeared to facilitate

TABLE 1
EFFECT OF TUMBLING BROILER BREASTS IN WATER AND ETHANOL (ETOH) SOLUTIONS FOR 2, 4 AND 8 MIN ON SHEAR VALUES. TUMBLING LOSS, H_2O AND COOKING LOSS

ETOH (%)	TUMBLE (min)	SHEAR VALUE (kg/g)	TUMBLING LOSS (%)	H_2O (%)	COOKING LOSS (%)
0	2	9.0 ± 1.0^{cd}[1]	3.8 ± .6^{c}	66.5 ± .3^{a}	24.2 ± .5^{c}
	4	5.7 ± .5^{a}	3.0 ± .5^{bc}	67.1 ± .2^{ab}	26.4 ± .6^{d}
	8	5.5 ± .5^{a}	1.4 ± .8^{abc}	67.9 ± .2^{ab}	26.6 ± .4^{d}
1	2	9.5 ± 1.3^{d}	2.2 ± .4^{abc}	67.3 ± .5^{ab}	27.5 ± .4de
	4	7.5 ± .9^{bc}	2.1 ± .6^{abc}	68.1 ± .4^{b}	27.2 ± .8^{d}
	8	8.0 ± 1.3^{bcd}	.2 ± .2^{a}	67.3 ± .5^{ab}	29.9 ± .4^{ef}
2	2	8.3 ± .8^{cd}	2.9 ± .6^{bc}	67.0 ± .3^{ab}	26.9 ± .5^{d}
	4	9.3 ± 1.3^{d}	1.0 ± .8^{ab}	66.4 ± .4^{a}	28.2 ± .3de
	8	5.2 ± .6^{a}	.8 ± .6^{ab}	67.3 ± .4^{ab}	31.7 ± .5^{f}
4	2	9.1 ± 1.0^{d}	2.9 ± .5^{bc}	70.2 ± .4^{c}	12.8 ± .6^{a}
	4	6.5 ± .5^{ab}	2.9 ± .6^{bc}	69.9 ± .3^{c}	22.3 ± 1.1^{c}
	8	5.9 ± .4^{a}	3.1 ± .4^{bc}	70.2 ± .5^{c}	17.7 ± 1.5^{b}

[1]Means ± Standard Deviation in each column with unlike superscripts are significantly ($P<.05$) different.

the reduction of shear values by tumbling when the ETOH content was increased to 2 or 4%

Significant ($P < 0.5$) differences were found when the tumbling loss data were analyzed. The range test (SNK) used to separate the means showed no trends important to this study.

The addtion of 4% ETOH to the tumbling solution caused an increase ($P < .05$) in the amount of H_2O retained in the cooked breast tissue and this could improve cooked yield and organoleptic quality. Length of time the breasts were tumbled had no affect on the amount of H_2O found after cooking and indicated that the increase in tenderness found when the breasts were tumbled was not a result of retained moisture.

Increased tumbling time in water and 4% ETOH from 2 to 4 min and in 2% ETOH from 4 to 8 min increased ($P < .05$) cooking losses. There was a decrease ($P < .05$) in cooking loss when tumbling time was increased from 4 to 8 min

in 4% ETOH. Breasts tumbled in 4% ETOH for 2, 4 and 8 min had smaller cooking losses than those tumbled in water or 1 and 2% ETOH when the respective tumbling times were compared. The 4% ETOH level also resulted in the largest amount of H_2O in the cooked tissue which agreed with the reduced cooking losses found in these samples. This agreed with the findings of Post (1981) when tumbling increased yield of drumsticks and this increase was attributed to the retention of tumbling solution by the tissue. These effects of ETOH concentration and tumbling time have implications in yield and marinade component retention after cooking.

Study 2

Analysis of variance showed that there were no ($P > .05$) differences between trials for any of the parameters so the data were combined for presentation in Table 2. The use of water or 2 and 4% ETOH as the tumbling media and increased holding times of 10, 30 and 50 min after tumbling had no affect ($P > .05$) on shear values or percent H_2O in the cooked breasts. Exposure of the tissue to ETOH concentrations shown in study 1 to have increased tenderness and the 10 min tumbling period produced a uniformly tender sample. Increased retention of H_2O found at the 4% ETOH level in study 1 was not found in this study. The

TABLE 2
EFFECT OF HOLDING BROILER BREASTS FOR 10, 30 AND 50 MIN AFTER TUMBLING FOR 10 MIN IN WATER AND ETHANOL (ETOH) SOLUTIONS ON SHEAR VALUES, TUMBLING LOSS, H_2O AND COOKING LOSS

ETOH (%)	HOLD (min)	SHEAR VALUE (kg/gm)	TUMBLING LOSS (%)	H_2O (%)	COOKING LOSS (%)
0	10	5.7 ± .8^{a}[1]	1.3 ± .5^{a}	66.9 ± .4^{a}	27.9 ± .4^{ab}
	30	5.7 ± .8^{a}	2.0 ± .6^{ab}	66.8 ± .4^{a}	28.4 ± .7^{ab}
	50	6.1 ± .7^{a}	2.1 ± .4^{ab}	67.1 ± .3^{a}	27.8 ± .7^{ab}
2	10	6.1 ± .6^{a}	.9 ± .1^{a}	67.1 ± .4^{a}	27.3 ± .8^{ab}
	30	5.8 ± .5^{a}	1.1 ± .4^{a}	67.7 ± .3^{a}	26.5 ± .6^{a}
	50	7.0 ± .7^{a}	3.5 ± .5^{bc}	67.1 ± .4^{a}	26.1 ± .
4	10	6.4 ± .8^{a}	.7 ± .1^{a}	67.0 ± .4^{a}	29.5 ±
	30	6.3 ± .7^{a}	2.4 ± .5^{abc}	67.2 ± .4^{a}	27.7 ±
	50	6.7 ± 1.0^{a}	3.9 ± .4^{c}	66.7 ± .4^{a}	26.7 ±

[1]Means ± Standard Deviation in each column with unlike superscripts are significantly ($P<.05$) different.

difference between the two studies could have been due to the length of time the breasts were held after they were tumbled.

Tumbling loss increased ($P < .05$) in samples when the length of time they were held after tumbling was increased from 30 to 50 min and from 10 to 50 min for breasts tumbled in 2 and 4% ETOH, respectively. These differences in yield were expected because of the differences in the length of time the breasts were allowed to drip. Samples tumbled in ETOH had increased ($P < .05$) tumbling losses as holding time increased but those tumbled in water did not have a similar difference.

Cooking losses decreased ($P < .05$) as holding time increased from 10 to 50 min in the samples tumbled in 4% ETOH. Much of the decrease in cooking losses could have resulted from less pick-up of the tumbling solution by the samples. This would provide fewer volatiles that could be lost during cooking. The amount of ETOH in the tumbling solution did not affect ($P > .05$) cooking losses. This differs from study 1 where 4% ETOH in the tumbling solution reduced cooking losses.

Extraction of components from the breast by the physical action of tumbling and by any solvent activity of the tumbling solution was not large. Proximate analysis of the tumbling solutions showed no differences ($P > .05$) in the material extracted from the breasts due to the various treatments (Table 3).

TABLE 3
COMPOSITION OF THE TUMBLING MEDIA AFTER TUMBLING BROILER BREASTS IN WATER AND ETHANOL (ETOH) SOLUTIONS[1]

ETOH (%)	H_2O (%)	PROTEIN (%)	ETHER EXTRACT (%)	ASH (%)
0	97.8	1.8	<.1	.16
2	98.2	1.5	<.1	.19
4	98.1	1.6	<.1	.16

[1]No significant (P>.05) differences were found for any parameter.

Study 3

Study 1 showed that <2% ETOH resulted in increased shear values that were not improved by tumbling for up to 8 min. Study 2 indicated that this problem of increased toughness may be reduced by tumbling for 10 min since no differences in shear values were found in Table 2 for the 2 and 4% alcohol levels. The lowest concentration of ETOH used in the two previous studies was 1%.

The purpose of this study was to investigate the effects of tumbling breasts in solutions containing 0 to 1.6% ETOH and holding them for 10 to 50 min before

cooking. Analysis of variance indicated no ($P > .05$) significant difference between trials for the various parameters so the data were combined for presentation in Tables 4 and 5.

TABLE 4
EFFECT OF TUMBLING BROILER BREASTS IN WATER AND ETHANOL (ETOH) SOLUTIONS FOR 10 MIN AND HOLDING THEM FOR 10, 30 AND 50 MIN ON SHEAR VALUES, TUMBLING LOSS, H_2O AND COOKING LOSS

ETOH (%)	HOLD (min)	SHEAR VALUE (kg/g)	TUMBLING LOSS (%)	H_2O (%)	COOKING LOSS (%)
0	10	5.1 ± .7^{abc}[1]	2.2 ± .9^{ab}	66.7 ± .4^{ab}	33.8 ± .9^{bc}
	30	5.9 ± .7^{bcd}	3.5 ± .6^{abcde}	65.8 ± .4^{ab}	34.1 ± .8^{bc}
	50	3.5 ± .5^{a}	4.1 ± 1.0^{bcdef}	67.9 ± .6^{b}	31.3 ± 1.0^{b}
.2	10	8.4 ± .9^{def}	1.4 ± .6^{a}	62.5 ± .5^{a}	33.6 ± .9^{bc}
	30	9.8 ± 1.3^{f}	3.9 ± .5^{bcdef}	66.2 ± .5^{ab}	34.0 ± .8^{bc}
	50	9.2 ± .9^{ef}	5.8 ± .7^{ef}	67.5 ± .5^{b}	27.9 ± 1.1^{a}
.4	10	7.0 ± .6^{cde}	1.4 ± .3^{a}	66.7 ± .5^{ab}	35.8 ± .7^{c}
	30	6.6 ± .7^{bcd}	5.3 ± .9^{def}	67.4 ± .7^{b}	33.1 ± .8^{bc}
	50	7.4 ± 1.0^{cde}	4.1 ± .9^{bcdef}	66.1 ± .6^{ab}	33.1 ± 1.3^{bc}
.8	10	5.4 ± .6^{abc}	2.1 ± 1.0^{ab}	66.0 ± .3^{ab}	35.1 ± .8^{c}
	30	6.0 ± .8^{bcd}	4.7 ± .5^{cdef}	65.3 ± .5^{ab}	34.5 ± 1.0^{bc}
	50	4.1 ± .5^{ab}	3.3 ± .5^{abcd}	65.5 ± .3^{ab}	32.3 ± .7^{bc}
1.6	10	5.1 ± .5^{abc}	2.6 ± .8^{abc}	66.6 ± .4^{ab}	33.4 ± .8^{bc}
	30	6.9 ± .7^{cde}	5.0 ± .9^{cdef}	67.2 ± .3^{b}	32.5 ± 1.4^{bc}
	50	6.2 ± .5^{bcd}	6.2 ± .7^{f}	66.2 ± .5^{ab}	32.3 ± .6^{bc}

[1]Means ± Standard Deviation in each column with unlike superscripts are significantly ($P<.05$) different.

Larger ($P < .05$) shear values were found when breasts that were tumbled in .2% ETOH for 10 min and held before cooking for 10, 30 and 50 min were compared to samples tumbled in water and held for the respective periods of time (Table 4). When the same comparisons were made for the .4, .8 and 1.6% ETOH treatments, with the exception of those breasts tumbled in .4 and 1.6% ETOH and held for 50 min, no ($P > .05$) differences were found. The two exceptions had larger shear values than the respective breasts tumbled in water. This showed

that addition of ETOH at .2% concentration resulted in increased shear values. In all but two comparisons for the respective holding times, breasts tumbled in .2% ETOH had higher shear values than those tumbled in .4, .8 and 1.6% ETOH indicating that higher concentrations of ETOH resulted in lower shear values. Holding the breasts for 50 min after tumbling in water resulted in smaller ($P < .05$) shear values than those found after holding for 30 min. No differences were found within the ethanol treatments attributable to holding time.

A significant ($P < .05$) interaction was found between holding time and ETOH level when tumbling loss means were analyzed. Tumbling loss tended to increase as holding time increased in the breasts tumbled in .2, .4 and 1.6% ETOH treatments. In the .8% ETOH treatments, tumbling loss increased from the 10 to 30 min holding period but when holding time was increased to 50 min, tumbling loss decreased slightly so that it was not different from the samples held for 10 and 30 min.

ETOH concentration in the tumbling solution had no ($P > .05$) affect on the percent H_2O in the tissue. Except when percent H_2O in the cooked breasts held for 10 and 50 min were compared after tumbling in .2% ETOH, no ($P > .05$) differences were found attributable to holding time. Essary *et al.* (1968) found that bound water contributed to tenderness. Since there was only one difference in water content of the tissue, changes in the water holding capacity in this study were not responsible for the changes in tenderness.

Increased ETOH in the tumbling solution had no ($P > .05$) affect on cooking losses after holding for 10 and 30 min. Tumbling in .2% ETOH resulted in smaller ($P < .05$) cooking losses after holding for 50 min than those tumbled in water and the other alcohol levels.

Proximate composition of the tumbling solutions after the various treatments were not different (Table 5).

TABLE 5
COMPOSITION OF THE TUMBLING MEIA AFTER TUMBLING BROILER BREASTS IN WATER AND ETHANOL (ETOH) SOLUTIONS[1]

ETOH %	H_2O %	PROTEIN %	ETHER EXTRACT %	ASH %
0	98.2	1.5	.1	.1
.2	98.5	1.2	.1	.1
.4	98.4	1.2	.1	.1
.8	98.4	1.4	.1	.1
1.6	98.4	1.3	.1	.1

[1]No significant (P>.05) differences were found for any parameter.

Study 4

Broiler breasts used in previous studies were from carcasses that had been handled in such a manner during evisceration and sample preparation that breasts would have uniform shear values before treatment. These studies used tissue which may not have allowed sufficient additional tenderization to demonstrate treatment effects. To determine if this was a problem, this study was designed to produce breasts that had larger shear values prior to treatment. The method used to produce tougher tissue was "cold shortening" and consisted of freezing the tissue immediately after evisceration and before the resolution of rigor mortis. The product was used after it was thawed completely (Smith *et al.* 1969). The desired results were realized and are evident when the shear values of breasts tumbled in water are compared to the same treatment in the other studies (Table 6).

TABLE 6
EFFECT OF TUMBLING COLD SHORTENED BROILER BREASTS IN WATER AND ETHANOL (ETOH) SOLUTIONS FOR 10 MIN AND HOLDING FOR 10, 30 AND 50 MIN ON SHEAR VALUES, TUMBLING LOSS, H_2O AND COOKING LOSS

ETOH (%)	HOLD (min)	SHEAR VALUE (kg/g)	TUMBLING LOSS (%)	H_2O (%)	COOKING LOSS (%)
0	10	10.2 ± 1.2[a][1]	2.8 ± 1.3[a]	66.9 ± .6[a]	32.2 ± 1.0[a]
	30	11.4 ± 1.1[a]	5.4 ± 1.0[a]	66.0 ± .5[a]	32.6 ± .3[a]
	50	8.5 ± 1.7[a]	5.0 ± .7[a]	67.3 ± .6[a]	28.9 ± .6[a]
.2	10	8.3 ± .9[a]	4.6 ± 1.3[a]	66.8 ± .6[a]	31.0 ± 1.0[a]
	30	10.9 ± 1.4[a]	6.2 ± 1.5[a]	67.3 ± .8[a]	30.9 ± .5[a]
	50	8.0 ± .9[a]	5.5 ± .8[a]	69.6 ± .6[a]	25.6 ± .7[a]
.4	10	9.1 ± 2.1[a]	3.8 ± 1.1[a]	66.6 ± .3[a]	29.5 ± 1.4[a]
	30	7.3 ± 1.6[a]	4.1 ± 1.3[a]	66.9 ± .4[a]	29.3 ± 1.4[a]
	50	9.1 ± 1.3[a]	6.4 ± .8[a]	67.0 ± .5[a]	30.5 ± 1.5[a]
.8	10	9.9 ± 1.2[a]	3.2 ± .5[a]	66.6 ± .4[a]	31.5 ± 1.3[a]
	30	8.8 ± 1.5[a]	6.1 ± .6[a]	67.6 ± .7[a]	29.5 ± .7[a]
	50	9.0 ± 1.9[a]	7.2 ± .8[a]	67.6 ± .4[a]	23.2 ± 1.2[a]
1.6	10	10.2 ± .9[a]	4.0 ± .5[a]	66.7 ± .3[a]	28.7 ± 2.0[a]
	30	7.0 ± .4[a]	6.6 ± .7[a]	68.0 ± .7[a]	30.5 ± .9[a]
	50	7.9 ± 1.1[a]	6.3 ± .6[a]	66.7 ± 1.0[a]	28.3 ± .7[a]

[1]Means ± Standard Deviation in each column with unlike superscripts are significantly ($P < .05$) different.

No ($P > .05$) differences were found in shear values, tumbling losses, percent H_2O, or cooking losses attributable to ETOH concentration or holding time (Table 6). Apparently the chemical and/or physical changes that occurred in the tissue prevented the ETOH from having the toughening and tenderizing effect demonstrated in the previous studies. The lack of difference could have been due to the larger amount of variation in certain parameters evidenced by the standard deviation. This variation was attributed to the response of the tissue to the method used to toughen the breast prior to treatment.

REFERENCES

AOAC. 1984. *Official Methods of Analysis*. Association of Official Analytical Chemists, Washington, DC.

BABJI, A. S., FRONING, G. W. and NGOKA, D. A. 1982. The effect of short-term tumbling and salting on the quality of turkey breast muscle. Poultry Sci. *61*, 300-303.

CHEN, T. C. 1980. Studies on the marinating of chicken parts for deep-fat frying. Poultry Sci. *59*, 1592. (Abstract)

ESSARY, E. O., NORRIS, I. M., SCHULER, G. A. and SINGH, S. P. 1968. Influence of cooking broiler meat in water on tenderness and percentage moisture. Poultry Sci. *47*, 1949-1953.

GOODWIN, T. L., BRAMBLETT, V. D., VAIL, G. E. and STADELMAN, W. J. 1962. Effects of end-point temperature and cooking rate on turkey meat tenderness. Food Tech. *16*, 101-102.

KOTULA, K. L. and HEATH, J. L. 1986. Precook tumbling of hot-boned broiler breasts in sodium chloride and acetic acid. Poultry Sci. *65*, 929-934.

LIND, J. M., GRISWOLD, R. M. and BRAMBLETT, V. D. 1971. Tenderizing effect of wine vinegar marinade on beef round. J. Am. Diet. Assoc. *58*, 133-136.

POST, R. C. 1981. Marination of chicken: Microbiological, chemical and organoleptic evaluation. M.S. Thesis. University of Maryland, College Park.

SMITH, M. C. JR., JUDGE, M. D. and STADELMAN, W. J. 1969. A "cold shortening" effect on avian muscle. J. Food Sci. *34*, 42-46.

SOKAL, R. A. and ROHLF, F. J. 1972. *Introduction to Biostatistics*. W. J. Freeman and Co., San Francisco, CA.

STEEL, R. G. D. and TORRIE, J. H. 1960. *Principles and Procedures of Statistics*. McGraw-Hill Co., New York.

WENHAM, L. M. and LOCKER, R. H. 1976. The effect of marinading on beef. J. Sci. Food Agri. *27*, 1079-1084.

WISE, R. G. and STADELMAN, W. J. 1959. Tenderness at various muscle depths associated with poultry processing techniques. Food Tech. *13*, 689-691.

MICROBIOLOGICAL AND SENSORY EVALUATION OF COOKED ROAST BEEF PACKAGED IN A MODIFIED ATMOSPHERE

CAROLYN BRISTOR HINTLIAN and JOSEPH H. HOTCHKISS[1]

Institute of Food Science
Department of Food Science
Cornell University
Ithaca, NY 14853

Accepted for Publication September 29, 1986

ABSTRACT

Sliced cooked roast beef was packaged in a modified atmosphere (MA) consisting of 75% CO_2, 15% N_2 and 10% 0_2 and stored at 4.4°C for 42 days to evaluate the MA's effectiveness at controlling the outgrowth of Pseudomonas fragi, Clostridium perfringens, Staphylococcus aureus *and* Salmonella typhimurium *after inoculation. Additionally, a taste panel was conducted to monitor the effects of modified atmosphere packaging (MAP) on sensory parameters of uninoculated samples.* P. fragi *was able to grow only to a limited extent in the MA, but grew to high numbers in air. The three pathogens were unable to grow in the MA, probably due to the effects of temperature. Numbers in* S. typhimurium *and C. perfringens declined more rapidly in the MA than in air but* S. aureus *numbers did not decrease in either atmosphere. High numbers of molds were recovered from the uninoculated samples stored in air but none were recovered from MAP samples. Sensory analysis indicated that most product deterioration occurred within the first 7 days of storage.*

INTRODUCTION

Modified atmosphere packaging (MAP) is a technology in which foods are packaged in high barrier packages in which air has been replaced with an ar-

[1]corresponding author

Journal of Food Processing and Preservation 11 (1987) 171-179. All Rights Reserved.

tificial (modified) atmosphere. MAP is currently employed commercially to extend the shelf-life of fresh meat and poultry. MAP could also be used for the development of perishable prepared food products for restaurant and institutional uses.

Inhibitory effects of CO_2 on common spoilage organisms such as *Pseudomonas* have been demonstrated (King and Nagel 1967; Eklund and Jarmund 1983; Enfors and Molin 1980) and it is well known that the use of modified atmospheres (MA's) containing elevated concentrations of CO_2 can significantly extend the microbiological shelf-life of refrigerated products. There has been less research on the effects of MA's on pathogens, particularly in cooked or processed products.

Concern over the micobiological safety of foods packaged in MA's is a major reason for the failure to take greater advantage of the benefits of MAP. Although some researchers have advocated the inclusion of O_2 in MA's to help maintain the bright red color of fresh meats (Clark and Lentz 1973), many MA's are anaerobic. CO_2 does not appear to have an inhibitory effect on anaerobic pathogens such as *Clostridum* and in an anaerobic MA, these organisms could multiply and produce toxin. However, these organisms should become significant only in instances of temperature abuse because *Clostridum perfringens* and most strains of *Clostridium botulinum* do not grow at refrigeration temperatures. Because certain aquatic type E *C. botulinum* strains can grow as low as 3.3 °C (Eklund 1982) MAP for fresh fish is not recommended (Post *et al.* 1985).

Fully-cooked or partially-processed products might be consumed without further heat treatment which is a final safeguard against exposure to low levels of contaminating pathogens. Because the absence of pathogenic microorganisms on non-sterile products cannot be guaranteed, the packaging system employed must not enhance their growth.

Warmed-over flavor (WOF) is the undesirable rancid odor/flavor which develops in cooked meats that are held for relatively short periods after cooking and which becomes more prominent when meat is refrigerated and then reheated (Sato and Herring 1973). WOF is caused by autooxidation and its secondary reactions. Cooked meat products which are stored in MA's for long periods of time and then reheated before consumption are likely to develop WOF if O_2 is present in the MA.

Our previous studies (Hintlian and Hotchkiss 1987) compared the growth of *P. fragi* and the three pathogens (*C. perfringens, S. typhimurium* and *S. aureus*) in several MA's under conditions of temperature abuse. A MA containing 75% CO_2, 15% N_2 and 10% O_2 was identified as the most effective.

Although the three pathogens do not generally grow at refrigeration temperatures, it was necessary to demonstrate that they do not grow under conditions of the MA at refrigeration temperatures. The objectives of this research were to compare the effectiveness of this optimum MA to air at refrigeration temperature (4.4 °C) for the shelf-life extension of both uninoculated and inoculated

sliced, cooked roast beef. A sensory evaluation was also conducted to determine the effects of MAP on product sensory characteristics.

MATERIALS AND METHODS

The methods used in this study have been described in detail in a previous report (Hintlian and Hotchkiss 1987).

Culture Maintenance and Sample Inoculation

Culture maintenance and inoculum preparation of *C. perfringens* (ATCC -3624), *P. fragi* (ATCC -27363), *S. typhimurium* (ATCC -13311) and *S. aureus* (ATCC -9996) has been previously described (Hintlian and Hotchkiss 1987).

Enumeration of Organisms

Dehydrated media were manufactured by Difco except where indicated. Samples were plated by adding sterile diluent to the glass sample jars and replacing the lid with a sanitized (iodophor solution) food blender blade apparatus (Hamilton Beach Model 600-2). The diluted sample was blended at high speed for one minute and serially diluted and inoculated onto various media as indicated.

Plating for aerobes and anaerobes was nonselective. Aerobes were enumerated on plate count agar (PCA) by the spread plate method and incubated at 23 °C for four days; anaerobes were enumerated on pour plates of brain heart infusion agar plus 5% defibrinated sheep blood (BBL) (Holdeman *et al.* 1977) and incubated anaerobically (BBL Anaerobic Jar) for 48 h at 37 °C. Mold was enumerated on spread plates of PCA plus 10 milligrams each of chlortetracycline and chloramphenicol per 100mL of medium (Speck 1984). Incubation was at 23 °C for 72 h.

C. perfringens was enumerated on pour plates of tryptose sulfite cycloserine agar (Speck 1984). *P. fragi* was enumerated by surface plating on *Pseudomonas* agar base plus a supplement containing centrimide, fucidin, and cephaloridine (Oxoid). Desoxycholate citrate agar was used for the enumeration of *S. typhimurium*. *S. aureus* was selectively enumerated on Baird Parker Agar plus Bacto egg yolk tellurite enrichment.

Sample Preparation

Top round beef roasts were obtained from a local grocery on the day they were to be cooked. They were cooked in a conventional oven at 163 °C to an internal temperature of 54 °C. Cooked roasts were cooled overnight at 4.4 °C. The beef was sliced 4 mm thick on a sanitized meat slicer. Using sterile scalpels and

tweezers, excess fat was trimmed and 50.0 ± 0.3 g samples were put into the sterile jars.

A commercially prepared gravy of 25 mL was added to samples for the inoculation and sensory evaluation studies. The gravy product contained no antimicrobial or antimycotic ingredients and was packaged in a glass jar. No viable organisms could be isolated from the gravy.

Packaging System

Prepared samples were packaged in sterilized glass one-pint jars, the lids of which were fitted with rubber septa.

The desired gas mixture was attained by blending CO_2 and N_2 using a Scott Flowmeter (2-31B Series Gas Blender). Oxygen was added to the jars by removing a volume of the N_2/CO_2 mixture with a gastight syringe and replacing with O_2. Jars containing samples to be stored in air were sealed tightly and not flushed.

Inoculated Samples

The inoculum containing *C. perfringens, P. fragi, S. typhimurium* and *S. aureus* was added to the gravy in a sterile container and blended thoroughly before the addition of the gravy to the beef samples in the jars. Jars were flushed with the MA or sealed tightly (air controls) and stored at 4.4 °C. One sample for each atmosphere was plated onto the selective media on Days 0, 7, 14, 21, 28, 35, and 42.

Uninoculated Samples

Uninoculated roast beef was stored at 4.4 ° in the MA or air. One sample from each atmosphere was plated on Days 0, 7, 14, 21, 28, 35, and 42 for aerobes, anaerobes and yeasts and molds.

Sensory Analysis

Experimental samples of uninoculated beef with gravy were packaged in the MA and stored at 4.4 °C. Control samples were cooked and weighed into jars and gravy was added each week in the same manner as for the experimental roasts. Taste panels were conducted on Days, 7, 14, 21, 28, 35 and 42. Twenty panelists each tasted one MAP and one freshly prepared control. Most panelists were unaware that the samples were packaged in MA's or that they represented a shelf-life study. Each panelist was presented with the contents of one control and one experimental sample jar, each of which had been placed on a glass plate, covered with plastic food wrap and microwaved at high power for one minute. The sample tasting order was directed by the response form so that each week, half of the panelists tasted the control and half tasted the experimental sample first.

Panelists were asked to evaluate each sample on hedonic scales of one to seven on flavor, texture, appearance and overall acceptability.

RESULTS AND DISCUSSION

Inoculated Samples

Figure 1 shows the development of the co-inoculated organisms at 4.4 °C in air and the MA. In the MA *Pseudomonas*numbers increased by less than one log and counts of the three pathogens did not increase. *S. typhimurium* and *C. perfringens* gradually decreased to zero recovery in the MA samples while numbers in the air samples decreased less. *S. aureus* counts remained unchanged in both atmospheres. *P. fragi* numbers increased by about seven logs in air controls. Clearly the inhibition of the psychrotroph *P. fragi* is attributable to the MA rather than the low temperature. It is not surprising that pathogen numbers did not increase, since the minimum growth temperature of *S. aureus* and *S. typhimurium* is 5-10 °C and of *C. perfringens* 15-20 °C (Banwart 1981). However, it is interesting to note that the MA shortened the period of time before zero recovery of *S. typhimurium* and *C. perfringens*. This clearly demonstrates the ability of the MA to control spoilage and that the MA did not enhance the growth of pathogenic organisms at refrigeration temperatures. The ability of these pathogens to grow on a temperature-abused product has been addressed (Hintlian and Hotchkiss 1987).

Uninoculated Samples

No anaerobes were recovered in MA or control samples throughout the study. Counts of molds recovered from uninolated samples stored in air reached 10^6 at 42 days (Fig. 2). No molds were recovered from the MA-stored samples. This supports observations made by Brown (1922), Moran *et al.* (1932) and Tompkins (1932) that high levels of CO_2 inhibits the growth of food spoilage molds. Counts of aerobes were essentially the same as counts of molds (data not shown) and all colonies recovered on PCA plates for the aeroabic plate count were molds. Molds were recovered from uninoculated samples but not from those which were inoculated because they are poor competitors against bacteria. The molds which grew may have resulted from the germination of spores which contaminated the cooked product during handling after roasting. Analyses showed the molds counts to be too low to be enumerated until 14 days of storage.

Sensory Analysis

Taste panelists noted more variability in the quality of the freshly prepared control roasts than in the stored samples (Table 1). On Day 7, on a seven point hedonic

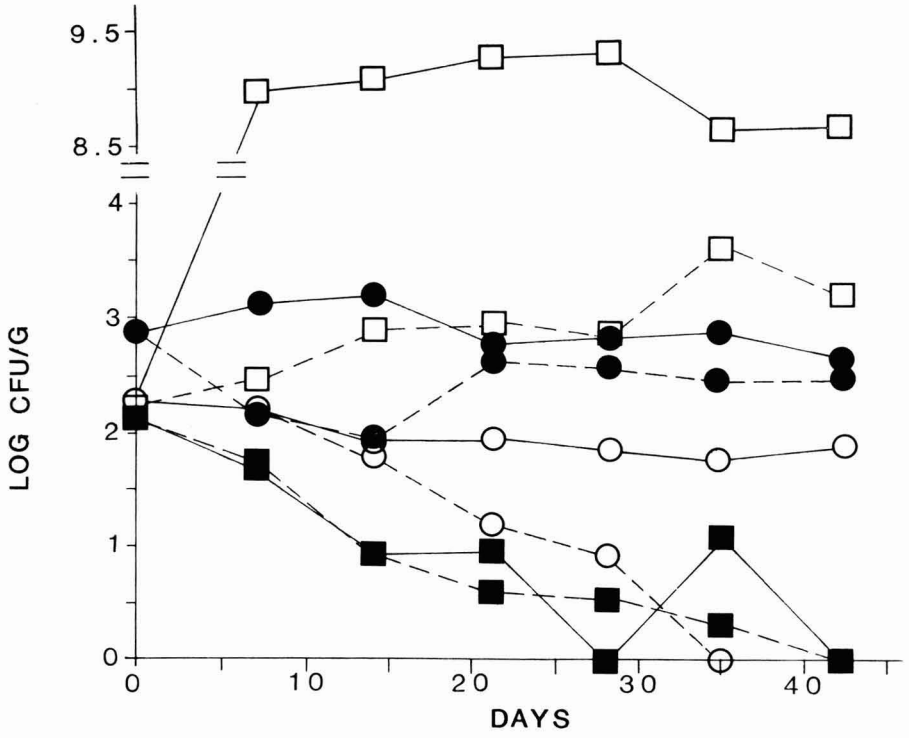

FIG. 1. THE DEVELOPMENT OF *C. PERFRINGENS* (O), *P. FRAGI* (□), *S. TYPHIMURIUM* (■) AND *S. AUREUS* (●) ON ROAST BEEF WITH GRAVY STORED AT 4.4°C IN AIR (—) OR THE MA (-----)

scale, panelists rated the stored samples an average of two points lower for flavor, texture and overall acceptability than the control and rated appearance about one point lower. The scores assigned to each of the two products, MAP and control, decreased very little over time. Scores for the fresh control should have a decreasing trend only as a result of panelist fatigue. It was expected that panelists would assign the stored samples decreasing scores with time due to sample deterioration. The data indicated, however, that the product deterioration occurred within the first seven days of storage. Average scores for all four parameters of the MAP samples were not significantly (Student's t, $p \leqslant 0.01$) different at weeks one and six. MAP cooked beef was clearly inferior to fresh roast beef.

The flavor deterioration likely results from warmed over flavor (WOF) rather than microbiological changes. Panelists sometimes commented on a slightly oxidized flavor, but never of acid or carbon dioxide flavors. Since WOF is the result of an oxidation process, it is probable that the deterioration occurred shortly after packaging.

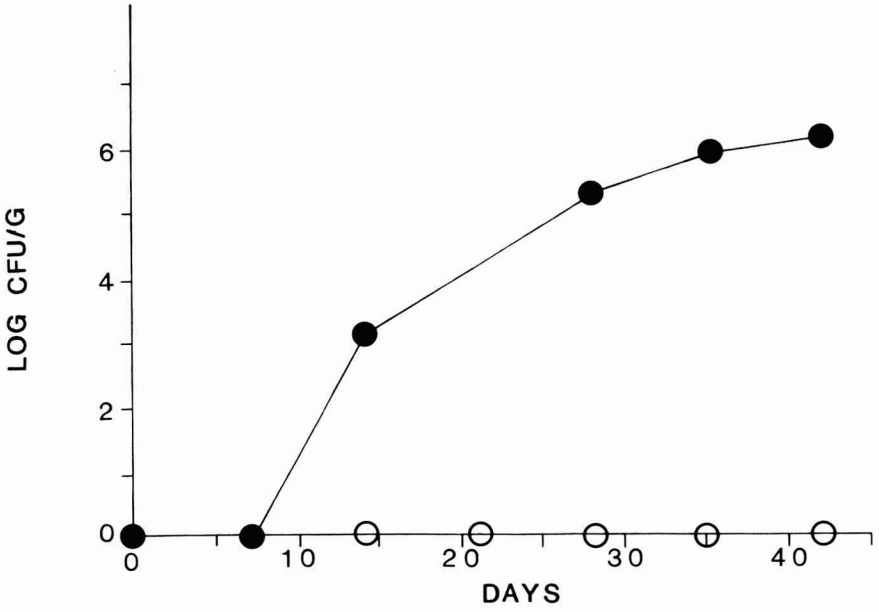

FIG. 2. THE DEVELOPMENT OF MOLDS ON UNINOCULATED COOKED ROAST BEEF STORED AT 4.4°C IN AIR (●) OR THE MA (O)

Shelf-life studies should optimally compare a stored product to one from the same batch which was stored under conditions which will maintain the initial quality (Sensory Evaluation Division, 1981). This is not possible for a product such as roast beef. It is possible that if frozen samples had been used as controls, the MAP samples would have rated more favorably relative to the controls. This may have been a better comparison because this technology, if applied commercially, would probably allow the replacement of certain frozen products by refrigerated products.

MAP using an atmosphere of 75% CO_2, 15% N_2 and 10% 0_2 was shown capable of extending the refrigerated shelf-life of cooked beef to at least 42 days and probably longer. Sensory analysis indicated that WOF developed early in the storage period but additional flavor deterioration did not occur. This method of preservation could be useful if the WOF question were addressed.

ACKNOWLEDGMENTS

The suggestions and discussion of R. B. Gravani and the technical assistance of A. J. Vecchio are gratefully acknowledged.

TABLE 1
AVERAGE SCORES ASSIGNED BY TASTE PANELISTS FOR FLAVOR, TEXTURE, APPEARANCE AND OVERALL ACCEPTABILITY TO COOKED ROAST BEEF[a]

Week	Sample	Parameters			
		Flavor	Texture	Appearance	Overall Acc.
1	Fresh	5.70	5.60	5.50	5.95
	MAP	3.70	3.25	4.50	3.85
2	Fresh	5.80	6.80	5.75	6.00
	MAP	3.85	3.45	4.55	3.70
3	Fresh	5.00	4.06	4.75	4.86
	MAP	3.83	3.95	5.39	3.95
4	Fresh	6.00	5.80	5.75	6.03
	MAP	3.50	3.35	4.15	3.35
5	Fresh	5.50	5.65	5.65	5.78
	MAP	3.65	3.75	4.08	3.73
6	Fresh	5.33	4.95	4.94	5.17
	MAP	3.67	3.53	4.50	3.56

[a]all fresh and MAP samples were significantly (Student's t, $p \leq 0.05$) different except for texture, appearance and overall acceptability on week 3 and appearance on week 6.

REFERENCES

BANWART, G. J. 1981. *Basic Food Microbiology,* AVI, Westport, Conn.

BROWN, W. 1922. On the germination and growth of fungi at various temperatures and in various concentrations of oxygen and carbon dioxide. Ann. Bot. *36*, 257-283.

CLARK, D. S. and LENTZ, C. P. 1973. Use of mixtures of carbon dioxide and oxygen for extending shelf-life of prepackaged fresh beef. Can. Inst. Food Sci. Technol. J. *6*, 194-196.

EKLUND, M. W. 1982. Significance of *Clostridium botulinum* in fishery products preserved short of sterilization. Food Technol. *36*, 107-112.

EKLUND, T. and JARMUND, T. 1983. Microculture model studies on the effect of various gas atmospheres on microbial growth at different temperatures. J. Appl. Bacteriol. *55*, 119-125.

ENFORS, S. O. and MOLIN, G. 1980. Effect of high concentrations of carbon dioxide on growth rate of *Pseudomonas fragi, Bacillus cereus,* and *Streptococcus cremoris.* J. Appl. Bacteriol. *48*, 409-416.

HINTLIAN, C. B. and HOTCHKISS, J. H. 1987. Comparative growth of spoilage and pathogenic organisms in modified atmosphere packaged cooked beef. J. Food Protect. *50*, 218-223.

HOLDEMAN, L. V., CATO, E. and MOORE, W. E. C., eds. 1977. *Anaerobe Laboratory Manual,* 4th ed., VPI Anaerobe Lab, Blacksburg, Virginia.

KING, A. D. JR. and NAGEL, C. W. 1967. Growth inhibition of a *Pseudomonas* by carbon dioxide. J. Food Sci. *32*, 575-579.

KING, A. D. JR. and NAGEL, C. W. 1975. Influence of carbon dioxide upon the metabolism of *Pseudomonas aeruginosa*. J. Food Sci. *40*, 362-366.

MORAN, R., SMITH, E. C. and TOMKINS, R. G. 1932. The inhibition of mould growth on meat by carbon dioxide. J. Soc. Chem. Ind. *5l*, 114T-116T.

POST, L. S., LEE, D., SOLBERG, M., FURGANG, D., SPECCHIO, J. and GRAHAM, C. 1985. Development of botulinal toxin and sensory deterioration during storage of vacuum and modified atmosphere packaged fish fillets. J. Food Sci. *50*, 990-996.

SATO, K. and HERRING, H. 1973. Chemistry of warmed-over flavor in cooked meats. Food Prod. Dev. *7*, 78-84.

Sensory Evaluation Division of the Institute of Food Technologists. 1981. Sensory evaluation guide for testing food and beverage products. Food Tech. *35*, 49-59.

SPECK, M. L. (ed.) 1984. Compendium of Methods for the Microbiological Examination of Foods. American Public Health Association, Washington, D.C.

TOMKINS, R. G. 1932. The inhibition of the growth of meat attacking fungi by carbon dioxide. J. Soc. Chem. Ind. *51*, 261T-264T.

BOOK REVIEW

Enzymic Hydrolysis of Food Proteins, Jens Adler-Nissen, Elsevier Applied Science Publishers, 427 pp., $87.50.

The intent of this book is to develop a systematic approach to efficiently design and optimize enzyme food protein hydrolysis. The justification for such a book is founded on the surging interest in biotechnology. Many of the examples used to illustrate principles of evaluating and modeling enzymic protein hydrolysis have come from work in the author's laboratory while at Novo Industri A/S of Denmark. Thus, the tone of the book reflects the author's philosophy and considerable experience in this field. However, much information is also taken from the work of others, providing the necessary balance in discussion of important concepts. The author has wisely delimited the extent of the material incorporated into the text. Protein hydrolysis is discussed within limits of that being affected by enzymes alone, and to the exclusion of that brought about by microorganisms or fermentations. The second delimitation imposed by the author is that the book addresses the use of hydrolysates for food use only, focusing especially on the technical aspects of the bitter, functional and nutritional qualities of protein hydrolysates. In addition, the author confines the discussion of food protein hydrolysis to those circumstances where enhanced utilization of a specific protein is desired, while excluding cases where the primary intent is to modify the properties of a food system as in cheesemaking or baking.

The text is composed of eleven chapters. Following an introduction, three chapters are devoted to the fundamentals of enzymic protein hydrolysis and reviews of general and specific areas of food protein hydrolysis. Together, these chapters provide a brief background on the dynamics of enzymic protein hydrolysis and the analytical methods often used for following its course. The last of these three chapters is devoted to the current state of knowledge and technical limitations of enzymic hydrolysis of proteins from specific food sources. Chapter five presents some selected analytic and ancillary procedures used for evaluating protein hydrolysis and its end-products. Sections of this chapter are presented in the format of a laboratory manual. Chapter six covers continuous protein hydrolysis and means for monitoring this process. This includes calibration methods for pH-stats and osmometers, characterization of relative reactivity of proteases and hydrolysis curves, and a discussion on the interrelationships between degree of hydrolysis, peptide chain length distribution and end-product solubility as influenced by reactor pH. The object here is to compile data gathered empirically and try to formulate a logical approach for the optimization of a protein hydrolysis reaction at the industrial level. Chapter seven discusses reaction kinetics for zipper and one-by-one mechanisms in relation to the nature of hydrolysis curves where substrate competition and product inhibition are possible. The following two chapters present a statistical method, developed previously by the author, to quantitatively monitor the course of enzymic protein hydrolysis. The method is presented in depth, complete with the underlying theories and experimental support of the method as illustrated by standard hydrolysis curves. These chapters constitute the cornerstone of the text as it presents the author's putative approach to the optimization of protein hydrolysis. The tenth chapter is devoted to the production, properties and functionality of soy protein hydrolysates. The concluding chapter encapsulates the author's suggestions for modeling and optimizing protein hydrolysis.

Journal of Food Processing and Preservation 11 (1987) 181-182. All Rights Reserved.

In considering the scientific literature that was available to compose this text, it becomes clear that there exists a gap between the fundamental understanding and applied aspects of enzymic protein hydrolysis. Perhaps this is best exemplified by the relative lack of understanding of how to control bitterness in the preparation of protein hydrolysates. However, in spite of this limitation the author is to be commended for his efforts to take existing information and begin to bridge this gap. The book is well written, provides the necessary background information where needed and the concepts are fairly well developed. The content and format of this book are reminiscent of a dissertation and is thus, somewhat specialized and limited in scope. However, this approach was necessary to allow the discussion of subject material in a manner that comes across as being complete and fully developed. Accordingly, this book should be of most utility for those familiar or involved with this field of research. However, the text is also written in a manner that will allow a novice to develop a good understanding of the field of enzymic protein hydrolysis. Portions of the text that are presented in a lab manual format may provide the seed for developing laboratory experiments highlighting some of the principles of protein hydrolysis. In addition, sections of this book may be useful as material for graduate engineering and enzymology courses.

KIRK L. PARKIN

F
N
P

PUBLICATIONS IN FOOD SCIENCE AND NUTRITION

Journals

JOURNAL OF SENSORY STUDIES, M.C. Gacula, Jr.
JOURNAL OF FOOD SERVICE SYSTEMS, O.P. Snyder, Jr.
JOURNAL OF FOOD BIOCHEMISTRY, J.R. Whitaker, N.F. Haard and H. Swaisgood
JOURNAL OF FOOD PROCESS ENGINEERING, D.R. Heldman and R.P. Singh
JOURNAL OF FOOD PROCESSING AND PRESERVATION, D.B. Lund
JOURNAL OF FOOD QUALITY, M.P. De Figueiredo
JOURNAL OF FOOD SAFETY, M. Solberg and J.D. Rosen
JOURNAL OF TEXTURE STUDIES, M.C. Bourne and P. Sherman
JOURNAL OF NUTRITION, GROWTH AND CANCER, G.P. Tryfiates

Books

THE SCIENCE OF MEAT AND MEAT PRODUCTS, 3RD ED., J.F. Price and B.S. Schweigert
HANDBOOK OF FOOD COLORANT PATENTS, F.J. Francis
ROLE OF CHEMISTRY IN THE QUALITY OF PROCESSED FOODS, O.R. Fennema, W.H. Chang and C.Y. Lii
NEW DIRECTIONS FOR PRODUCT TESTING AND SENSORY ANALYSIS OF FOODS, H.R. Moskowitz
PRODUCT TESTING AND SENSORY EVALUATION OF FOODS, H.R. Moskowitz
ENVIRONMENTAL ASPECTS OF CANCER: ROLE OF MACRO AND MICRO COMPONENTS OF FOODS, E.L. Wynder *et al.*
FOOD PRODUCT DEVELOPMENT IN IMPLEMENTING DIETARY GUIDELINES, G.E. Livingston, R.J. Moshy, and C.M. Chang
SHELF-LIFE DATING OF FOODS, T.P. Labuza
RECENT ADVANCES IN OBESITY RESEARCH, VOL. IV, J. Hirsch *et al.*
RECENT ADVANCES IN OBESITY RESEARCH, VOL. III, P. Bjorntorp *et al.*
RECENT ADVANCES IN OBESITY RESEARCH, VOL. II, G.A. Bray
RECENT ADVANCES IN OBESITY RESEARCH, VOL. I, A.N. Howard
ANTINUTRIENTS AND NATURAL TOXICANTS IN FOOD, R.L. Ory
UTILIZATION OF PROTEIN RESOURCES, D.W. Stanley *et al.*
FOOD INDUSTRY ENERGY ALTERNATIVES, R.P. Ouellette *et al.*
VITAMIN B_6: METABOLISM AND ROLE IN GROWTH, G.P. Tryfiates
HUMAN NUTRITION, 3RD ED., F.R. Mottram
FOOD POISONING AND FOOD HYGIENE, 4TH ED., B.C. Hobbs *et al.*
POSTHARVEST BIOLOGY AND BIOTECHNOLOGY, H.O. Hultin and M. Milner

Newsletters

FOOD INDUSTRY REPORT, G.C. Melson
FOOD, NUTRITION AND HEALTH, P.A. Lachance and M.C. Fisher
FOOD PACKAGING AND LABELING, S. Sacharow

GUIDE FOR AUTHORS

Typewritten manuscripts in triplicate should be submitted to the editorial office. The typing should be double-spaced throughout with one-inch margins on all sides.

Page one should contain: the title, which should be concise and informative; the complete name(s) of the author(s); affiliation of the author(s); a running title of 40 characters or less; and the name and mail address to whom correspondence should be sent.

Page two should contain an abstract of not more than 150 words. This abstract should be intelligible by itself.

The main text should begin on page three and will ordinarily have the following arrangement:

Introduction: This should be brief and state the reason for the work in relation to the field. It should indicate what new contribution is made by the work described.

Materials and Methods: Enough information should be provided to allow other investigators to repeat the work. Avoid repeating the details of procedures which have already been published elsewhere.

Results: The results should be presented as concisely as possible. Do not use tables *and* figures for presentation of the same data.

Discussion: The discussion section should be used for the interpretation of results. The results should not be repeated.

In some cases it might be desirable to combine results and discussion sections.

References: References should be given in the text by the surname of the authors and the year. *Et al.* should be used in the text when there are more than two authors. All authors should be given in the References section. In the Reference section the references should be listed alphabetically. See below for style to be used.

DEWALD, B., DULANEY, J. T. and TOUSTER, O. 1974. Solubilization and polyacrylamide gel electrophoresis of membrane enzymes with detergents. In *Methods in Enzymology*, Vol. xxxii, (S. Fleischer and L. Packer, eds.) pp. 82–91, Academic Press, New York.

HASSON, E. P. and LATIES, G. G. 1976. Separation and characterization of potato lipid acylhydrolases. Plant Physiol. *57*, 142–147.

ZABORSKY, O. 1973. *Immobilized Enzymes*, pp. 28–46, CRC Press, Cleveland, Ohio.

Journal abbreviations should follow those used in *Chemical Abstracts*. Responsibility for the accuracy of citations rests entirely with the author(s). References to papers in press should indicate the name of the journal and should only be used for papers that have been accepted for publication. Submitted papers should be referred to by such terms as "unpublished observations" or "private communication." However, these last should be used only when absolutely necessary.

Tables should be numbered consecutively with Arabic numerals. The title of the table should appear as below:

Table 1. Activity of potato acyl-hydrolases on neutral lipids, galactolipids, and phospholipids

Description of experimental work or explanation of symbols should go below the table proper. Type tables neatly and correctly as tables are considered art and are not typeset.

Figures should be listed in order in the text using Arabic numbers. Figure legends should be typed on a separate page. Figures and tables should be intelligible without reference to the text. Authors should indicate where the tables and figures should be placed in the text. Photographs must be supplied as glossy black and white prints. Line diagrams should be drawn with black waterproof ink on white paper or board. The lettering should be of such a size that it is easily legible after reduction. Each diagram and photograph should be clearly labeled on the reverse side with the name(s) of author(s), and title of paper. When not obvious, each photograph and diagram should be labeled on the back to show the top of the photograph or diagram.

Acknowledgments: Acknowledgments should be listed on a separate page.

Short notes will be published where the information is deemed sufficiently important to warrant rapid publication. The format for short papers may be similar to that for regular papers but more concisely written. Short notes may be of a less general nature and written principally for specialists in the particular area with which the manuscript is dealing. Manuscripts which do not meet the requirement of importance and necessity for rapid publication will, after notification of the author(s), be treated as regular papers. Regular papers may be very short.

Standard nomenclature as used in the scientific literature should be followed. Avoid laboratory jargon. If abbreviations or trade names are used, define the material or compound the first time that it is mentioned.

EDITORIAL OFFICE: Dr. D. B. Lund, Editor, Journal of Food Processing and Preservation, University of Wisconsin, Department of Food Science, 1605 Linden Drive, Madison, Wisconsin

JOURNAL OF FOOD PROCESSING AND PRESERVATION VOL. 11, NO. 2

CONTENTS