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JOURNAL

OF

FOOD

PROCESSING

AND

PRESERVATION

D.B. LUND  
EDITOR

FOOD & NUTRITION  
PRESS, INC.

VOLUME 11, NUMBER 4

DECEMBER 1987

## JOURNAL OF FOOD PROCESSING AND PRESERVATION

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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.

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**Journal of  
FOOD PROCESSING  
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**VOLUME 11  
NUMBER 4**

**Editor: D. B. LUND**

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

11 4 2531

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ISSN: 0145-8892

Printed in the United States of America

## EDITORIAL

In 1986 we successfully introduced two new sections to the *Journal of Food Processing and Preservation*: (1) Computer Codes and Their Applications and (2) Data Bank. For both additions, we have received very favorable comments. I would like to encourage our readership to submit papers appropriate for these special sections.

I would like to thank the Editorial Board for their effort on behalf of the *Journal of Food Processing and Preservation* this past year. The Editorial Board members are appropriately listed on the inside cover of this issue. In addition, I would like to thank all of those who served as reviewers (listed below) for 1987. Their input is absolutely essential to make the *Journal of Food Processing and Preservation* a quality journal.

Finally, I would like to acknowledge the authors whose papers have been published in this year's volume. I would encourage them to continue to consider the *Journal of Food Processing and Preservation* for their papers. In addition, we are continually seeking high quality papers for publication. Since we have a "no page charge" policy, I would encourage you to consider the *Journal of Food Processing and Preservation* for processing related papers.

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## CONTENTS

Editorial .....	v
Engineering and Quality Aspects of Particle-To-Particle Heat Transfer to Black Beans <b>J.M. AGUILERA, J. ROMAN and A. HOHLBERG</b> .....	263
Softening of Fish Bone. I. Relation Between Softening Rate and Solubilization Rate of Organic Matter from Fish Bone <b>M. ISHIKAWA, S. MORI, H. WATANABE and Y. SAKAI</b> .....	277
Yield and Tenderness of Cured Chicken Roll Products as Affected by Water Addition <b>S.W. WANG and T.C. CHEN</b> .....	289
Composition and Physico-Chemical Properties of Yam ( <i>Dioscorea</i> Species) Flour Prepared Using Different Processes <b>A. KAMENAN, L.R. BEUCHAT, M.S. CHINNAN and E.K. HEATON</b> .....	299
Correlation of Time-Temperature Indicator Response with Microbial Growth in Pasteurized Milk <b>R. GRISIUS, J.H. WELLS, E.L. BARRETT and R.P. SINGH</b> ...	309
Effects of Phosphate Type, Packaging Method and Storage Time on the Characteristics of Chinese Sausage <b>J.C. KUO, S.Y. WANG, C.J. HUANG and H.W. OCKERMAN</b> ...	325
Computer Codes and Their Application Development of a Sampling Plan for Specified Risks by Microcomputers <b>G. PAPPAS and V.N.M. RAO</b> .....	339
Book Review .....	347
Author Index .....	349
Subject Index .....	353

# ENGINEERING AND QUALITY ASPECTS OF PARTICLE-TO-PARTICLE HEAT TRANSFER TO BLACK BEANS

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Accepted for Publication April 10, 1987

## ABSTRACT

*A hot sand roaster was used for drying, desinfestation and inactivation of enzymes in mature and dry black beans. Measured transient temperature inside beans agreed values calculated using a model for heat transfer into an ellipsoid. Overall heat transfer coefficients during the transient period (less than 2 min) ranged from 171 to 281 W/m<sup>2</sup>°C for sand temperature between 100 and 140°C. Moisture removal from dried beans (15–17% moisture content) varied between 1 and 4.5 percentage points and correlated well with the final temperature of the sand. Roasting mature beans at different moisture levels was effective for inactivating peroxidase, enzyme believed to be involved in deteriorative reactions in dry beans. Infestation was also controlled with a mild roasting. A disadvantage of roasting was the structural damage of the outer cotyledon cells as studied with SEM. Particle-to-particle roasting proved to be a simple and effective pretreatment to preserve quality of dry beans during storage.*

## INTRODUCTION

Legumes are major sources of protein in many regions of the world. In developing countries the production of dry beans exceeds 12.5 million metric tons per year, accounting for about 20% of the world's pulse production (FAO 1984).

Recent reviews by Aguilera and Stanley (1985) and Stanley and Aguilera (1985) have addressed the issue of quality losses in beans under adverse storage conditions. Major vectors in losses during storage are insects and a pervasive hardening phenomenon, both highly dependent on the moisture content and storage temperature of the grain.



Molina *et al.* (1976) suggested that stored beans subjected to a wet heat pretreatment hardened at a slower rate. This was in accordance with some theories that postulate an enzymatic origin for the hardening phenomenon. Although the most utilized method for checking insect infestation is chemical, irradiation and heat have also good potential (Nakayama *et al.* 1983; Tilton and Brower 1985; Aguilera and Steinsanir 1985). Thus, it appears that controlled heating of beans may have an effect on the rate of hardening, enzymatic reactions and insect activity.

Particle-to-particle heat transfer seems to have several advantages as a fast, dry heating mechanism for solid, particulate foods. Some of the applications already proposed are in roasting, drying, blanching and desinfestation of grains (Richard and Raghavan 1980; Aguilera *et al.* 1982; Mittal *et al.* 1983).

The objectives of the present study were to determine some engineering parameters important in particle-to-particle heat transfer to black beans in a hot sand roaster, and assess its application in drying, enzyme inactivation and desinfestation.

## MATERIAL AND METHODS

### Materials

Mature and field dried black beans (*Phaseolus vulgaris*) were of the 1985 harvest from the Agricultural Experimental Station, University of Chile (Santiago, Chile). The heating medium was sand particles with diameters between 0.42 and 0.21 mm.

### Equipment

Two types of roasters were used in the experiments. A laboratory scale roaster consisted of a rotating cylinder (20 cm long  $\times$  14 cm diameter) made of 3 mm steel with an external 3 cm polyurethane insulation (Fig. 1). Rotation speed was controlled with a variable speed motor. Thermocouples were introduced into the cylinder through a hollow shaft and connected with a WB-31 interface (Omega Engineering, Inc., Stamford, CT) to an Apple IIe microcomputer for instant data recording. A weighed amount of sand was heated with gas burners in a large tin tray and introduced into the cylinder, where the temperature was allowed to stabilize before introducing the beans.

The batch pilot-size roaster consisted of two rotating metallic drums (Fig. 1). The sand was heated in the upper drum by gas burners and discharged into the lower drum where it was mixed with beans at the desired bean-to-sand (B/S) ratio. Thermocouples were also introduced through hollow shafts. After heating beans and sand were poured over a metallic screen (5 mm openings) and separated.

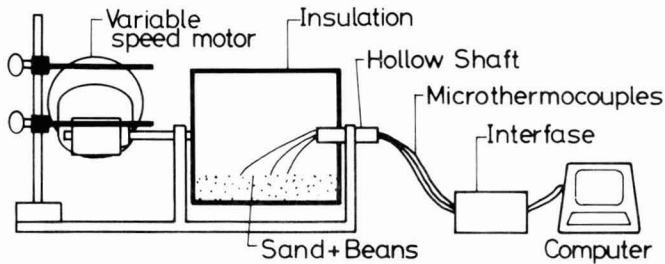
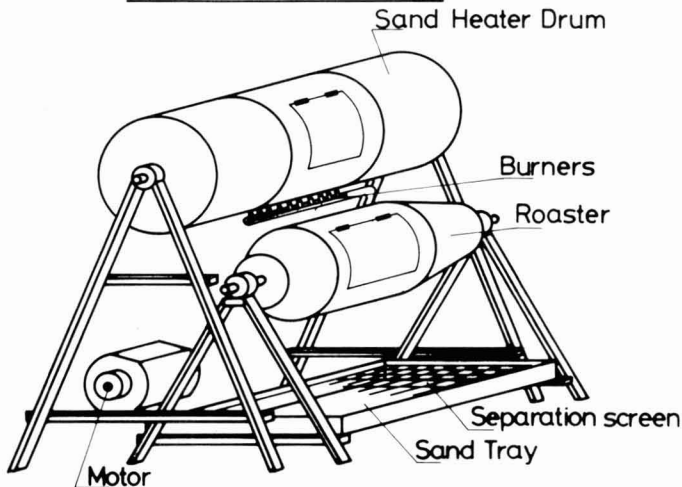
LABORATORY SCALE ROASTERPILOT-SCALE ROASTER

FIG. 1. SCHEMATIC DIAGRAMS OF THE LABORATORY AND PILOT-SIZE ROASTERS

### Experimental Procedure

Experiments carried out in the laboratory roaster were designed to determine the overall heat transfer coefficient, temperature gradients inside the beans, and moisture removal during roasting. A predetermined amount of beans was mixed with hot sand when the desired initial temperature of the medium was reached. A  $2 \times 2 \times 2$  factorial experiment was carried out in the laboratory roaster having as variables rotation speed (10 and 40 rpm), B/S ratio (0.1 and 0.2) and initial sand temperature (100 and 140°C). To achieve good contact, microthermocouples (0.005 in. diameter) were inserted in beans while fresh and soft (50% moisture) and then dried in an oven at 50°C to 17% moisture. The position of the thermocouples inside the seeds was determined afterwards using X-ray photography. Temperatures in three positions inside the beans were recorded every 15 s.

A second factorial experiment was carried-out in the pilot size roaster to determine a relationship between the final temperature of the sand and the % points of water removed from the beans. The experiments were undertaken with initial sand temperatures ( $T_{mi}$ ) of 100, 120 and 140 °C, B/S ratios of 0.15, 0.20 and 0.25, and residence times (RT) of 1, 2 and 3 min.

### Analytical Model

Overall heat transfer coefficients from the medium ( $U$ ) were determined numerically from a heat balance in a single seed. The bean was divided in three concentric ellipsoids with surface area  $A_i$  where the temperature ( $T_i$ ) was measured:

$$U = \frac{m_p c_p}{A} \frac{T(t_0) - T(t_f)}{\int_{t_0}^{t_f} (T_m - T_s) dt}$$

where  $m_p$ : average mass of a bean,  
 $c_p$ : heat capacity of the bean,

$T(t) = \Sigma(T_i A_i d) / \Sigma(A_i d) = \Sigma(T_i A_i) / \Sigma A_i$ : average temperature of the bean at time  $t$ , determined as a weighted average of the temperatures measured by each thermocouple, where:

$A_i$ : area of each section where the temperature was determined.

$d$ : thickness of the thermocouple

The analytical solution for the transient heat transfer to an ellipsoid suddenly submerged in a media with temperature  $T_m$  was used to model under the following assumptions:

- (1) beans were homogeneous solids with axial symmetry,
- (2) heat transfer occurred unidimensionally,
- (3) the effect of moisture loss during roasting was negligible,
- (4) temperature of the media remained homogeneous and constant,
- (5) perfect mixing of particles in the roaster,
- (6) beans were ellipsoids where:

$$r = (a b c)^{1/3} \quad (1)$$

$r$ : radius of an equivalent sphere

$a, b, c$ : dimensions of the main axes in a bean

The general solution for this heat transfer problem is (Grober *et al.* 1961):

$$\frac{T(r,t) - T_m}{T_0 - T_m} = 2 \sum_{n=1}^{\infty} \frac{\sin(\lambda_n R) - \lambda_n R \cos(\lambda_n R)}{\lambda_n R - \sin(\lambda_n R) \cos(\lambda_n R)} e^{-\alpha \lambda_n^2 t} \frac{\sin(\lambda_n r)}{\lambda_n r} \quad (2)$$

where  $\lambda$  was obtained from:

$$\lambda_n R \cos(\lambda_n R) = (1 - UR/K) \sin(\lambda_n R)$$

and:

- $T(r,t)$  = temperature at a distance  $r$  from the center of the bean after a period  $t$ ,
- $T_m$  = media temperature (sand),
- $T_0$  = initial temperature of the beans,
- $R$  = equivalent radius of the bean,
- $\alpha$  = thermal diffusivity of the beans,
- $U$  = overall heat transfer coefficient,
- $K$  = thermal conductivity of the beans.

The boundary conditions for this case were:

- (1)  $\odot(r,0) = T_0 - T_m = \odot_0 = \text{cte}$
- (2)  $\partial \odot(O,t) / \partial r = 0$  (symmetry)
- (3)  $-K \partial \odot(R,t) / \partial r = U(T(R,t) - T_m) = U \odot(R,t)$

### Hardening Studies

Control and beans roasted in the pilot roaster to 80°C were stored in sealed polyethylene bags, inside temperature controlled chambers at 17°C and 32°C over a period of 12 months. Moisture was kept constant at 9 and 12% throughout storage. Hardness (maximum force) of cooked beans was determined periodically in an OTMS shear cell attached to an Instron as described by Aguilera and Ballivian (1986).

### Peroxidase Activity

Another set of experiments with the pilot-size roaster was targeted at enzyme inactivation. Fresh beans were adjusted to initial moisture contents of 52, 30, 24 and 16% to assess the effect of moisture on the effectiveness of heat transfer and

enzyme inactivation. Samples were roasted with sand at 150°C and B/S ratio 1 to 5, and later dried to 10–12% moisture content. Seeds were stored in sealed polyethylene/aluminum bags at 30°C. Every second month peroxidase activity was determined in the samples as described by Sharon-Raber and Kahn (1983).

### Thermal Desinfestation

Heating in the pilot roaster was also tested for desinfection from bean-weevil (*Acanthoscelides obtectus*). Beans were roasted for 2 min in a B/S ratio 1 to 5 with sand at 120°C. Control and roasted beans were stored with moisture contents between 9 a 12% at 30°C/85% RH for 8 months. The number of defective grains (with holes) was determined bimonthly by visual inspection from 200 randomly selected seeds.

## RESULTS AND DISCUSSION

### Laboratory Determination of Heat Transfer Coefficients

Typical temperature-time profiles inside the bean and in the medium are shown in Fig. 2. In all experiments, the temperature of the surrounding medium remained within 5°C of the final temperature after the initial 15 s and reached thermal equilibrium with the beans before 2 min. Temperature differences between the outer portion of the bean ( $T_1$ ) and the center ( $T_3$ ) never exceeded 30°C. Thus, roasting under reported conditions was a rapid and homogeneous mean of heat transfer.

Table 1 summarizes the U values obtained from Eq. 1. The overall heat transfer coefficients varied between 162.9 and 218.5 W/m<sup>2</sup>°C and were in the range of values reviewed by Richard and Raghavan (1980) for sand-to-solid heat transfer. The U coefficient increased with increasing initial temperature of the sand ( $T_{mi}$ ), rotation speed (N) and, except in one case, with lowering the B/S ratio. Optimization of the process was not attempted as it will depend strongly on the roasting system.

Simulation of the transient temperatures within the bean were very close to the real values, in spite of simplifications of the model (Fig. 2). Thermal diffusivity of the beans was assumed to be  $0.8 \times 10^{-7}$  m<sup>2</sup>/s (Sullivan and Sabersky 1975), while the heat transfer coefficients (U) were obtained from Table 1.

### Drying Effect

Reductions in moisture content of beans (expressed as percentage points), as a function of B/S ratio,  $T_{mi}$  and RT are presented in Table 2. A moisture decrease between 1 and 4.5 points was obtained with the prototype roaster. Percentage points of moisture removed from the seeds (MR) correlated well with the final



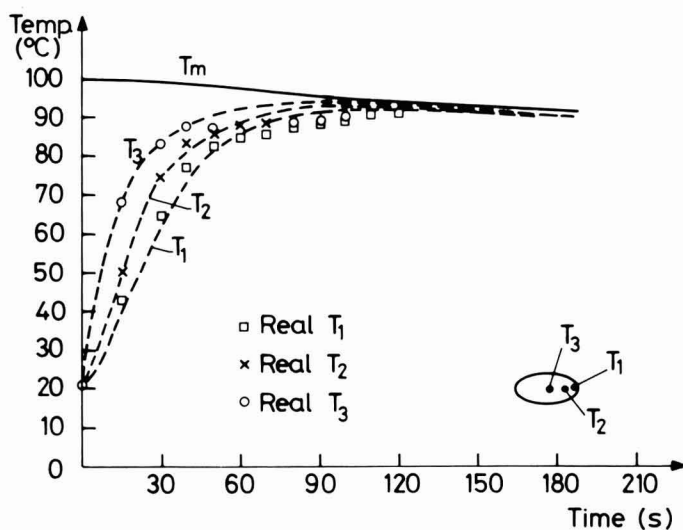


FIG. 2. SAND AND BEAN TRANSIENT TEMPERATURE OBTAINED FROM THE LABORATORY-SIZE ROASTER Dotted lines indicate simulated temperatures inside the bean

TABLE 1.  
OVERALL HEAT COEFFICIENT FOR SAND MEDIA

Exp. #	(B/S) (w/w)	N (rpm)	$T_{mi}$ (°C)	$T_{mf}$ (°C)	U (W/m <sup>2</sup> °C)
1	0.1	10	100	87	171.2
2	0.1	10	140	119	217.6
3	0.1	40	100	88	262.8
4	0.1	40	140	120	281.5
5	0.2	10	100	80	162.9
6	0.2	10	140	109	238.7
7	0.2	40	100	82	182.3
8	0.2	40	140	116	204.5

(B/S): Bean-to-sand ratio in the roaster

N: Roaster rotation speed

$T_{mi}$ : Initial temperature of the media (sand)

$T_{mf}$ : Final temperature of the media (sand)

U: Overall heat transfer coefficient

temperature of the sand ( $MR = 0.0241 T_{mf} - 0.61$ ;  $r=0.84$ ,  $n=27$ )(Fig. 3). This can be explained by the fact that the net amount of energy entering the grain is proportional to the difference between the initial and final temperature of the beans. Since the media and bean reached thermal equilibrium and the initial temperature of the beans was the same, the net energy flow was directly related to the final media temperature. Mittal *et al.* (1983) showed in laboratory experiments that the moisture of wheat could be reduced from 17.0 to 14.5% using hot sand, but, they correlated moisture loss with the initial temperature of the sand.

TABLE 2.  
EFFECT OF ROASTING IN BEAN MOISTURE (N=20 rpm)

Exp. #	B/S (w/w)	RT (min)	$T_{mi}$ (°C)	$T_{mf}$ (°C)	MR (%)	$T_{bf}$ (°C)
1	0.15	3	140	112	4.04	105
2	0.15	3	120	99	2.59	93
3	0.15	3	100	87	2.30	78
4	0.15	2	140	112	3.05	104
5	0.15	2	120	100	3.27	95
6	0.15	2	100	86	2.50	81
7	0.15	1	140	111	2.95	103
8	0.15	1	120	97	1.96	91
9	0.15	1	100	80	1.38	75
10	0.20	3	140	104	2.86	95
11	0.20	3	120	93	1.96	84
12	0.20	3	100	85	1.72	78
13	0.20	2	140	111	4.42	100
14	0.20	2	120	97	3.36	89
15	0.20	2	100	85	2.18	77
16	0.20	1	140	100	2.37	90
17	0.20	1	120	90	1.95	83
18	0.20	1	100	80	1.85	71
19	0.25	3	140	116	3.13	105
20	0.25	3	120	97	2.27	90
21	0.25	3	100	83	1.83	78
22	0.25	2	140	111	3.83	100
23	0.25	2	120	99	2.32	90
24	0.25	2	100	86	1.00	76
25	0.25	1	140	110	2.19	97
26	0.25	1	120	93	1.67	85
27	0.25	1	100	81	1.00	75

RT: Roasting time

MR: Moisture reduction

$T_{bf}$ : Final temperature of beans

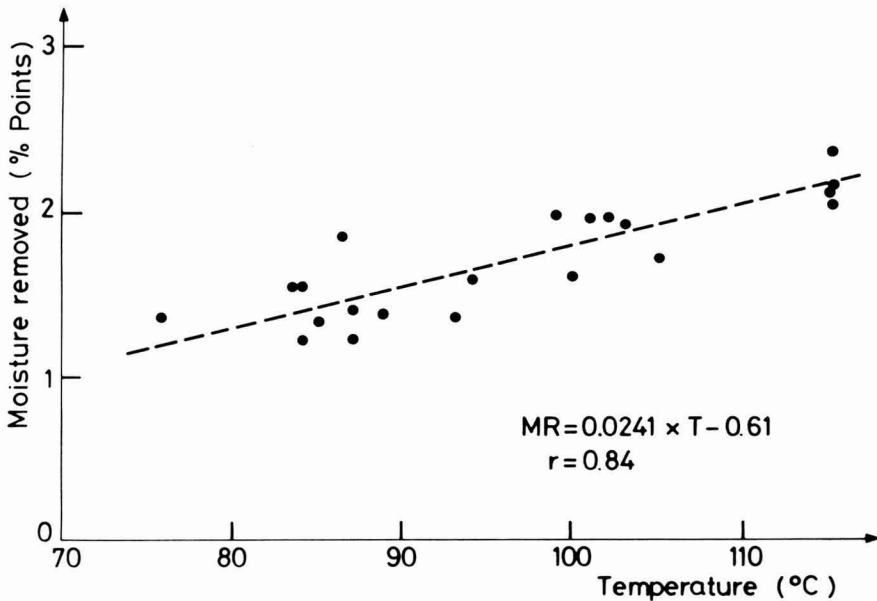


FIG. 3. RELATION BETWEEN FINAL TEMPERATURE OF THE MEDIA AND POINTS OF MOISTURE REMOVED FROM BEANS

At relatively high moisture contents (13%), temperature had an important effect on the rate of hardening (Fig. 4). This was evident at 32 °C where roasted beans, whose moisture content has been reduced from 13 to 9% during roasting, were more than three times softer than control beans (13% moisture) after 12 months of storage. This result is of particular importance in the case of tropical countries where high temperatures and high seed moisture contents are usually found. Thus, roasting may prove to be an adequate method to remove a small but critical amount of water from the seeds.

Roasting induced microstructural damage (Fig. 5), and promoted hardening possibly by increased contact between precursors (Aguilera and Ballivian 1986). However, this effect was more than offset by the reduction in hardening rate at the lower moisture contents achieved through the drying effect (Fig. 4).

### Enzyme Inactivation and Hardening

Enzymes have been reported to contribute in deteriorative reactions in beans during storage. Peroxidase has been related to the formation of lignin in intercellular spaces leading to toughening of the middle lamellae and hardening (Hincks and Stanley 1986). Since peroxidase is also supposed to be the most heat

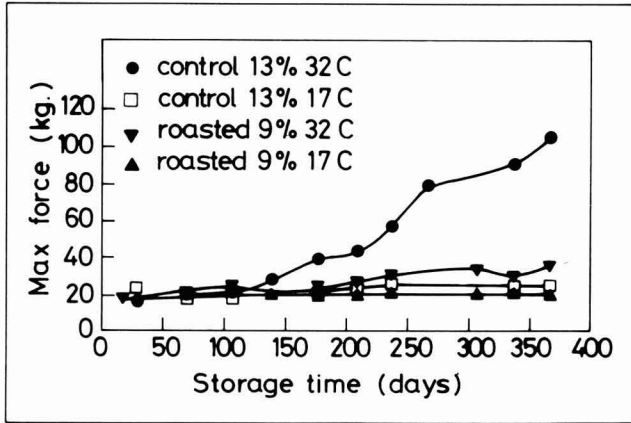


FIG. 4. EFFECT OF ROASTING AND STORAGE TEMPERATURE IN THE HARDENING OF BLACK BEANS

stable enzyme present, its destruction by heat ensures absence of enzyme activity (Schwimmer 1981). Lipoxigenase, phytase and several proteases have also been reported to contribute to quality losses of beans over storage (Stanley and Aguilera 1985).

The effect of different temperature-time contributions and moisture content of the beans on inactivation of peroxidase is shown in Table 3. Higher temperatures were needed to inactivate peroxidase at lower moisture contents. Moisture promotes higher heat transfer rates and increases enzyme liability to heat. Temperature-moisture-time combinations of 102/52/3, 104/30/3 and 120/16/5 were effective in completely inactivating the enzyme. The slight increase in peroxidase activity with roasting at 80/8/2 may be interpreted as a potentiation effect (Schwimmer 1981; Muftugil 1985).

### Thermal Desinfestation

Percentage of infested beans in control and roasted samples stored at different moisture contents is presented in Table 4. Control beans stored at 11 and 13% moisture content showed 15 and 17.5% infested beans after 10 months of storage, while those stored at 9% presented only 1.5% defectives. Less than 1% of the roasted beans were infested after 10 months of storage at 10.5 and 12% moisture content.

The critical effect of moisture on insect development in untreated beans is another reason to reduce the water level of beans during storage. Thermal desinfestation by roasting at 120 °C for 2 min seemed to be an adequate treatment for insect control without appreciable damage to the grain. It is a safe and simple

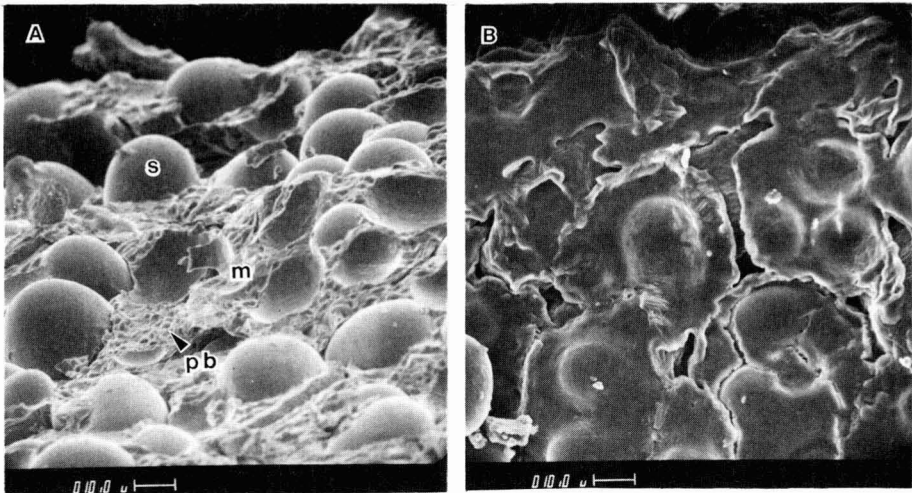


FIG. 5. PHOTOMICROGRAPHS OF CROSS SECTION OF BEAN COTYLEDONS FROM (A) CONTROL BEANS AND (B) ROASTED BEANS s=strach granules, pb=protein bodies, m=matrix

TABLE 3.  
PEROXIDASE ACTIVITIES AFTER ROASTING TREATMENTS

Heating Temp. (°C)	Moisture (%)	Heating Time (min)	Residual Activity (%)
87	52.3	3	3.76
102	52.3	3	< 0.1
104	29.6	3	< 0.1
104	24.5	4	1.49
120	15.6	5	< 0.1
104	15.6	5	10.24
80	8.4	2	102.29
-*	12.0	-	100.0

\*Control



method to be implemented at the village level in less-developed countries. Similar time-temperature combinations during solid heat transfer were shown effective in controlling insects in wheat (Mittal *et al.* 1981). Care should be taken to avoid reinfestation of pasteurized grains during storage or to use them as seeds since the germination rate may be reduced (Aguilera and Ballivian 1986).

TABLE 4.  
PERCENTAGES OF INFESTED BEANS IN ROASTED AND CONTROL SAMPLES

	Average Moisture (%)	STORAGE (months)					
		1	2	4	6	8	10
		(% Defectives)					
Control	9.0	1.0	0.0	0.5	1.5	1.0	1.5
Control	11.0	0.0	2.0	1.0	0.5	3.0	15.0
Control	13.0	1.0	0.5	2.0	0.0	5.5	17.5
Roasted	10.5	1.0	0.0	0.0	0.0	0.5	1.0
Roasted	12.0	1.0	0.0	0.5	0.5	0.0	0.0

## CONCLUSIONS

The use moist and/or dry solid-to-solid heat pretreatments proved to be successful for improving storage quality of beans. A short heat treatment with hot sand was adequate for desinfestation, enzyme inactivation and moisture reduction. This simple technique was also promising for hardening control at high storage temperature through reduced moisture content.

## ACKNOWLEDGMENTS

The authors acknowledge financial support from the International Development Research Centre (IDRC), from Canada. Appreciation is also expressed to Elizabeth Vega and Jorge Rivera for technical assistance.

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# SOFTENING OF FISH BONE. I. RELATION BETWEEN SOFTENING RATE AND SOLUBILIZATION RATE OF ORGANIC MATTER FROM FISH BONE

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Accepted for Publication May 18, 1987

## ABSTRACT

*Cooking rate of spines of mackerels was experimentally examined at various temperatures in distilled water. Hardness ( $F/a$ ) was determined by the maximum load applied in shearing the sample,  $F(N)$ , divided by the maximum width of the sample,  $a(m)$ . Softening rate could be described as a first-order reaction with reference to hardness with activation energy of 61 kJ/mol.*

*Solubilization rate of organic matter and release rate of ash from fish bones were determined. Solubilization rate of organic matter was also described as a first-order rate process with activation energy of 73 kJ/mol. Ash was not released from fish bone during cooking.*

*Hardness after cooking was proportional to the fourth power of degree of remaining of organic matter. It was suggested that hardness of cooked spine of mackerel was dependent on organic matter remaining in spine after cooking.*

*Observations of surfaces of spine confirm that cooking caused change of mechanical structure in spine.*

## INTRODUCTION

Recently, cooking of fish bone has been studied (Watanabe *et al.* 1985; Hatae *et al.* 1980), because the bone is a calcium-rich natural food. Since the separation of bone from fish flesh requires much labor especially in the case of small fishes, development of an appropriate softening process of fish bone in situ may expand its utilization as food. Several processes to make fish bone edible can be conceived: mincing, grinding, cooking and acid treatment (Watanabe *et al.* 1985; Hatae *et al.* 1980; Ohta 1981). The present study has dealt with cooking.

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Early in the 1940's, the hardness of fish bone was examined for the purpose of utilizing it as an index of degree of cooking of canned fish (Shostrom 1940; Matsuike *et al.* 1941). Shostrom (1940) measured the hardness of vertebrae of four kinds of salmon and found that the longer the cooking time, the more the vertebrae softened and hardness depended on the species. Matsuike *et al.* (1941) investigated the hardness of two kinds of salmon and found that the softening rate increased with the cooking temperature. Hatae *et al.* (1980) investigated the effect of green tea infusion on cooking of fish bone. Watanabe *et al.* (1985) investigated the softening rate of mackerel spine and found that the softening rate could be described as a first-order reaction with reference to hardness, and an apparent activation energy of the reaction was 93.2 kJ/mol.

Except for the research cited above, available information about the mechanical strength of bone is limited to bones of mammals (e.g. Vose *et al.* 1959; Currey 1969a,b; Bell *et al.* 1947; Evans 1953). Vose *et al.* (1959), Bell *et al.* (1947) and Currey (1969a) found a positive correlation between the breaking stress of human femora and its ash content. Currey (1969a) speculated that the end to end fusion of apatite crystals, as the matrix becomes saturated with minerals, might increase the mechanical strength of bone. This thought suggests that removal of ash makes bone soft. On the other hand, collagen which accounts for 80% of the organic matter in bone (e.g. Herring 1972; Kubota *et al.* 1975) has a tensile strength which is about five times that of bone (Ascenzi *et al.* 1972). Collagen is solubilized through gelatinization in hot water of 50 °C–80 °C (Irie 1962). These suggest that solubilization of organic matter might cause softening of bone.

For the purpose of clarifying the softening process of fish bone, the present study has dealt with the effect of release of organic matter and ash on softening of fish bone in water cooking.

## MATERIALS AND METHODS

Changes of hardness and solubilization of organic matter and release of ash from fish bone were determined at temperatures ranging from 80 °C to 140 °C, and the surface changes accompanied by them were observed by scanning electron microscope (SEM).

### Materials

The spines of mackerels landed at Hachiohe, Japan in October 1985 were used as bone samples. Mackerels were stored at –20 °C until they were thawed at room temperature before use.



## Methods

Ten pieces of spines were put into a test tube made of stainless steel to which distilled water was added. The test tube was sealed with a screw thread cap and kept in a heating block at 80 °C to 140 °C for a preset period of time. After an appropriate interval, the tubes were immediately cooled in tap water. The contents, washed with water, were subjected to determinations of hardness, organic matter and ash. The definition and measurement of hardness were the same as those described by Watanabe *et al.* (1985). The sample was mounted on the stage which moved upwards at a constant speed, and was pressed against a razor blade fixed to the upper part. Finally the blade cut the sample (Fig. 1). The load on the blade was recorded continuously. The cutting force,  $F(N)$ , was defined as the maximum load recorded. The position to be cut was marked in advance and its width was measured by a digital slide caliper (500-111, MITUTOYO MFG. Co., Ltd., Tokyo) and called the cutting width,  $a(m)$ . The hardness was defined as the cutting force divided by the cutting width,  $F/a$  (N/m). After the measurement of hardness, all spines cooked at each temperature were collected in a small aluminum cup and oven-dried at 105 °C for 8 h and weighed. Then the dried matter was ignited in an electric furnace at 600 °C for 8 h and the ash left behind was weighed. The weight was called residual ash. The weight of the dry matter minus the ash was defined as residual organic matter.

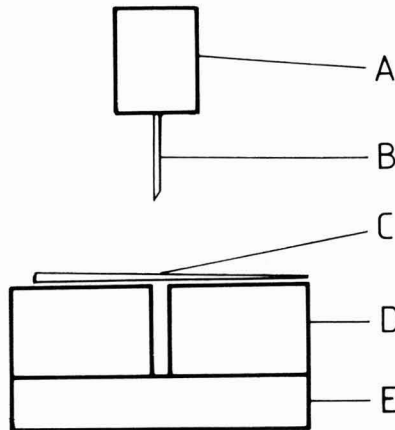


FIG. 1. A SCHEMATIC DIAGRAM OF CUTTING APPARATUS

- A: Blade holder
- B: Razor blade
- C: Sample
- D: Sample table
- E: Elevating stage.

## RESULTS AND DISCUSSION

After bones had been cooked in water at 80°C to 140°C for different periods of time, hardness, residual organic matter and residual ash were determined.

When the logarithm of  $F/a$  was plotted against time, a straight line was obtained for each temperature, as shown in Fig. 2. These results show that softening of fish bone proceeds as a first-order reaction with reference to hardness. The slope of the straight line multiplied by  $-2.303$  gave the first-order reaction rate constant,  $k_h$  ( $s^{-1}$ ).

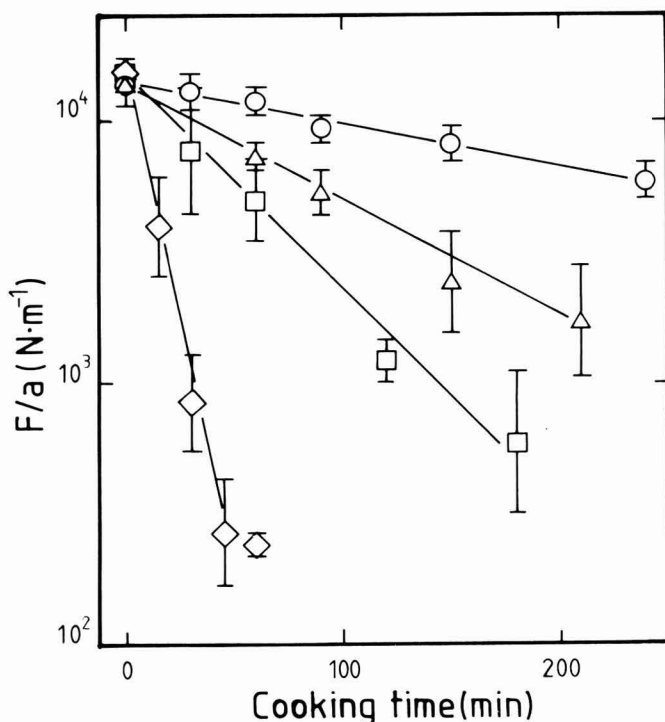


FIG. 2. SOFTENING OF MACKEREL SPINE IN HOT WATER. The variation ( $\delta_{n-1}$ ) are shown in the figure.  $\circ$ : 80°C,  $\triangle$ : 100°C,  $\square$ : 120°C,  $\diamond$ : 140°C.

Let us define the degree of remaining organic matter,  $\eta$ , as the ratio of residual organic matter in cooked bone to that in raw bone. The logarithm of the degree of remaining organic matter,  $\eta$ , decreased linearly with time at temperatures from 80°C to 140°C (Fig. 3). The results represent that the solubilization of organic matter can be described by a first-order rate process with reference to organic matter. The first-order reaction rate constant,  $k_o$  ( $s^{-1}$ ), was evaluated from the slope of Fig. 3. The remaining ash was  $100 \pm 12\%$  of ash in the raw bone and showed no tendency to change with time at temperatures ranging from

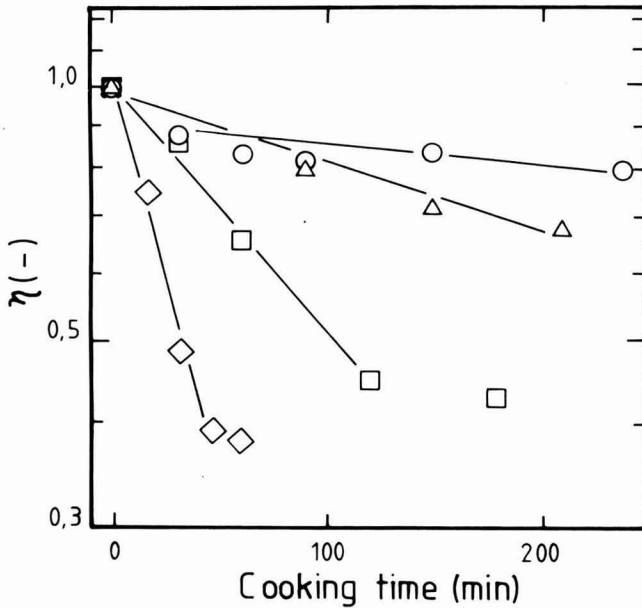


FIG. 3. SOLUBILIZATION OF ORGANIC MATTER FROM MACKEREL SPINE IN HOT WATER ○:80°C, △:100°C, □:120°C, ◇:140°C.

80°C to 140°C. The results indicate that the ash constituents were not released in water at or below 140°C. It can be concluded that the softening of fish bone in water cooking is not caused by the release of ash constituents.

The logarithm of  $k_h$  and  $k_o$  clearly showed linear dependence of the reciprocal of cooking temperature (Fig. 4). The apparent activation energy  $E_a$  (kJ/mol) was evaluated from the slope of straight line in Fig. 4:

$$E_a = -2.303 \cdot R \cdot (\text{slope of line}) \quad (1)$$

where  $R$  denotes gas constant ( $8.314 \times 10^{-3}$  kJ/(mol·K)). The apparent activation energies of softening rate and solubilization rate of organic matter from fish bone were 61 kJ/mol and 73 kJ/mol, respectively. When we consider the scatter of data in Fig. 4, the difference between these data can be ignored.

Matsuike *et al.* (1941) measured the load applied on the vertebrae when the vertebrae were crushed between two plates, and defined hardness as the ratio of applied load to cross sectional area of the vertebra. Since the logarithm of hardness thus defined decreased linearly within 30 min, we evaluated the first-order reaction rate constants from Tables 2 and 3 which appeared in Matsuike *et al.* (1941). These values were plotted in Fig. 4. The figures shows that activation energies of softening of bone from salmon or mackerel is in the range from 61 kJ/mol to 113 kJ/mol.

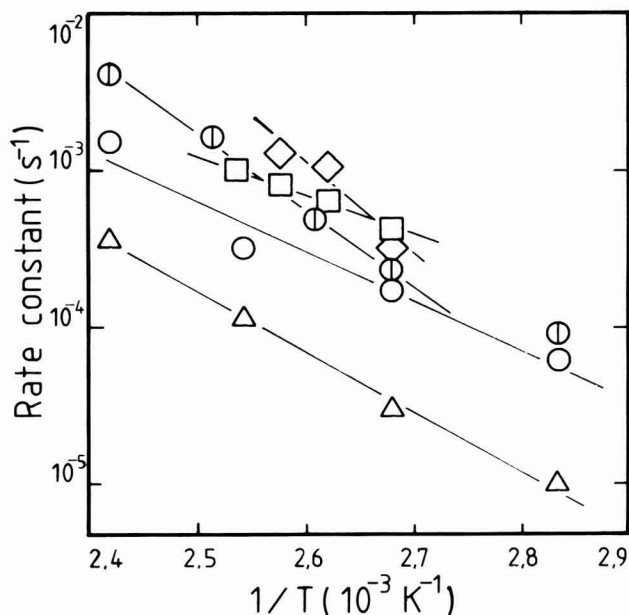


FIG. 4. ARRHENIUS PLOTS OF SOFTENING RATES OF MACKEREL, PINK SALMON AND KETA SALMON AND SOLUBILIZATION RATE OF ORGANIC MATTER FROM MACKEREL SPINE ○: softening rate of mackerel spine of the present study, △: release rate of organic matter from mackerel spine, ⊙: softening rate of mackerel spine (Watanabe *et al.* 1985), □ and ◇: softening rates of vertebrae of pink and keta salmon (calculated from Tables 2 and 3 of Matsuike *et al.* 1941).

The relationship between the hardness,  $F/a$ , and the degree of remaining of organic matter,  $\eta$ , is shown in Fig. 5. Equation (2) describes the straight line in the figure.

$$\begin{aligned} F/a &= 1.6 \cdot 10^4 (\eta)^{4.1} & (2) \\ r &= 0.959, n = 18 \end{aligned}$$

Equation (2) indicates that the hardness is approximately proportional to the fourth power of the degree of remaining of organic matter.

Photomicrographs of a mackerel spine obtained by SEM are presented in Fig. 6-a,b,c and Fig. 7-a,b,c. In the low magnification photographs ( $\times 100$ ; Fig. 6-a,b,c), it looks as if the connective tissue on the surface of a mackerel spine has been removed during cooking. After 40 min cooking at  $125^\circ\text{C}$ , cracks and numerous small holes appeared on the surface (Fig. 6-c) which could not be observed on the raw surface (Fig. 6-a). In the high magnification photographs ( $\times 1000$ ; Fig. 7-a,b,c) we can observe in detail changes of these small holes.

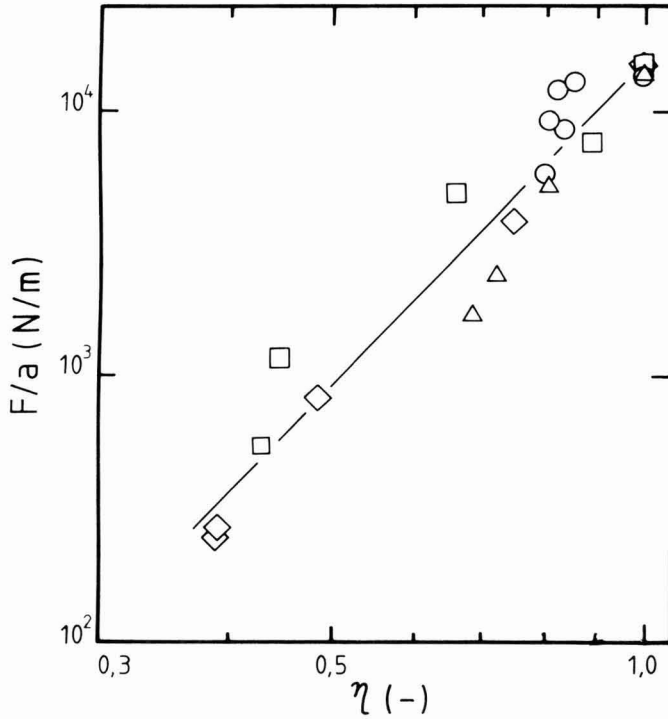


FIG. 5. CORRELATION BETWEEN THE HARDNESS,  $F/a$ , AND THE DEGREE OF REMAINING ORGANIC MATTER,  $\eta$ .

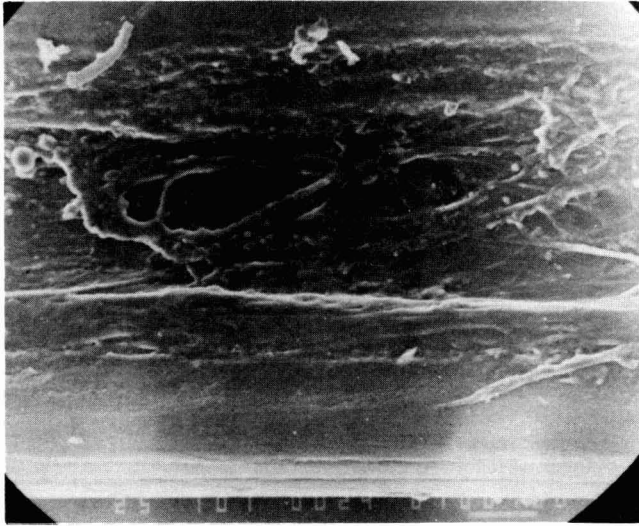


FIG. 6-a. SURFACE OF A RAW MACKEREL SPINE PHOTOGRAPHED THROUGH SEM ( $\times 100$ ) Scale (lower right) corresponds to 100  $\mu\text{m}$ .

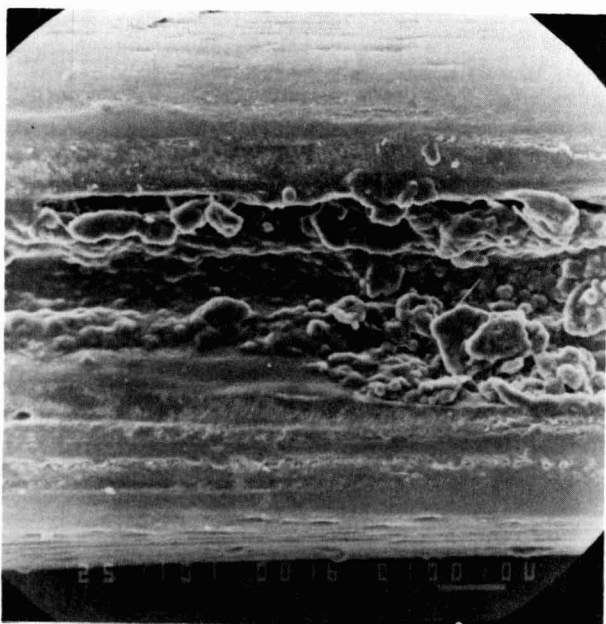


FIG. 6-b. SURFACE OF A MACKEREL SPINE COOKED FOR 15 MIN AT 125°C PHOTOGRAPHED THROUGH SEM ( $\times 100$ ) Scale (lower right) corresponds to 100  $\mu\text{m}$ .

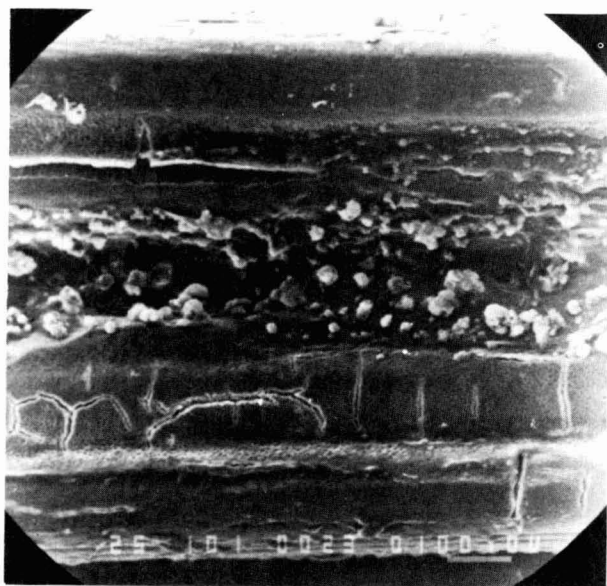


FIG. 6-c. SURFACE OF A MACKEREL SPINE COOKED FOR 40 MIN AT 125°C PHOTOGRAPHED THROUGH SEM ( $\times 100$ ) Scale (lower right) corresponds to 100  $\mu\text{m}$ .

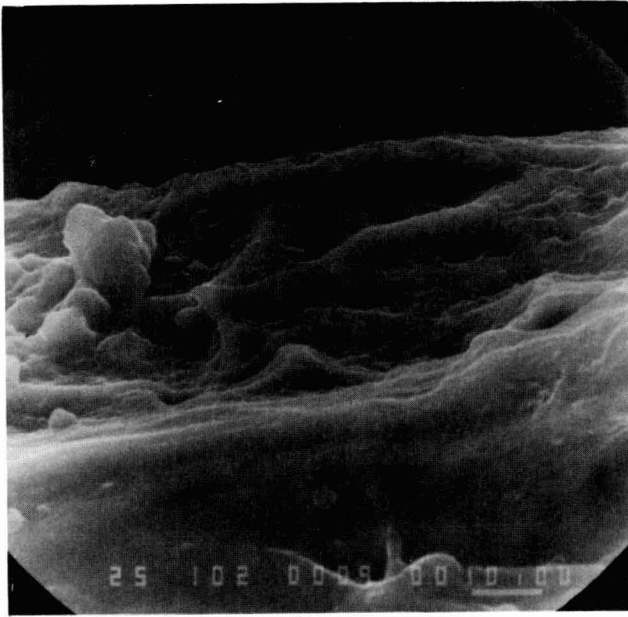


FIG. 7a. SURFACE OF A RAW MACKEREL SPINE PHOTOGRAPHED THROUGH SEM ( $\times 1000$ ) Scale (lower right) corresponds to 10  $\mu\text{m}$ .

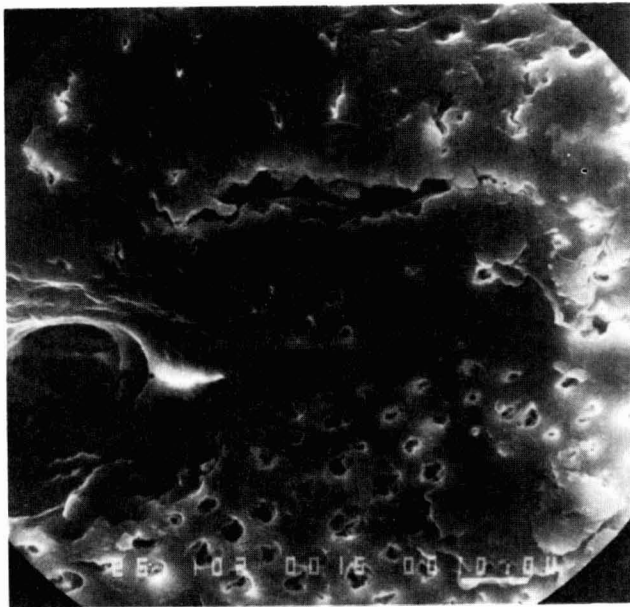


FIG. 7-b. SURFACE OF A MACKEREL SPINE COOKED FOR 15 MIN AT 125°C PHOTOGRAPHED THROUGH SEM ( $\times 1000$ ) Scale (lower right) corresponds to 10  $\mu\text{m}$ .

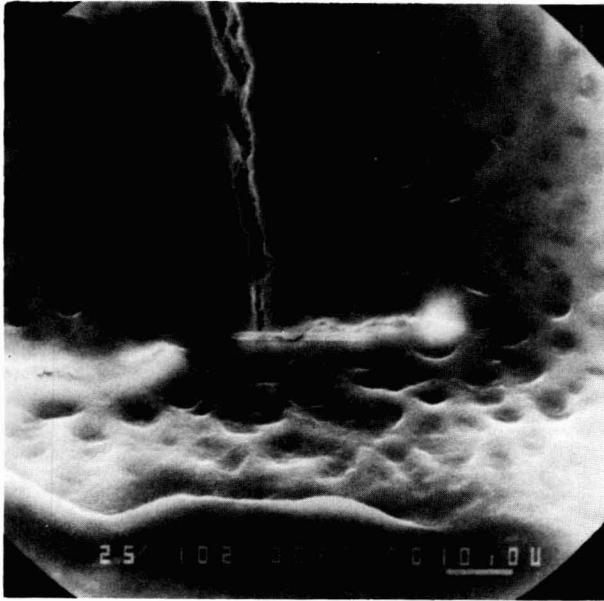


FIG. 7-c. SURFACE OF A MACKEREL SPINE COOKED FOR 40 MIN AT 125°C PHOTOGRAPHED THROUGH SEM ( $\times 1000$ ) Scale (lower right) corresponds to 10 $\mu$ m.

After 15 min cooking, numerous small holes with irregular shape were observed (Fig. 7-b), and after 40 min cooking, these holes became larger and the edges of the holes became smooth (Fig. 7-c). These changes may suggest that the organic matter was released during cooking. Although the quantitative interpretation of the effect of surface change on hardness is impossible, observations by SEM confirm the disappearance of the connective tissue accompanied with softening, and suggest the importance of the connective tissue on the hardness of fish bone.

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# YIELD AND TENDERNESS OF CURED CHICKEN ROLL PRODUCTS AS AFFECTED BY WATER ADDITION<sup>1</sup>

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Accepted for Publication March 2, 1987

## ABSTRACT

*Cured chicken roll products were prepared with either skinless breast meat of skinless dark meat from thighs and drumsticks. Similar products were also prepared from pork for comparison. Processing yields were higher for chicken breast meat than for other meat types when added water was greater than 30%. The yield of dark chicken meat products was always the lowest. Added water did not affect ( $P \leq 0.05$ ) the Warner-Bratzler shear values of the products. Dark chicken meat products were found to be the most tender, while those from pork were the least tender. Increase in mixing time increased ( $P \leq 0.05$ ) the processing yield of dark chicken meat products.*

## INTRODUCTION

The use of sectioned and formed meat products is becoming increasingly popular due to the intrinsic advantages of these types of products: easier and more accurate portion control; simulation of boneless high quality cuts; improved tenderness; and decreased processing time (Siegel *et al.* 1978). The binding of pieces or chunks of meat to form rolls or loaves has received considerable attention, especially by the poultry industry. This is due primarily to the small size and the lack of cuts such as roasts and steaks on a poultry carcass.

The water-holding capacity of muscle foods has been utilized as an index of palatability, microbial quality and manufacturing potential (Dagbjartsson and Solberg 1972). It is known to vary with animal species, age, slaughter conditions, cut of meat, storage conditions and manufacturing processes such as comminution, mechanical action, addition of salt (sodium chloride), and phosphate (Ranken 1976). The moisture content of processed meat products is the highest single component, with most originating from the lean meat ingredients. Moreover, water is added to many products as part of the product formulation.

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There are several reasons for adding water: to improve the tenderness and juiciness of the products, to aid in product temperature reduction during emulsification when water is added as ice, to serve as the carrier for distributing the curing ingredients into noncomminuted smoked meat items such as hams and bacons, and to replace moisture that will be lost during processing operations, particularly in heating processes (Forrest *et al.* 1975).

Puolanne and Terrell (1983) demonstrated that when sausages were made with low levels of fat, the desirable properties of tenderness and juiciness were achieved by increasing levels of added water. Uram *et al.* (1984) reported that tenderness and juiciness scores as well as overall yields of smoked sausage increased as the amount of added water was increased to 10%; however, there was no further increase in these factors as the amount of added water was increased to 20%. They also emphasized the importance of added water in improving sensory characteristics and lowering production costs.

"Water-added" products are generally more tender and juicy and meet with consumer acceptance. However, processed meat products must comply with federal meat inspection regulations; moisture content must not exceed four times the meat protein content (by analysis) plus 10% (Forrest *et al.* 1975; Puolanne and Terrell 1983).

Breclaw and Dawson (1970) reported that light chicken meat rolls contained more moisture, gave a higher yield, were more tender and exhibited better binding characteristics than the dark chicken meat rolls. Acton *et al.* (1979) reported that turkey-ham yields significantly ( $P \leq 0.05$ ) decreased from 96.5% to 37.8% internal temperature to 87.3% at 71.1 °C internal temperature. Shear values also decreased as internal temperature increased, indicating tenderness development. Baker and Darfler (1981) prepared poultry ham products from turkey thigh meat, broilers, light and heavy fowls. Their results showed that the most acceptable hams were made by mixing with 0.5% polyphosphates and at least 1% salt or adding as much as 20% water for an increased yield. They also found that a greater yield was obtained by cooking in water than in a smoker or canning under pressure.

This study was conducted to investigate the effect of added water on the cooking yields, moisture distribution and shear values of cured chicken roll products.

## MATERIALS AND METHODS

### Sample Preparation

Commercial type broiler carcasses were obtained from the Mississippi State University poultry processing plant and stored at  $-18^{\circ}\text{C}$ . Carcasses were thawed in a  $2-4^{\circ}\text{C}$  refrigerator overnight prior to each study. The breast and dark

meat (from thighs and drumsticks) were separated from the carcass by hand and the skin and external fat were removed. Fresh pork (Boston butt) was purchased from a grocery store and the excess fat and connective tissue were trimmed off. Meat samples were cut separately into 2.5 cm cubes and kept in a 2–4 °C refrigerator.

The basic product formula of this study consisted of 1000.0 g meat, 20.0 g salt, 12.0 g monosodium glutamate, 10.0 g sugar, 5.0 g polyphosphate (Kena, Stauffer, CT), 3.5 g white pepper powder, 3.0 g onion powder, 0.8 g garlic powder, 1.0 g potassium sorbate, 0.6 g sodium ascorbate, and 0.2 g sodium nitrite. Water was added at 10% increments based on the raw meat weight and ranged from 0 to 90% for chicken breast meat and 0 to 70% for dark chicken meat. For pork, 0, 10, 20, 40, 60, and 80% added water levels were tested. Curing agents were added to the experimental meat chunks and mixed in a Kitchen Aid Model K5SS mixer for 3 min using a flat paddle at speed #1. Flavoring ingredients and water were then added and mixed at the same speed for an additional 2 min.

The prepared mixtures were cured in a 2–4 °C refrigerator for 1 h and stuffed into easy peel casings (Tipper Tie, Inc., Chicago, IL) to form 6 cm diameter chubs. The chubs were weighed and water-cooked at 180 °F (82.2 °C) to an internal temperature of 155 °F (68.3 °C) as determined by copper constantan thermocouples with a Speedmax M multipoint recording potentiometer (Leeds and Northrup Co., North Wales, PA).

### Yield Determinations

After cooking, the chubs were cooled at room temperature approximately 30 min, peeled and weighed. The weight of the peeled casing was also recorded. Yields were calculated as follows:

$$\% \text{ Overall processing yield} = \frac{\text{weight of peeled product}}{\text{weight of all ingredients excluding added water}} \times 100$$

$$\% \text{ Cooking yield} = \frac{\text{weight of peeled product}}{\text{weight of all ingredients including added water}} \times 100$$

### Shear Value Measurements

A Warner-Bratzler coring tool was used to remove triplicate 1.27 cm diameter cores from each cooked sample. Ten readings per core were obtained using a Warner-Bratzler shearing apparatus (50 × 0.1 lb capacity), and measurements were expressed as the kg of force required to shear each core. For the products with poor binding appearance, samples were removed from the center of cooked meat chunks.

### **Moisture Content Determination**

Sample cores used for shear measurement were diced into approximately 0.5 cm cubes. Subsamples were dried in a 100–102 °C oven for 16–18 h then weighed for moisture content calculation (AOAC 1980).

### **Mixing Time and Yield**

The effect of short-term mixing time on the yield of cured products from dark chicken meat with 50% added water was investigated. Product preparation procedures as described earlier were followed, but the total mixing time after the addition of water was increased in increments of 2 min from 5 to 13 min.

### **Distribution of Moisture in Products**

Refrigerated samples (approximately 2.5 cm thickness) of the dark chicken meat product with 11 and 13 min of mixing at 50% added water were carefully sliced so that the subsamples could be collected from both inside the individual meat chunks as well as between the chunks. Moisture content of the subsamples was then determined.

### **Statistical Analysis**

All data were subjected to analysis of variance by Statistical Analysis System (SAS, 1982). Student-Newman-Keuls (SNK) test at the 5% level of probability was used to separate means.

## **RESULTS AND DISCUSSION**

The overall processing yield of cured chicken roll products from chicken breast meat increased as the amount of added water increased (Table 1). However, poor binding in the product was observed when 70% or more water was added. Poor binding was observed for the dark chicken meat products when 50% or more water was added. Unlike chicken breast meat products, the overall processing yield of dark chicken meat products did not increase ( $P \geq 0.05$ ) beyond a 10% water added level (Table 1). In addition, the products from chicken breast meat produced higher overall processing yields than the comparable products from dark chicken meat. This higher water binding capacity of breast meat has been observed in chicken rolls prepared from ground meat (Froning and Norman 1967) and in chicken meat loaves (Maesso *et al.* 1970). Products with water addition greater than "four times the meat protein content plus 10% red meat would be labeled "imitation" and greater than 50% in poultry meat would have severe problems with USDA labeling since water would be the first ingredient.

TABLE 1.  
OVERALL PROCESSING YIELDS OF CURED POULTRY MEAT AND PORK PRODUCTS AS  
AFFECTED BY WATER ADDITION AND MEAT TYPES<sup>1,2</sup>

% Water added <sup>3</sup>	Overall processing yield (%)		
	Breast meat	Dark meat	Pork
0	92.59 <sup>i</sup>	92.08 <sup>b</sup>	94.14 <sup>d</sup>
10	97.57 <sup>h</sup>	97.47 <sup>a</sup>	100.06 <sup>c</sup>
20	105.46 <sup>g</sup>	100.07 <sup>a</sup>	106.81 <sup>b</sup>
30	110.12 <sup>f</sup>	98.57 <sup>a</sup>	-----
40	115.30 <sup>e</sup>	100.32 <sup>a</sup>	110.93 <sup>a*</sup>
50	118.62 <sup>d</sup>	101.29 <sup>a*</sup>	-----
60	125.41 <sup>c</sup>	100.83 <sup>a*</sup>	113.31 <sup>a*</sup>
70	126.83 <sup>bc*</sup>	100.68 <sup>a*</sup>	-----
80	129.97 <sup>ab*</sup>	-----	112.54 <sup>a*</sup>
90	132.43 <sup>a*</sup>	-----	-----

<sup>1</sup>Mean of 3 observations.

<sup>2</sup>Means within a column not followed by the same letter are significantly different ( $P \leq 0.05$ ).

<sup>3</sup>Based on the raw meat weight.

\*Poor binding.

The effect of added water on the overall processing yield of the pork product was slightly greater than the chicken breast meat product when added water was 20% or lower, but no increase ( $P \geq 0.05$ ) was observed when the added level was above 40% (Table 1). It should be kept in mind that in this study, a short-term mixing was used in product preparation instead of "massaging" as used in commercial ham manufacturing.

Based on all components, including water, the cooking yield of products from these three meat types decreased as the amount of added water increased (Table 2). In general, chicken breast meat had the highest cooking yields while the dark chicken meat had the lowest. The decrease in cooking yield appeared to be linearly related to the increase in added water. One of the factors affecting water retention properties of the heated products is pH value of the meat. According to Lyon *et al.* (1984), regardless of post-mortem holding time, pH of chicken breast meat was lower than that of leg meat. In addition, the pH was not significantly affected by holding time.

TABLE 2.  
COOKING YIELDS OF CURED POULTRY MEAT AND PORK PRODUCTS AS AFFECTED  
BY WATER ADDITION AND MEAT TYPES

% Water added <sup>3</sup>	Cooking yield (%)		
	Breast meat	Dark meat	Pork
0	92.00 <sup>a</sup>	91.69 <sup>a</sup>	93.94 <sup>a</sup>
10	90.53 <sup>ab</sup>	89.55 <sup>a</sup>	91.14 <sup>b</sup>
20	88.76 <sup>bc</sup>	84.24 <sup>b</sup>	90.07 <sup>b</sup>
30	86.92 <sup>cd</sup>	78.27 <sup>c</sup>	----
40	84.83 <sup>d</sup>	74.18 <sup>cd</sup>	81.55 <sup>c*</sup>
50	81.98 <sup>e</sup>	70.03 <sup>d*</sup>	----
60	80.40 <sup>e</sup>	64.33 <sup>e*</sup>	73.07 <sup>d*</sup>
70	77.80 <sup>f*</sup>	61.71 <sup>e*</sup>	----
80	75.02 <sup>g*</sup>	----	64.91 <sup>e*</sup>
90	71.98 <sup>h*</sup>	----	----

<sup>1</sup>Mean of 3 observations.

<sup>2</sup>Means within a column not followed by the same letter are significantly different ( $P \leq 0.05$ ).

<sup>3</sup>Based on the raw meat weight.

\*Poor binding.

Regardless of meat sources, Warner-Bratzler (W-B) shear values of the products were not affected ( $P \geq 0.05$ ) by the amount of added water (Table 3). Products with a poor binding appearance were observed to possess a gravy-type structure between the cooked meat chunks. It was expected that this section had a lower W-B shear reading than that of the meat chunks. The products from dark chicken meat were lower in W-B shear values than those from chicken breast meat. Results agree with the data from chicken rolls (Froning and Norman 1967) and cooked chicken meat (Steedman *et al.* 1979). Pork products were much less tender than the chicken counterparts.

Below 40% added water, the moisture content of chicken breast meat products increased ( $P \leq 0.05$ ) as the amount of added water increased. For dark chicken meat products, added water above 20% did not ( $P \geq 0.05$ ) affect the moisture content. Product sections with a gravy-type structure were expected to have a higher moisture content than that of the meat chunk sections. It is of interest to note that the moisture of the pork product was not significantly ( $P \geq 0.05$ ) affected by the addition of water (Table 4). In processed red meat products, the moisture content must comply with federal meat inspection standards and must not exceed four times the meat protein content (by analysis) plus 10% (Forrest *et al.* 1975).



TABLE 3.  
WARNER-BRATZLER (W-B) SHEAR VALUE OF CURED POULTRY MEAT AND PORK  
PRODUCTS AS AFFECTED BY WATER ADDITION AND MEAT TYPES<sup>1,2</sup>

% Water added <sup>3</sup>	W-B shear value (kg/1.27 cm cores)		
	Breast meat	Dark meat	Pork
0	0.65	0.50	1.03
10	0.58	0.49	1.00
20	0.57	0.50	1.06
30	0.60	0.51	----
40	0.55	0.44	1.07*
50	0.61	0.45*	----
60	0.62	0.54*	1.14*
70	0.55*	0.50*	----
80	0.58*	----	1.07*
90	0.67*	----	----

<sup>1</sup>Mean of 10 observations.

<sup>2</sup>Means within a column are not significantly different ( $P \geq 0.05$ ).

<sup>3</sup>Based on the raw meat weight.

\*Poor binding.

In general, both the overall processing yield and the cooking yield of cured chicken rolls from dark chicken meat with 50% water added increased as mixing time increased (Table 5). Dark chicken meat products with 50% or more added water were poor in binding (Table 1); therefore, it was selected for the mixing time study. Increased mixing time also improved the binding quality of the cooked products. Poor binding was observed for the dark chicken meat products mixed for 50 min, but this was improved by increasing mixing time.

Previous work (Aref and Tape 1966; Acton 1972a, Pepper and Schmidt 1975) reported that mechanical mixing caused much cell destruction, but promoted surface extraction of soluble protein which functioned as an excellent binder. Booren *et al.* (1980) reported a linear improvement ( $P \leq 0.01$ ) in cooking yield in sectioned and formed beef steak due to mixing for up to 18 min. Acton (1972b) stated that cooking loss decreased and binding strength increased in chicken meat when tissue rupture increased.

As indicated in Table 1, the processing yield of dark chicken meat products was the lowest overall. An increase in mixing time up to 13 min improved the yields, making it comparable to those of chicken breast meat products with the same level of added water.

TABLE 4.  
MOISTURE CONTENT OF CURED POULTRY MEAT AND PORK PRODUCTS AS  
AFFECTED BY WATER ADDITION AND MEAT TYPES<sup>1,2</sup>

% Water added <sup>3</sup>	Moisture content (%)		
	Breast meat	Dark meat	Pork
0	70.53 <sup>f</sup>	72.53 <sup>b</sup>	72.44 <sup>a</sup>
10	72.45 <sup>e</sup>	73.50 <sup>b</sup>	72.98 <sup>a</sup>
20	74.52 <sup>d</sup>	74.59 <sup>a</sup>	74.11 <sup>a</sup>
30	76.07 <sup>c</sup>	75.41 <sup>a</sup>	-----
40	77.39 <sup>b</sup>	76.25 <sup>a</sup>	74.92 <sup>a*</sup>
50	78.22 <sup>ab</sup>	76.25 <sup>a*</sup>	-----
60	78.27 <sup>ab</sup>	76.00 <sup>a*</sup>	75.93 <sup>a*</sup>
70	79.41 <sup>a*</sup>	76.60 <sup>a*</sup>	-----
80	79.94 <sup>a*</sup>	-----	74.56 <sup>a*</sup>
90	78.42 <sup>ab*</sup>	-----	-----

<sup>1</sup>Mean of 3 observations.

<sup>2</sup>Means within a column not followed by the same letter are significantly different ( $P \leq 0.05$ ).

<sup>3</sup>Based on the raw meat weight.

\*Poor binding. Samples from the center of the cooked meat chunks were used for the determination.

TABLE 5.  
MEAN OVERALL PROCESSING AND COOKING YIELDS OF CURED CHICKEN ROLL  
PRODUCTS FROM DARK CHICKEN MEAT WITH 50% ADDED WATER AS AFFECTED BY  
THE LENGTH OF MIXING TIME<sup>1,2</sup>

Mixing time (min)	Yields (%)	
	Overall processing	Cooking
5	101.29 <sup>c</sup>	70.03 <sup>c</sup>
7	104.82 <sup>bc</sup>	72.69 <sup>bc</sup>
9	107.23 <sup>bc</sup>	74.13 <sup>bc</sup>
11	110.73 <sup>b</sup>	75.56 <sup>b</sup>
13	117.04 <sup>a</sup>	80.70 <sup>a</sup>

<sup>1</sup>Mean of 3 observations.

<sup>2</sup>Means within a column not followed by the same letter are significantly different ( $P \leq 0.05$ ).

Moisture of cooked meat samples taken from the inside portion of meat chunks was lower ( $P \leq 0.05$ ) than those from in-between the meat chunks (Table 6). The increase in mixing time was also observed to increase the moisture content inside the meat chunks.

TABLE 6.  
MOISTURE CONTENT OF CURED CHICKEN ROLL PRODUCTS FROM DARK CHICKEN MEAT AS AFFECTED BY MIXING TIME AND LOCATION<sup>1</sup>

Location	Yields (%)	
	11 min mixing	13 min mixing
Inside meat chunks	75.04 <sup>bB</sup>	76.16 <sup>bA</sup>
In-between meat chunks	78.57 <sup>aA</sup>	78.12 <sup>aA</sup>

<sup>1</sup>Mean of 9 observations.

<sup>ab</sup>Means within a column not followed by the same letter are significantly different ( $P \leq 0.05$ ).

<sup>AB</sup>Means within a row not followed by the same letter are significantly different ( $P \leq 0.05$ ).

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# COMPOSITION AND PHYSICO-CHEMICAL PROPERTIES OF YAM (*DIOSCOREA SPECIES*) FLOUR PREPARED USING DIFFERENT PROCESSES

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Accepted for Publication March 26, 1987

## ABSTRACT

*Five processes were evaluated for producing flour from yam (Dioscorea) tubers. Variations in processes included drying raw peeled slices of tubers in a forced-air oven, a solar dryer and a drum dryer before milling. Tubers, both peeled and unpeeled, were also boiled before drying in an oven and milling. Flours with acceptable sensory qualities were produced using all test procedures; however, considering the need for human and mechanical energy input, flours produced from raw tubers involving oven- or solar-drying appear to be most practical for application in developing countries where yam tubers are an integral part of the human diet.*

## INTRODUCTION

Yam tubers (*Dioscorea* spp.) are a major part of the diets of many West Africans. Tubers are consumed in several forms, including boiled, mashed or fried, and in the form of steamed and baked pastes and dough. Consumption is highest during the harvest period and decreases to nil over a 6- to 8-month period during which availability is substantially reduced due to sensory quality and microbiological deterioration. Up to 25% of the crop may be lost due to physiological degradation (Coursey 1961). Gamma irradiation and storage at refrigeration temperatures prolong storage stability, but neither technique is economically feasible.

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Studies have been conducted to determine the role of acid phosphatases in physiological degradation processes in yams (Ugochukwu *et al.* 1977; Kamenan and Diopoh 1982). Information about metabolic changes in tubers during degradation and their effects on chemical and physical properties of products eventually produced will be valuable when designing processes for preserving and preparing tubers.

Freshly cut surfaces of yam tubers rapidly undergo undesirable enzymatic browning reactions. These reactions can be minimized by reducing the availability of oxygen, heating and/or removal of water. One process by which the storage life of yams could be lengthened would be to dry the tubers and then process them into flour. A combination of yam and legume or cereal flours has been suggested as a way of improving the nutritional quality of food products in countries where cereals are not produced in sufficient quantities to meet local needs (Hanh and Rasper 1974; Martin and Ruberte 1975; Collins and Falasinnu 1977; Sefa-Dedeh *et al.* 1977). The procedures followed to obtain yam flours may, however, influence the extent of enzymatic browning and the physicochemical properties of the end product. A study was therefore designed to compare proximate composition and physicochemical properties of yam flours prepared by five different methods.

## MATERIALS AND METHODS

### Preparation of Flours

Yam tubers (*Dioscorea* spp.) were purchased from a farmer's market in Atlanta, Georgia. Flours were prepared using five methods:

**Flour 1.** Washed yams were peeled by hand and sliced (2 mm)(Eagle Tool and Machine Co., Springfield, IL). The slices were then dried in a forced-air oven at 64 °C for 22 h and milled into flour with a Wiley mill (Thomas Wiley Laboratory Mill, Model 4, Arthur H. Thomas Co., Philadelphia, PA) equipped with a 0.5-mm screen. Flour was stored in hermetically sealed bags at 4 °C until analyzed.

**Flour 2.** Yams were peeled and sliced as described for preparing flour 1. Slices were then dried in a solar dryer (Nakayama *et al.* 1983) for 20 h at 60–65 °C. Slices were milled into flour as described in flour 1.

**Flour 3.** Yams were peeled and sliced as described for preparing flour 1. Slices were dried at 153 °C using a drum dryer (Baw-Knox Food and Chemical Equipment, Inc., Model AL C-4, Buffalo, NY) and milled into flour as described for flour 1.

**Flour 4.** Yam tubers were boiled in water for 40 min, peeled, sliced (2 mm) and dried in a forced-air oven at 64 °C for 16 h. Flour was then prepared as described above for flour 1.

**Flour 5.** Yam tubers were peeled, cut into 2- and 3-cm<sup>3</sup>, boiled in water for 20 min, sliced (2 mm) and dried in a forced-air oven at 64 °C for 16 h. Flour was then prepared as described above for flour 1.

### Proximate Composition

**Moisture Content.** Moisture content of flours was determined by calculating the loss in weight of duplicate 10-g samples during a 1-h period at 132 °C.

**Lipid Content.** The lipid content of flours was determined by extracting duplicate 5-g samples with petroleum ether for 16 h using a Goldfisch Extractor (Laboratory Construction Co., Kansas City, MO).

**Protein Content.** Analysis for nitrogen was carried out in duplicate by the macro-Kjeldahl method (AOAC 1975). A conversion factor of 6.25 was used to calculate percentage protein.

**Ash.** The ash content of flours was determined by treating 10-g samples at 450 °C for 15 h.

### Physico-chemical Property Determinations

**Color.** Color of five flours were measured using a Gardner color difference meter (Pacific Scientific, Model XL-845, Bethesda, MD). Instrumental color values are presented in terms of L, *a* and *b* color coordinates. Positive L values signify that the color is approaching white, whereas negative L values indicate that the color is approaching black. Positive *a* values signify red, whereas negative *a* values signify green; positive *b* values signify yellow and negative *b* values signify blue. Reference values for the standard for this experiment were: L = 78.50, *a* = -2.10 and *b* = +23.34.

**pH.** The pH values of flour slurries (10 g flour in 20 mL of distilled water) were determined electrometrically.

**Specific Gravity.** The ratio of the weight of equal volumes of flour and water is reported as specific gravity. Values carry no units (Campbell *et al.* 1979).

**Hygroscopicity.** The amount of water adsorbed by flours stored at 35 °C for 13 days under an atmospheric relative humidity of 30% was determined using a temperature and relative humidity controlled oven (Tenney Benchmark, Tenney Engineering, Inc., Union, NJ).

**Viscosity.** Viscosities of flour slurries (15 g flour suspended in 85 g of distilled water) were measured after 10, 20 and 30 min at 80, 85 and 90 °C using a Brookfield Digital Viscometer (Brookfield Engineering Laboratories, Inc., Model HATD, Stoughton, MA). All measurements were made using a No. 29 spindle at 100, 50, 20, 10, 5, 2.5, 1.0 and 0.5 rpm. Shear rates were calculated by multiplying rpm values by the manufacturer supplied factor of 0.25. Viscosity values corresponding to spindle rotational speeds were computed by the following formula:

$$\text{Viscosity (cp)} = \text{dial reading} \times 5000/\text{spindle speed (rpm)}.$$

All values reported are means of two or more replicates. Variations of no more than 4–5% difference existed between individual values used to calculate these means.

## RESULTS AND DISCUSSION

### Proximate Composition

The proximate composition of flour processed using five different methods is shown in Table 1. The moisture content of flours ranged from 2.46 to 8.19% depending upon the method of processing. The drum-dried product (flour 3) had the highest moisture content, but simple adjustment of drum temperature and/or speed could be made to result in flour with reduced moisture content.

The protein content of yams varies from 5 to 10%, depending upon the species (Rasper 1969a,b; Rasper and Coursey 1967). In the present study, drum-dried flour (number 3) had the lowest protein content (5.46%) and flour prepared from sun-dried tubers (flour 3) had the highest (8.50%). Lipid content was 0.40% or less in all test flours, whereas ash content ranged from 2.74 to 4.05%. The higher ash contents in flours 4 and 5 may be due to loss of non-ash constituents during boiling, thus resulting in a disproportionately higher concentration of ash in the milled flour. The carbohydrate content of flours ranged from 87.92 and 91.52%.

### Physico-Chemical Properties

The pH values of flours ranged from 5.90 to 6.13 and the specific gravities were 0.64 to 0.89 (Table 2). Flours 4 and 5 had the highest specific gravities, indicating that boiling may have resulted in leaching of low specific gravity constituents from the tubers before drying and milling.

The moisture contents of flours adjusted to equilibrate with an atmospheric relative humidity of 30% are listed in Table 3. Values ranged from 6.40% for flour 3 to 6.83% for flour 4 after 13 days of equilibration. The method of preparation did not have a significant influence on water-binding capacity of flours. Thus, sensory quality and microbiological stability of flours as affected by equilibrium moisture content would not be influenced by method of preparation.



TABLE 1.  
PROXIMATE COMPOSITION OF YAM FLOURS PROCESSED  
USING FIVE DIFFERENT METHODS

Flour number	Process method <sup>a</sup>	Moisture	Protein <sup>b</sup>	Lipid <sup>b</sup>	Ash <sup>b</sup>	Carbohydrate <sup>bc</sup>
1	Oven-dried	2.46	7.06	0.40	3.58	88.94
2	Sun-dried	7.10	8.50	0.39	3.15	87.92
3	Drum-dried	8.19	5.46	0.23	2.74	91.52
4	Boiled before peeling, oven-dried	5.41	6.39	0.30	4.05	89.80
5	Boiled after peeling, oven-dried	7.39	6.53	0.21	3.42	89.84

<sup>a</sup>See Materials and Methods section for detailed description of method of processing

<sup>b</sup>Dry weight basis

<sup>c</sup>By difference

TABLE 2.  
pH AND SPECIFIC GRAVITY OF YAM FLOURS PROCESSED  
USING FIVE DIFFERENT METHODS

Flour number	Process method <sup>a</sup>	pH	Specific gravity
1	Oven-dried	5.90	0.64
2	Sun-dried	6.00	0.65
3	Drum-dried	6.13	0.64
4	Boiled before peeling, oven-dried	5.96	0.79
5	Boiled after peeling, oven-dried	5.97	0.89

<sup>a</sup>See Materials and Methods section for detailed description of methods of processing

Color values for yam flours are shown in Table 4. The high L values indicate that all flours are light in appearance, approaching white. Flour 1 had the highest *a* value, indicating a higher level of redness than in other flours. Flour 1 also had the highest *b* value, signifying the greatest yellow color of all test flours.

TABLE 3.  
EQUILIBRIUM MOISTURE CONTENT OF YAM FLOURS PROCESSED  
USING FIVE DIFFERENT METHODS<sup>a</sup>

Time (days) at 30% Relative Humidity, 35°C	Moisture Content (%) in Flour Number				
	1	2	3	4	5
0	2.46	7.10	8.19	5.41	7.39
13	6.65	6.55	6.40	6.83	6.69

<sup>a</sup>See Materials and Methods section for detailed description of methods of processing

TABLE 4.  
COLOR VALUES OF YAM FLOURS PROCESSED USING FIVE DIFFERENT METHODS

Value <sup>a</sup>	Reference	Flour Number <sup>b</sup>				
		1	2	3	4	5
L	+78.50	86.70	89.66	89.65	90.72	86.34
<i>a</i>	-2.10	1.12	0.74	0.65	0.32	0.32
<i>b</i>	+23.34	13.05	7.96	10.83	9.42	9.89

<sup>a</sup>Indices of color (L, *a* and *b*) were determined on quadruplicate samples; data listed represent average values.

<sup>b</sup>See Materials and Methods section for detailed description of methods of processing

## Viscosity

Selected yam flour slurry viscosity curves as a function of shear rates are presented in Fig. 1–4. All curves indicate non-newtonian behavior for yam flour slurries, which was expected. Viscosities for shear rates of 12.5 and 25 s<sup>-1</sup>, i.e., corresponding to spindle speeds of 50 and 100 rpm, respectively, were either negligible or could not be measured by the viscometer, and thus are not illustrated in Fig. 1–4.

Figure 1 shows the effect of method of processing flour on viscosity after holding the flour slurry at 80°C for 10 min. Flour 1 exhibited higher viscosity than all other flours studied. Although products were not prepared from flours, it appears that flour 1 will yield products with highest acceptability.

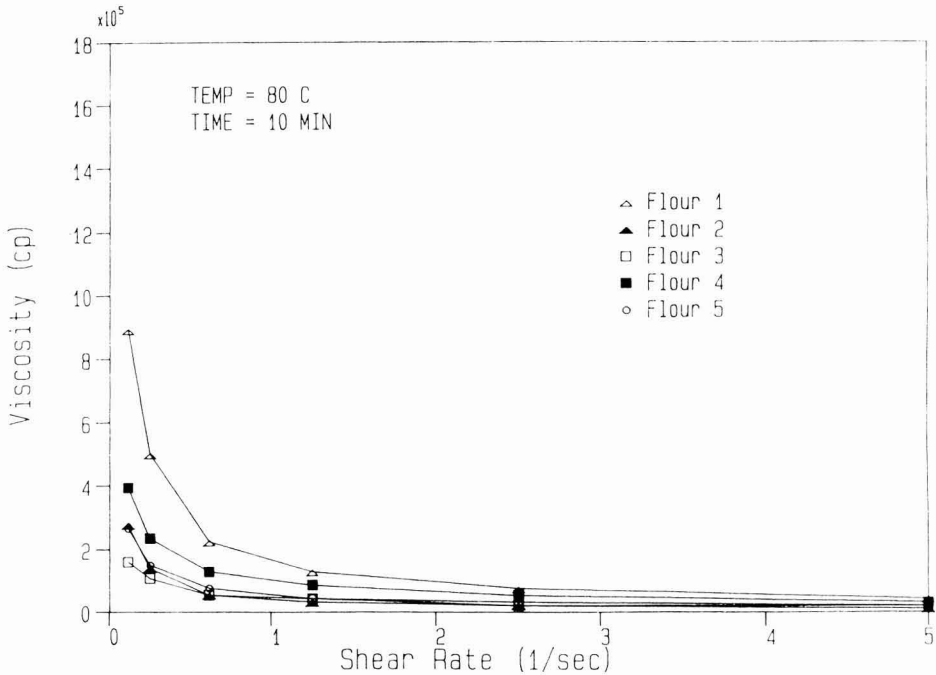


FIG. 1. BROOKFIELD APPARENT VISCOSITY AND SHEAR RATE CURVES OF FIVE YAM FLOUR SLURRIES AT 10 MIN AND 80°C

Cuevas *et al.* (1985) studied viscosity characteristics of precooked corn flour and reported that smaller particle size fractions contribute to lower viscosity values. Difference in viscosity profiles for various flours in Fig. 1 could be due to difference in particle size distribution of flours which could have been influenced by the method of processing the flours.

The effect of temperature on the viscosity profile for flour 1 after holding the slurry for 10 min is shown in Fig. 2. An increase in temperature resulted in an increase in viscosity of the flour slurry at any given shear rate. Similar effects of temperature were observed at other time periods of 20 and 30 min (Fig. 3 and 4, respectively). The effect of time on viscosity at any particular temperature is evident by comparing viscosity curves in Fig. 2–4 for that temperature. It was observed that measurement at higher time values yielded lower viscosity values. Between viscosity measurements at 10, 20 and 30 min, slurry samples were stirred with the spindle rotating at 100 rpm, which probably caused a thinning effect and contributed to lower viscosity readings.

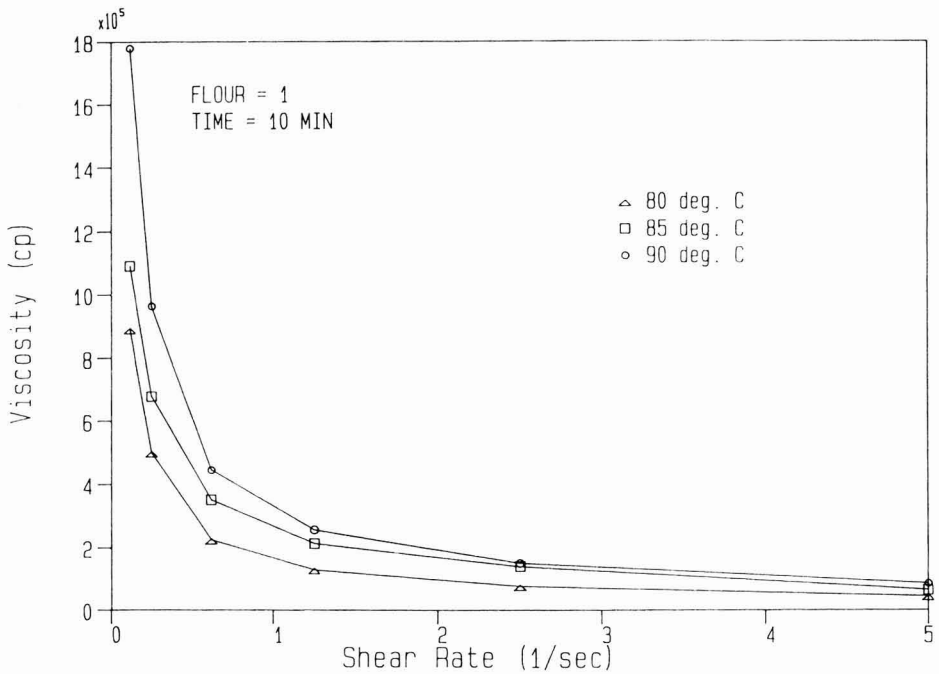


FIG. 2. BROOKFIELD APPARENT VISCOSITY AND SHEAR RATE CURVES FOR SLURRY OF YAM FLOUR 1 AT 10 MIN AND 80, 85 AND 90°C

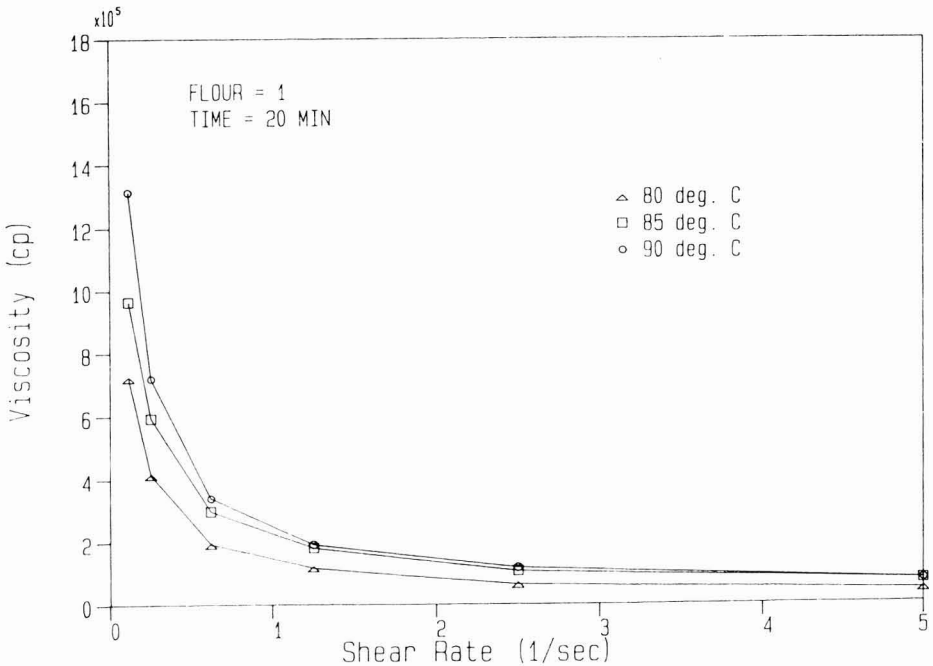


FIG. 3. BROOKFIELD APPARENT VISCOSITY AND SHEAR RATE CURVES FOR SLURRY OF YAM FLOUR 1 AT 20 MIN AND 80, 85 AND 90°C

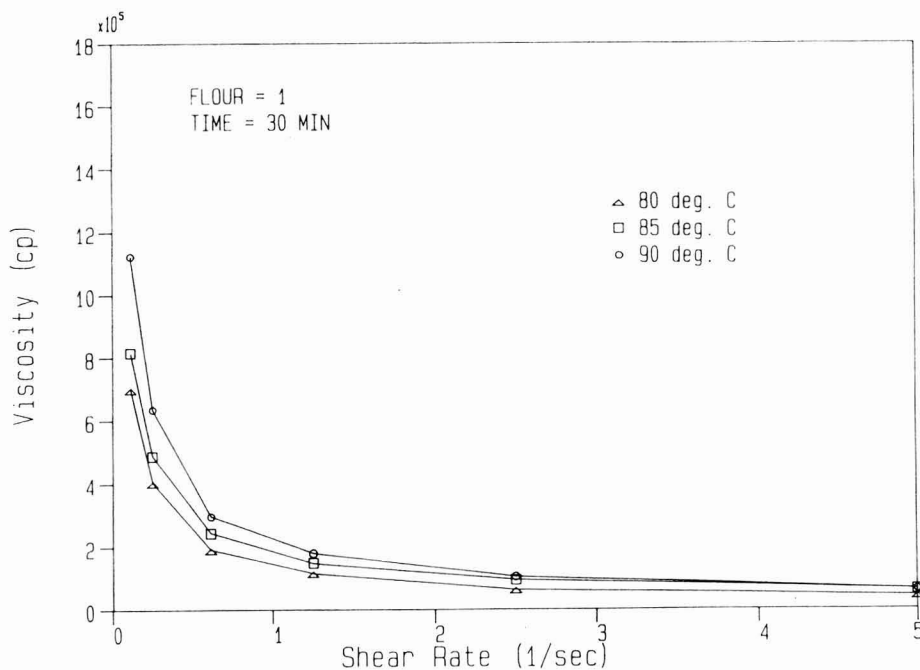


FIG. 4. BROOKFIELD APPARENT VISCOSITY AND SHEAR RATE CURVES FOR SLURRY OF YAM FLOUR 1 AT 30 MIN AND 80, 85 AND 90°C

The data for time and temperature effect on viscosities of slurries made from flours 2-5 are not included in this report, but the effects were similar to those observed for flour 1. Changes in viscosities with temperature and shearing rates observed in our study are consistent with reports in the literature on starchy flours (Rasper and Coursey 1967; Rasper 1969a; Rodriguez-Sosa and Gonzalez 1972).

## SUMMARY AND CONCLUSIONS

Yam flour was prepared by five different processing methods and their proximate composition and physicochemical properties were compared. The results showed that high quality flour can be prepared by any of the five methods for use in the manufacture of paste and dough-based food products. Although storage studies were not conducted, it appears that flours made from solar-dried or oven-dried yams have excellent potential for making yam products available over a longer period throughout the year in developing tropical countries.

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# CORRELATION OF TIME-TEMPERATURE INDICATOR RESPONSE WITH MICROBIAL GROWTH IN PASTEURIZED MILK

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Accepted for Publication March 30, 1987

## ABSTRACT

*Commercially obtained pasteurized whole milk was stored at three constant temperatures (0°C, 5°C, and 10°C), and one variable temperature condition (cyclic exposure of 0°C for 14 days and 10°C for 2 days). Daily analyses were conducted to enumerate the growth of total bacteria, coliforms, psychrotrophs, and spore forming organisms in samples from each storage treatment. Microbial growth was correlated with the response of the I-POINT and LifeLine full-history time-temperature indicators. Response of the I-POINT model 2140 was strongly related to germination of the psychrotrophic bacteria, and significant correlations ( $r > 0.95$ ) were found between total count enumeration and the LifeLine model 57 indicator.*

## INTRODUCTION

Retailers in this country rely on the processor expiration date for quality maintenance of pasteurized milk. An expiration date is a valid quality assurance tool only if the product is held at specific constant temperature conditions during all phases of transport and handling. Pasteurized milk is usually stored between 4° and 10°C, however storage temperatures may be poorly controlled during distribution, and the resulting temperature fluctuations can have tremendous impact on the growth of spoilage bacteria. Much of the uncertainty related to the growth of bacteria in milk could be eliminated by monitoring the cumulative time and temperature exposure which a product receives during distribution.

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Commercially available time-temperature indicators provide a simple means to monitor cumulative time and temperature exposure. Review articles by Scheon and Byrne (1972) and Kramer and Farquhar (1976) provided information on patented and commercially developed indicators that monitor changes in temperature with time. There are two classifications of time-temperature indicators (Wells and Singh 1985). Devices which respond only after a predetermined threshold temperature has been exceeded are said to be "partial-history" indicators, whereas devices which respond to all temperature conditions encountered during storage are called "full-history" indicators. Storage study investigations conducted by Singh *et al.* (1984 and 1986) demonstrated the use of full-history time-temperature indicators as monitors of changes in specific attributes of food quality.

Mistry and Kosikowski (1983) investigated the use of indicators as quality control devices for fluid milk, and concluded that a combination of different models of the I-POINT time-temperature indicators could be used to predict milk spoilage. This work however did not examine the use of time-temperature indicators as monitors of the growth patterns of different microorganisms. The development of an electronic time-temperature indicator based on the growth rates of specific organisms observed in foods has discussed by Ratkowsky *et al.* (1982).

After examination of the Lifeline and I-POINT full-history time-temperature indicators, Wells and Singh (1986) concluded that these two kinds of indicators respond in a reproducible fashion over both extended periods of time, and wide ranges of temperature. Enzymatic or chemical reactions within each of these indicators proceed with time, and are accelerated with increased temperature. The enzymatic or chemical reactions either result in the formation of colored products or cause pH changes detected by a color indicator. In turn, the color changes are monitored either visually, with the aid of a color reference scale, or electronically, with an optical scanner. Thus, an indicator's color directly reflects the temperature history to which the indicator has been exposed.

The objective of this research was to evaluate the performance of two commercially available full-history time-temperature indicators when correlated with the microbial growth in pasteurized whole milk stored at refrigeration temperatures.

## MATERIAL AND METHODS

### Pasteurized Whole Milk

One pint cartons of homogenized whole milk obtained from the Crystal Cream and Dairy Company (3013 D Street, Sacramento, CA) were used in this in-



vestigation. The milk samples were pasteurized (76°C for 16.5 s) in the dairy, the evening before the beginning of the study, and were delivered to the university laboratory the following morning. Case lots of 36 milk cartons each, were randomly assigned to one of four treatment groups. Three treatment groups were placed into cold rooms strictly maintained at 0°, 5°, and 10°C for the duration of the study. The fourth treatment consisted of a variable temperature storage cycle of 14 days at 0°C, followed by 2 days storage at 10°C. Initial microbial analysis began six hours after samples were placed in cold storage.

### Microbial Testing Procedures

Each morning bacteria from two cartons were enumerated. Serial dilutions of milk were made in 0.1% Bactopeptone, and duplicate pour plates were prepared using four different media. Standard Methods Agar, incubated at 30°C for 48 h, was used to determine total counts. Coliforms were enumerated using Violet Red Bile Agar incubated at 37°C for 24 h. Psychrotrophic bacteria were detected by plating on Standard Methods Agar incubated at 7°C for 10 days. Spore counts were obtained by incubating the milk in Dextrose Tryptone Agar for 30 min at 80°C, plating, and further incubating the media at 30°C for 48 h. All counts are expressed as colony forming units (CFU) per mL milk.

### Time-Temperature Indicators

The I-POINT Time/Temperature Monitor (I-POINT Technologies, Malmo, Sweden) and the LifeLine Inventory Freshness Monitor (LifeLine Technologies, Morristown, NJ) were studied in this investigation. The LifeLine indicator contains an acetylenic monomer that changes color irreversibly as a result of polymerization. The color change is quantified with a hand-held microcomputer and an optical wand which indicates the decreases in light reflectance (100%-0%) as the indicator darkens. The I-POINT indicator contains a proprietary enzyme and substrate that undergo a hydrolysis reaction. As the hydrolysis reaction proceeds the solution pH changes, which in turn is exhibited as a gradual color change by a pH indicator dye. The color change is visually compared to a reference color scale which corresponds to four discrete increments (0, 1, 2, and 3).

Ten each of the I-POINT Time/Temperature Monitors (model 2140) and the LifeLine Inventory Freshness Monitors (model 57) were attached one per carton to representative milk cartons in each storage treatment (Fig. 1). Indicators were inspected within their respective storage room, at 3 and 4 day intervals. The cartons with indicators attached remained in storage until the conclusion of the study, and were not included in microbial sampling. At the end of the investigation, indicator response was correlated with changes in the selected microbial populations according to the procedure established by Singh *et al.* (1984).



FIG. 1. LIFELINE FRESHNESS MONITOR MODEL 57 (LEFT) AND I-POINT TIME/TEMPERATURE MONITOR MODEL 2140 (RIGHT) PLACED ON 1-PINT CARTONS OF PASTEURIZED MILK

## RESULTS AND DISCUSSION

### Effect of Temperature on Microbial Growth

Microbial growth profiles obtained at the four different treatment conditions are shown in Fig. 2a, 3a, 4a and 5a. The initial bacterial counts agree with recent reports of other investigators (Credit *et al.* 1972; Langeveld *et al.* 1972; Langeveld and Cuperus 1980). The keeping quality of the milk, as assessed by the time required to reach  $5 \times 10^6$  CFU/mL (Langeveld and Cuperus 1980), is indicative of a very low level contamination by psychrotrophic gram-negative rods. The CFU/mL exceeded  $5 \times 10^6$  only after 20 days at 5 °C and after 10 days at 10 °C. In contrast, Schroder *et al.* (1982) reported that milk experimentally contaminated with small numbers of gram-negative rods contained more than  $2 \times 10^6$  CFU/mL in three days at 5 °C and in as little as six days at 11 °C. These psychrotrophs are invariably post-pasteurization contaminants (Langeveld *et al.* 1972), and very few are needed to initiate spoilage (off-flavors) as the most effective competitors are destroyed by pasteurization (Dulshretha and Marth 1975).

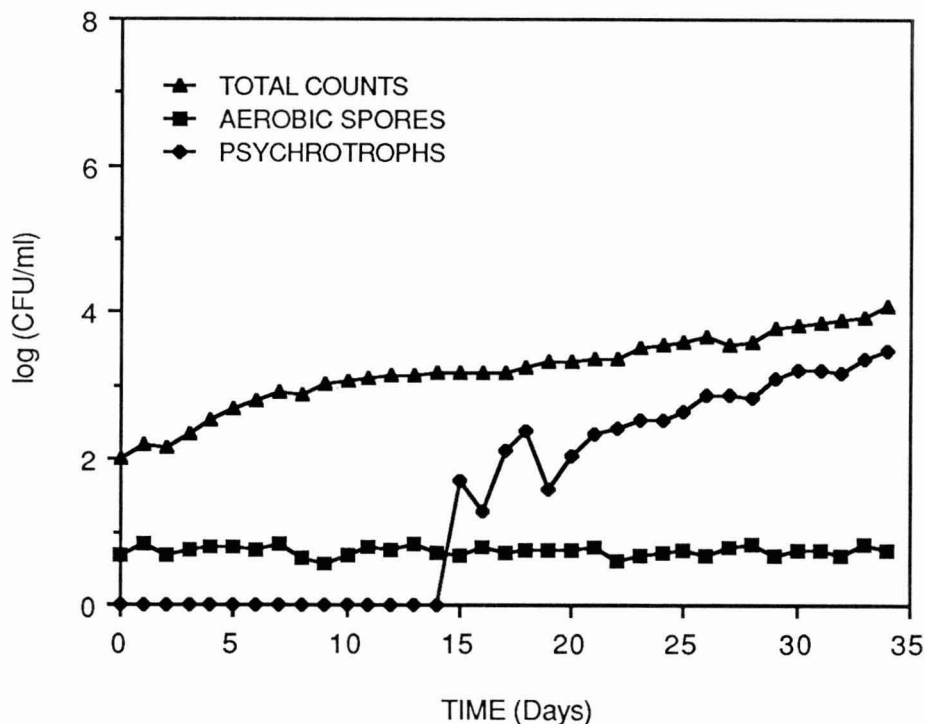


FIG. 2a. CHARACTERIZATION OF THE MICROBIAL GROWTH IN PASTEURIZED MILK FOR THE 0°C STORAGE TREATMENT

The microbial growth pattern observed in this investigation was more indicative of contamination by *Bacillus*, presumably spores that had survived pasteurization. At refrigeration temperatures, psychrotrophic *Bacillus* species grow much more slowly than do the commonly occurring psychrotrophic pseudomonads (Langeveld and Cuperus 1980). Psychrotrophic *Bacilli* contaminants are not unusual. Overcast and Atmaram (1974) reported that almost 30% of commercially pasteurized milk samples stored at 7°C were spoiled by *Bacilli cereus*. Credit *et al.* (1972) reported that milk experimentally contaminated with *Bacillus* spores which had survived pasteurization showed signs of spoilage only after 30 days at 4.5°C. In the present study, microscopic examination of bacteria constituting the predominant colony types of the total count population did, in fact, reveal large spore-containing gram-positive rods.

The seemingly contradictory finding that spore counts did not simultaneously increase is probably the result of inefficient sporulation in milk stored at low temperatures (Shehata and Collins 1972; Rodriguez and Barrett 1986). Exposure to a carbon or nitrogen limiting environment stimulates spore production in most

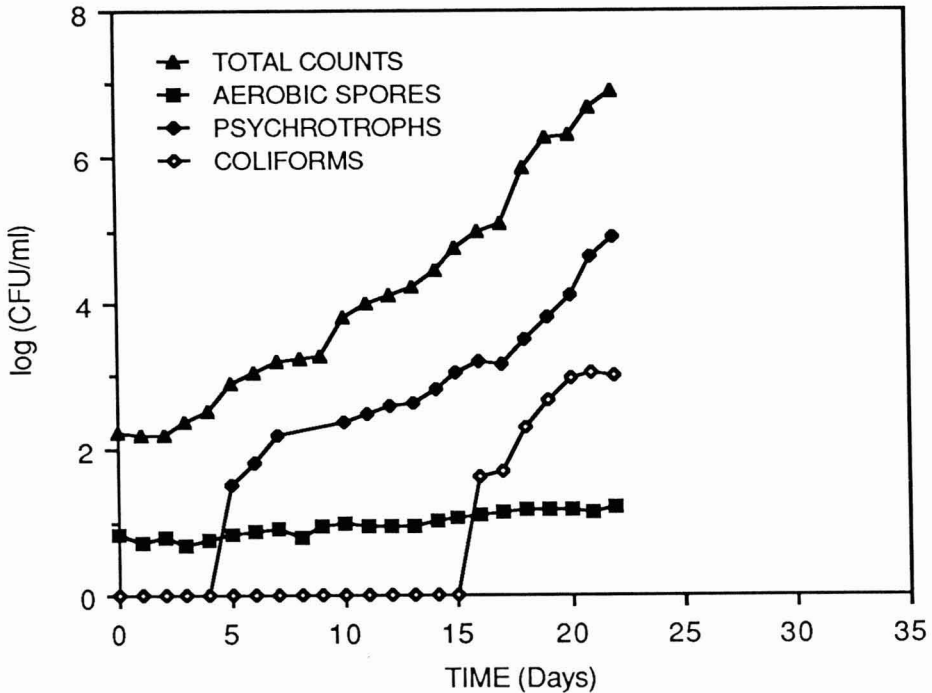


FIG. 3a. CHARACTERIZATION OF THE MICROBIAL GROWTH IN PASTEURIZED MILK FOR THE 5°C STORAGE TREATMENT

Bacilli strains (Piggot and Coote 1976). Thus, a rich medium such as milk usually will not provide the nutritional impetus for spore development. The temperature-dependent lag which preceded the psychrotroph growth may reflect poor germination at low temperatures. Chung and Cannon (1971) reported lag times of 8–14 days for the outgrowth of *Bacillus* spores in milk held at 5°C. Similarly, Mikolajcik and Simon (1978) showed that milk containing no detectable psychrotrophic Bacilli directly after pasteurization frequently contains high counts of these organisms after 2–4 weeks of storage at 7°C.

Dahlberg (1946) argued that coliform bacteria were the most significant class of common milk contaminants because of their ability to multiply rapidly, and their severe health threat. In the present study coliforms were detected in the 5°C samples, but failed to proliferate at both 0°C and 10°C. Coliform appearance at 5°C was preceded by a 15 day lag, which suggests the need for recovery from injury. The near-freezing incubation of the 0°C treatment probably inhibited coliform recovery (Dabbah and Moats 1968; Maxcy 1970), and the product stored at 10°C most likely spoiled by the predominant bacteria before coliforms were able to repair pasteurization damage.

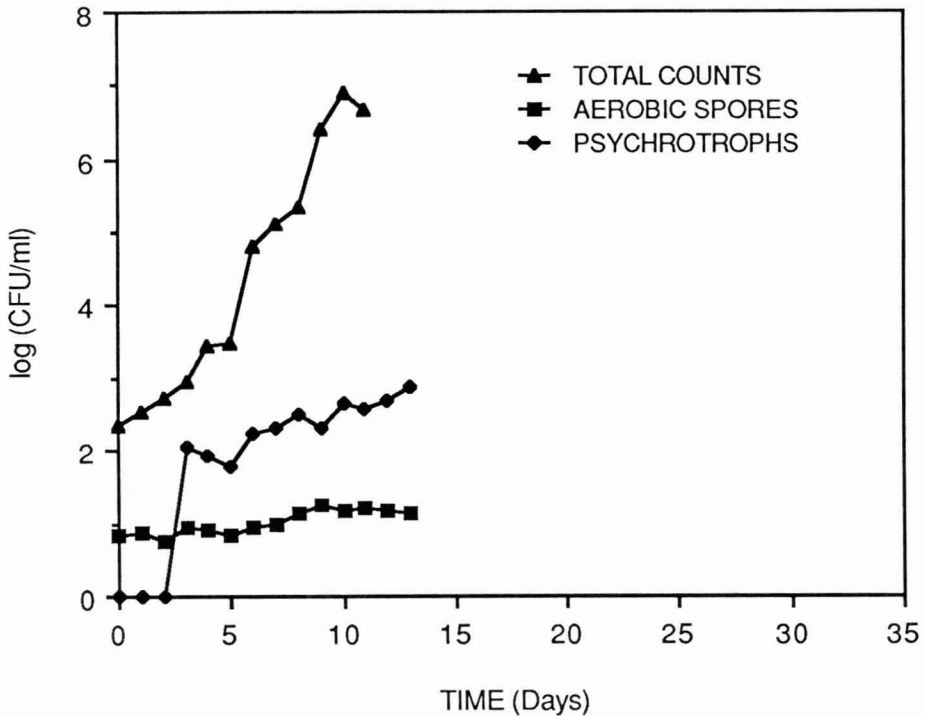


FIG. 4a. CHARACTERIZATION OF THE MICROBIAL GROWTH IN PASTEURIZED MILK FOR THE 10°C STORAGE TREATMENT

### Time-Temperature Indicator Correlations

Responses of the Lifeline and I-POINT time-temperature indicators observed at the four different treatment conditions are shown in Fig. 2b, 3b, 4b and 5b. For proper comparison between indicator response and microbial growth, both microbial growth and indicator response must change consistently with time, and be consistently correlated with each other at different storage temperatures. Table 1 shows the linear correlation coefficients and levels of significance between storage time, indicator responses, and microbial counts at each constant temperature treatment. Because of the extended lag period and the variation in growth of heat damaged coliforms, no correlations between coliform growth and indicator response were attempted. Correlations between indicator response and counts of aerobic spore formers were also inappropriate, as neither spore former growth nor indicator response were consistently correlated with storage time.

Changes in the total counts were the only data which proved to be significantly correlated to indicator response at all three constant temperature storage conditions. Correlations between the LifeLine model 57 response and total counts

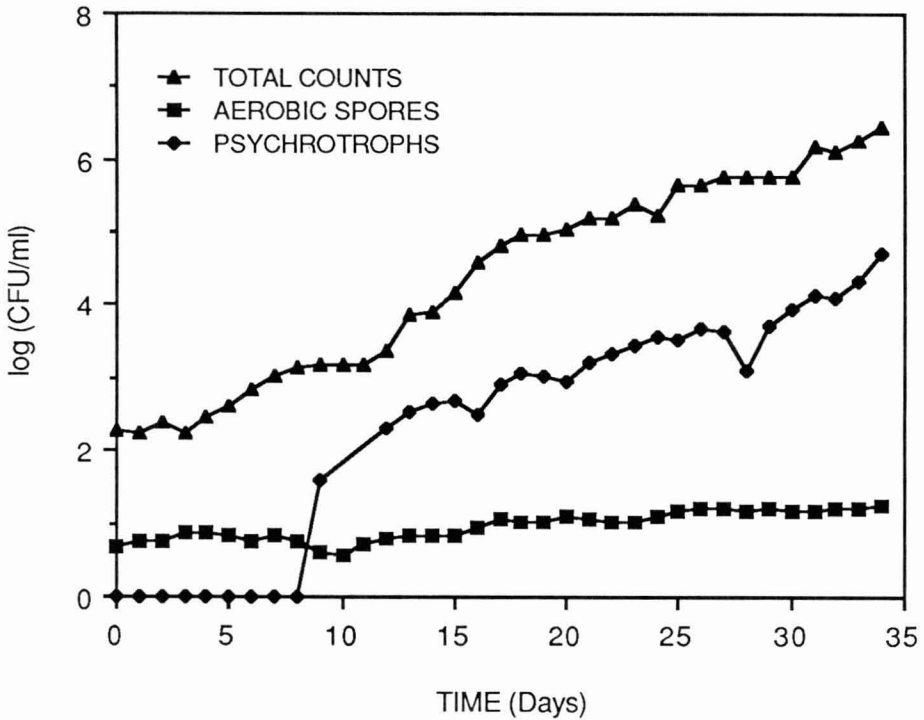


FIG. 5a. CHARACTERIZATION OF THE MICROBIAL GROWTH IN PASTEURIZED MILK FOR THE VARIABLE TEMPERATURE STORAGE TREATMENT

were significant ( $p < 0.05$ ) at  $10^{\circ}\text{C}$ , and were highly significant ( $p < 0.001$ ) at  $0^{\circ}\text{C}$  and  $5^{\circ}\text{C}$ . The slight decrease in correlation coefficient significance at  $10^{\circ}\text{C}$  was a result of fewer indicator inspections than at the other treatment conditions, thus reducing the number of degrees of freedom contributing to the calculation of the correlation coefficient.

Campbell *et al.* (1986) suggested that if consistent correlations exist at several isothermal storage conditions, regression analysis could be used to obtain a prediction equation to estimate food quality change based on indicator response. Figure 6 illustrates the correlation between total counts and LifeLine model 57 indicator response for the constant and variable temperature treatments. Regression analysis was used to construct 99% confidence limits between LifeLine 57 indicator response and normalized logarithm of total count CFU/mL collected at the constant temperature storage. Indicator response and total count data obtained from the variable treatment fall within the confidence limits of the prediction equation. This demonstrates the validity of the regression approach in estimating food quality changes from indicator response.

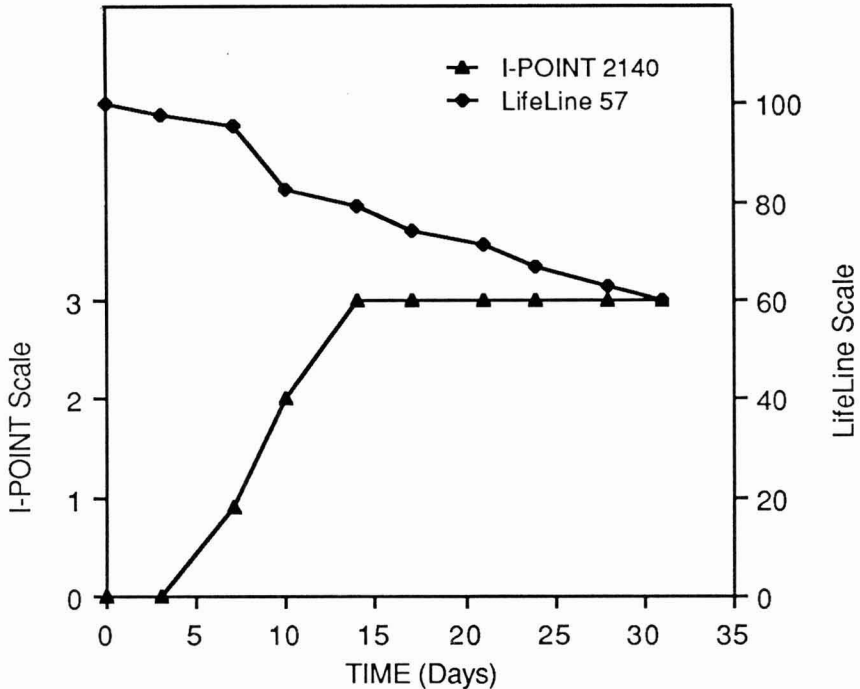


FIG. 2b. RESPONSE OF THE LIFELINE 57 AND I-POINT 2140 TIME-TEMPERATURE INDICATORS DURING THE 0°C STORAGE TREATMENT

Although psychrotrophic counts were not consistently correlated with indicator response, the length of the lag periods prior to the appearance of psychrotrophic growth might be an important determinant of shelf-life. The time required for injury recovery or spore germination is dependent on the storage temperature. Likewise, the I-POINT indicators initiate a discernible color change (the indicator response) only after a predetermined time and temperature combination has been achieved (Blixt and Tiru 1976). Thus, I-POINT indicators exhibit a time-temperature dependent lag prior to initiating a color change, much like the time-temperature dependent lag observed in the psychrotrophic bacteria counts. Figure 7 illustrates the relation between response lag of I-POINT model 2140 and the lag in psychrotrophic growth for all treatments. Because the response of the I-POINT model 2140 indicator also mimicked the delay in the growth lag in the variable treatment condition, it would appear that the I-POINT indicator might be particularly useful in situations where microbial spoilage in milk are typically the result of contamination by psychrotrophic *Bacillus* species.

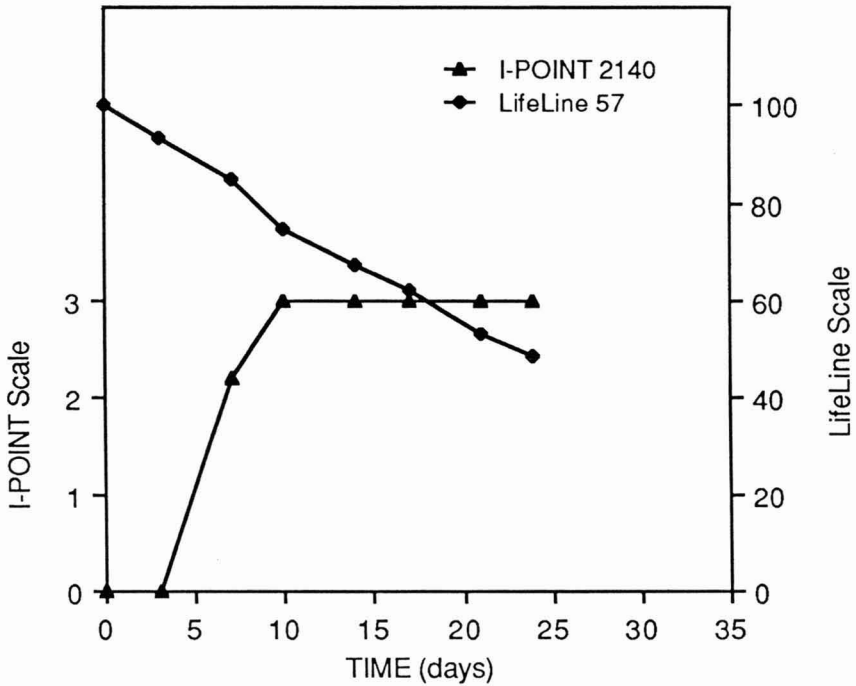


FIG. 3b. RESPONSE OF THE LIFELINE 57 AND I-POINT 2140 TIME-TEMPERATURE INDICATORS DURING THE 5°C STORAGE TREATMENT

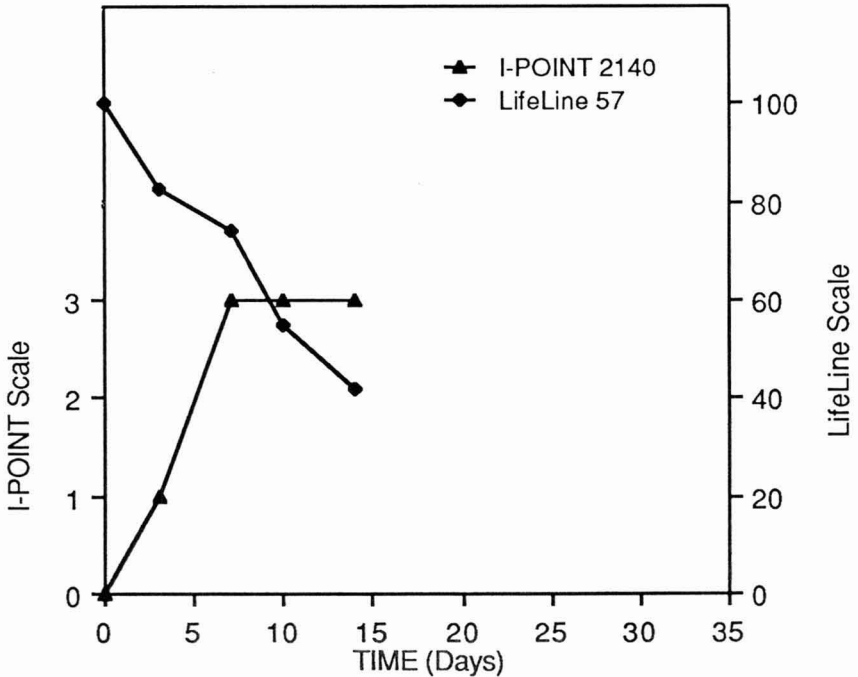


FIG. 4b. RESPONSE OF THE LIFELIFE 57 AND I-POINT 2140 TIME-TEMPERATURE INDICATORS DURING THE 10°C STORAGE TREATMENT



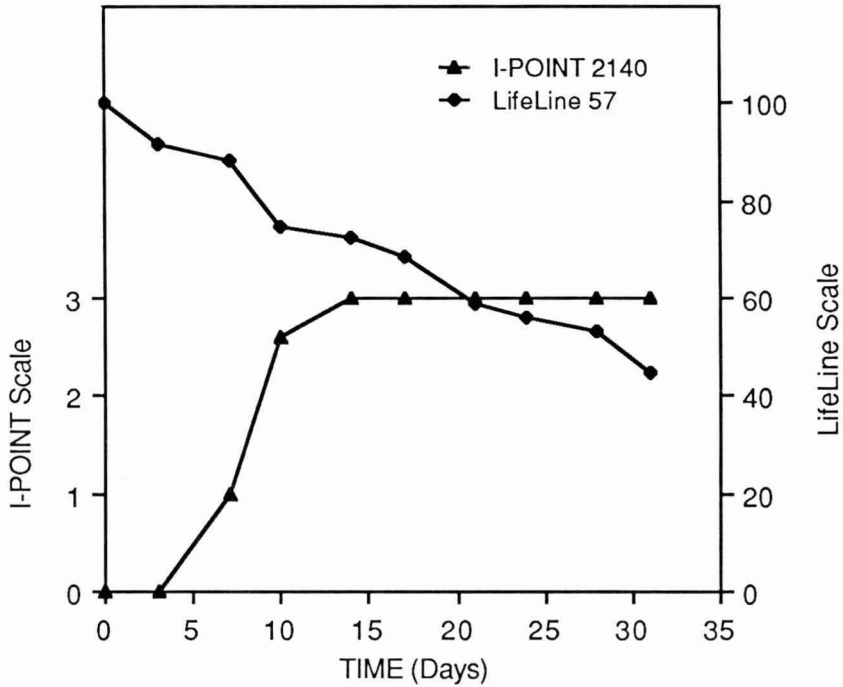


FIG. 5b. RESPONSE OF THE LIFELINE 57 and I-POINT 2140 TIME-TEMPERATURE INDICATORS DURING THE VARIABLE TEMPERATURE STORAGE TREATMENT

Results of this investigation illustrate the potential use for time-temperature indicators in estimating microbial growth in pasteurized whole milk. A single time-temperature indicator may not account for the differences in the quality and type of contaminants in milk from different processors; nor can a single indicator describe the complexity of microbial growth patterns arising from mixed populations incubated at changing temperatures. However, a combination of the different types of full-history time-temperature indicators could be used to estimate the presence and growth of spoilage organism within pastuerized milk.

TABLE 1.  
CORRELATION COEFFICIENTS AND SIGNIFICANCE LEVELS OF CORRELATIONS  
BETWEEN STORAGE TIME, TIME-TEMPERATURE INDICATOR RESPONSE, AND  
SELECTED MICROBIAL COUNTS AT EACH CONSTANT TEMPERATURE TREATMENT

		Correlation Coefficients and Significance Levels					
		Total Counts		Spore Formers		Psychrotrophics	
I-POINT TTM	Time	0.951 <sup>a</sup>	***	0.308	n.s.	0.923	***
		0.981 <sup>b</sup>	***	0.918	**	0.965	***
		0.985 <sup>c</sup>	*	0.948	n.s.	0.875	n.s.
	2140	0.905	***	0.094	n.s.	0.722	*
		0.794	*	0.910	**	0.908	**
		0.926	n.s.	0.825	n.s.	0.871	n.s.
	2180	0.828	**	0.295	n.s.	0.943	***
		0.890	**	0.901	**	0.848	*
		0.965	*	0.823	n.s.	0.715	n.s.
	2220	0.841	*	0.291	n.s.	0.951	***
		0.935	**	0.890	**	0.865	*
		0.989	*	0.921	n.s.	0.686	n.s.
2340	0.680	*	0.244	n.s.	0.755	*	
	0.871	*	0.663	n.s.	0.751	n.s.	
	0.823	n.s.	0.888	n.s.	0.498	n.s.	
LifeLine	57	-0.940	***	-0.171	n.s.	-0.897	***
		-0.973	***	-0.926	**	-0.963	***
		-0.962	*	-0.988	*	-0.893	n.s.

a correlation coefficient for 0°C storage treatment

b correlation coefficient for 5°C storage treatment

c correlation coefficient for 10°C storage treatment

Levels of significance designated as

\* p < 0.05

\*\* p < 0.01

\*\*\* p < 0.001

n.s. not significant

p > 0.05

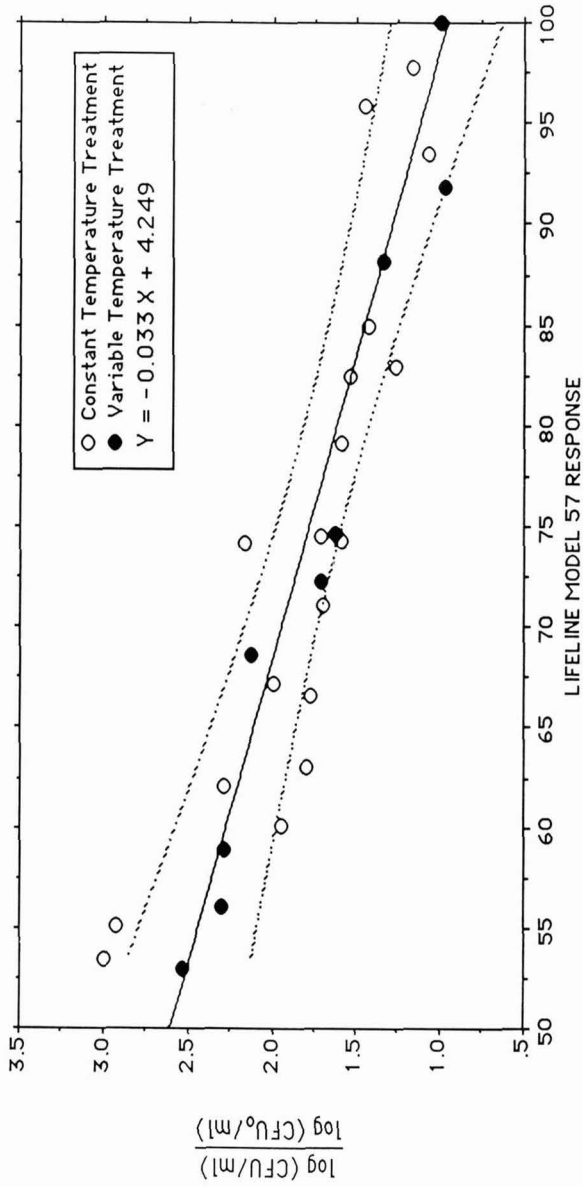


FIG. 6. PLOT OF NORMALIZED TOTAL COUNT ENUMERATION AND RESPONSE OF LIFELINE INDICATOR MODEL 57 FOR CONSTANT AND VARIABLE TEMPERATURE TREATMENTS

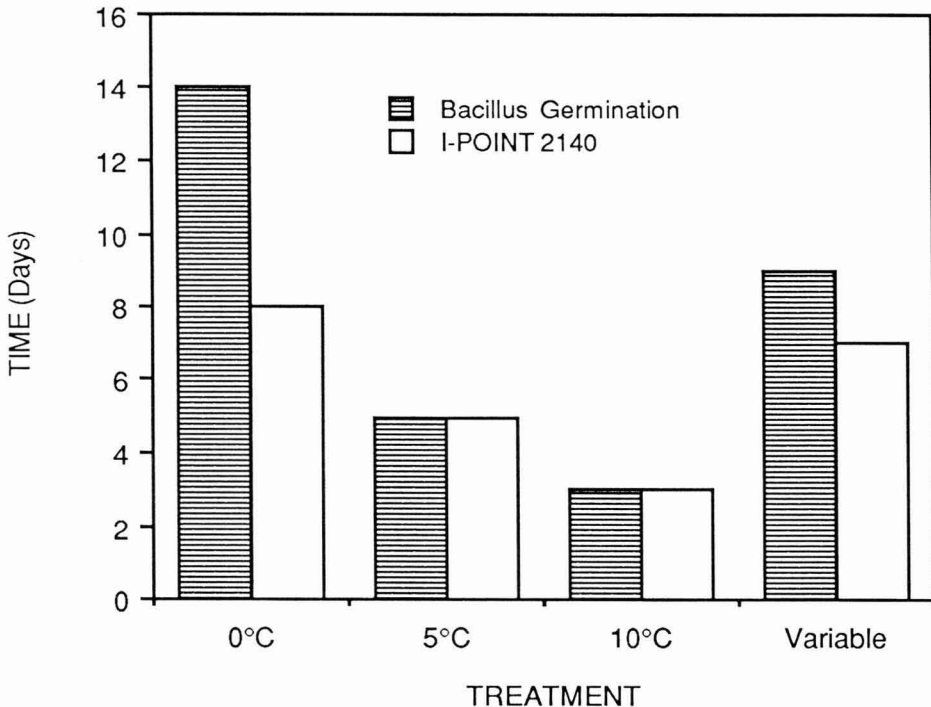


FIG. 7. COMPARISON OF THE LENGTH OF TIME TO OBSERVE A DISCERNIBLE COLOR CHANGE FROM 0 TO 1 (GREEN TO YELLOW) IN THE I-POINT MODEL 2140 AND DETECTION OF THE PRESENCE OF PSYCHROTROPHIC BACTERIA IN PASTEURIZED MILK

## CONCLUSIONS

(1) Full-history time-temperature indicators which exhibit a delay prior to initiating a discernible response (e.g. the I-POINT type indicator) are suitable for estimating the time delay prior to psychrotrophic Bacilli spore germination in pasteurized milk.

(2) Full-history time-temperature indicators which respond in a continuous fashion (e.g. the LifeLine type indicator) are suitable for estimating growth of the total microbial population in pasteurized milk.

## ACKNOWLEDGMENTS

This research was supported by U.S. Army Contract No. DAAK60-84-C-0076. The authors greatly appreciated the technical assistance provided by Mr. Satbir S. Nijjar and Mr. Hongda Chen. Mention of manufacturer names and particular models in this article should not be considered an endorsement.

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# EFFECTS OF PHOSPHATE TYPE, PACKAGING METHOD AND STORAGE TIME ON THE CHARACTERISTICS OF CHINESE SAUSAGE<sup>1</sup>

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Accepted for Publication May 19, 1987

## ABSTRACT

*The effects of phosphate type, packaging method and storage time on pH, moisture, water-holding capacity, TBA values, residual nitrite and total aerobic and anaerobic plate counts on Chinese sausage were determined. Sausage containing a blended phosphate had the highest water holding capacity. The pH and residual nitrite levels of Chinese sausage were higher ( $P < 0.05$ ) in the group containing tetrasodium pyrophosphate, than the other phosphate treatments. Sausages containing sodium hexametaphosphate had the lowest pH ( $P < 0.05$ ), the least amount of residual nitrite and the highest TBA values. Chinese sausage which was vacuum packaged had lower ( $P < 0.05$ ) TBA values than nonvacuum packaged sausage during 4–6 weeks of storage. No major microbial problems were observed in any of the samples during 6 weeks of storage.*

## INTRODUCTION

Approximately 22% of fresh pork produced in Taiwan is consumed as processed or cured meat products (TMDF, 1985) and these include Chinese sausage, dried pork, fried shredded pork or Chinese bacon. Chinese sausage is

<sup>1</sup>Portions of salaries and research support provided by State and Federal Funds appropriated to the Ohio Agricultural Research and Development Center, The Ohio State University. Research conducted in Taiwan, edited in Ohio, Journal Article No. 53-86.

one of the most popular of these Chinese processed meat foods and/or snacks in Taiwan, China and the Chinese communities in Asia and the United States.

Most western style sausages are prepared from ground and/or comminuted and seasoned meat which is formed into a symmetrical shape; however, Chinese sausage is not comminuted in the same manner as western style sausages. Raw materials for Chinese sausage are primarily lean pork and pork back fat. Beef and poultry meat are not usually added. Only the lean pork portion is coarsely ground, while the back fat is cut into cubes manually or by machine. These sausages are stuffed into small hog or lamb intestines. Chinese sausage is normally dried and/or smoked during manufacture until it shrinks (loses weight) to approximately 65% to 70% of its green weight. The finished product contains 30% to 45% moisture and 20 to 40% fat. This product may be classified (Kuo *et al.* 1986) as a semi dried sausage which could be deep fat fried, pan fried or steamed before serving.

Phosphates are known to increase water-holding capacity, stabilize meat emulsions, improve juiciness and tenderness, and maintain flavor of processed meat products (Ellinger 1972). Mahon *et al.* (1970) reported that sodium triphosphate (STPP) was the most widely used and the best phosphate for meat products; however, STPP is sometimes blended with other phosphates to produce even superior effects in meat product, particularly when sodium hexametaphosphate (SHMP) is used in the blend. This is probably due to the synergistic effects of phosphates. Shults *et al.* (1972) also reported that a blended phosphate including tetrasodium pyrophosphate (TSPP) and SHMP could increase some of the functional properties of meat products. Phosphates have been added to Chinese sausage to improve texture, prevent fat oxidation, inhibit microbial growth and retain moisture during storage (Kuo *et al.* 1986). There are many different phosphates used in this type of product, yet, which one produces better effects in Chinese meat type products is not known.

Traditionally, after the manufacture, the nonpackaged sausages are hung in a cool area having adequate air circulation. This storage method not only accelerates the moisture loss and fat oxidation during storage, but also increases the opportunities for the sausages to become contaminated with microorganisms and insects. A better packaging method for this product should be developed and investigated.

The purposes of this research were to determine the effects of four added phosphates (STPP, TSPP, SHMP and blended phosphate), two packaging methods (vacuum and nonvacuum) and several storage times (0, 2, 4 and 6 weeks) on pH, moisture, water-holding capacity, TBA values, residual nitrite, total aerobic plate counts and total anaerobic plate counts of a Chinese sausage.



## MATERIALS AND METHODS

### Sample Preparation

Hot-boned hams (most of the fresh meat sold in Taiwan is hot-boned) and pork back fat were purchased at approximately 4 h after slaughtering from a local (Taiwan) retail store. All visible fat and connective tissue was removed from the hams. Lean meat from the hams was ground through a 1.0 cm grinder kidney plate. The pork back fat was cut manually into cubes (0.5 cm<sup>3</sup>). The meat block was composed of 7 parts of coarse ground lean meat and 3 parts of cubed fat by weight. Lean meat and fat were thoroughly blended in a mixer and then divided into 4 groups (See Fig. 1) with each batch containing approximately 25 kg of the meat block. To Group 1 was added 0.2% tetrasodium pyrophosphate (TSPP); to Group 2 was added 0.2% sodium tripolyphosphate (STPP); to Group 3 was added 0.2% sodium hexametaphosphate (SHMP) and to Group 4 was added 0.2% blended phosphate (STPP:SHMP:TSP = 6:3:1). All phosphates were solubilized in approximately 100 mL of water prior to addition. Except for phosphate, other curing ingredients added to each group were the same and consisted of sugar (7.3%), sodium chloride (1.3%), monosodium glutamate (1.3%), potassium sorbate (0.2%) and sodium nitrite (0.012%). Percentage of phosphate and other curing ingredients was based on the weight of the meat block. All curing ingredients were added to the meat block and mixed thoroughly at room temperature for 5 min and then cured at 4 °C for 12 h. This cured meat was then stuffed into natural hog casings and linked into 10 cm units. Casings were pricked generously on all sides of the sausage by a needle to allow air to escape from the casing. The sausage was then dried in a forced air oven (R.H. = 25%) at 50 °C for 8–10 h or until it reached approximately 70–75% of the original weight. Finished sausages from each treatment were randomly assigned to vacuum or nonvacuum packaging (NY/PE/EVA/ = 15/30/30 mil) and vacuum sealed (684 mm Hg) or sealed in a multivac (A 300/12) packaging machine. Eight sausages (approximately 400 g) were placed in one packaging bag (Sun A Enterprise Co., LTD, Taiwan). Four packages from each treatment were randomly assigned to storage periods of 0, 2, 4 and 6 weeks at 4 °C. This experiment was replicated 3 times.

### Microbiological Evaluation and pH Determination

After each storage interval, a package of sausage samples (400 g) from each treatment were ground and mixed through a grinder which had been cleaned and sterilized with 75% ethyl alcohol. A 10 g ground sample was then aseptically removed and mixed with 90 mL of sterilized distilled water in an aseptic blender for 2 min.

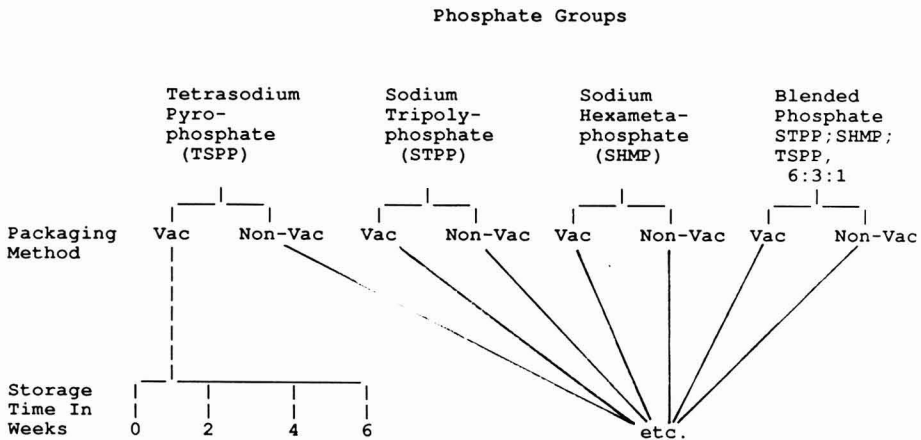


FIG. 1. EXPERIMENTAL DESIGN

Total aerobic plate counts were enumerated by using Tryptone Glucose Extract Agar (DIFCO) and the plates (containing  $1:10^3$ ,  $1:10^4$ , and  $1:10^5$  dilutions) were incubated at  $37^\circ\text{C}$  for 48 h. Total anaerobic plate count was determined by using Anaerobic Agar (DIFCO) and the plates (containing the same dilutions as total aerobic plate counts) were incubated at  $25^\circ\text{C}$  for 72 h in an anaerobic jar flushed with nitrogen. The same solutions used for microbial determination also was used to determine the pH value of the sausage with a pHM82 standard pH meter (Radiometer, Copenhagen, Denmark).

### Moisture Content and Water Holding Capacity

Moisture was determined by oven drying according to AOAC (1980) procedures. The water holding capacity (WHC) was measured by the press method (Ockerman 1985) and expressed as percent of free water. The meat sample (0.5 g) was placed on a filter paper and pressed between two plexiglass sheets with the Casver Lab Press at 500 psi for one minute. The inner and outer surface of pressed meat and juice was measured ( $\text{in}^2$ ) with a polar planimeter.

$$\text{Free water (\%)} = \frac{(\text{total surface area} - \text{meat film area})(61.10)}{\text{total moisture (mg) of meat sample}} \times 100$$

### Residual Nitrite

Residual nitrite of each package of sausage was determined according to the AOAC (1980) procedures.

### TBA Values

The development of oxidative rancidity in the sausage was measured using the 2-thiobarbituric acid (TBA) analysis according to Tarladgis *et al.* (1960) as described by Ockerman (1985). TBA procedures were modified by mixing 10 g of blended meat with 49 g of 50 °C distilled water and 1 mL of sulfanilamide reagent [0.5% sulfanilamide in 20% HCl (v/v)]. Sulfanilamide forms a diazonium salt with nitrite and prevents it from interfering with the test. Results were expressed as mg malonaldehyde per kg of sausage.

### Statistical Analysis

Data were analyzed by the analysis of variance procedures (BMDP 1981). Individual F-test was used to determine the significance of phosphates types, packaging methods and storage times, and the interaction effects. Means were separated by the LSD (Least Significant Difference) technique.

## RESULTS AND DISCUSSION

Alkaline phosphates have been reported to increase the pH of meat and meat products (Ellinger 1972; Ockerman 1980); however, the increase in pH resulting from addition of different phosphates varied. In this current research, the analysis of variance indicated that sausage pH was significantly affected by type of phosphate added. Sausages which contained TSPP had the highest pH (6.60) among these four phosphate groups, followed by STPP group (pH = 6.53), blended phosphate group (pH = 6.51) and SHMP group (pH = 6.41). The differences among these four groups were significant ( $P < 0.05$ ) except between STPP and the blended phosphate groups. Due to the natural characteristic of the high alkalinity of TSPP (Ellinger 1972), Chinese sausage containing the TSPP had a higher ( $P < 0.05$ ) pH than the other three groups. This agreed with the work reported by Ockerman (1980) who found that meat products containing 0.5% TSPP had higher pH than meat products containing the same level of STPP or SHMP. Molins *et al.* (1985) studied the pH of cooked, vacuum packed bratwurst and found that meat product containing 0.5% TSPP had a higher pH than bratwurst containing 0.5% STPP. Trout and Schmidt (1984) also reported that the pH of uncooked beef roll containing TSPP has the highest pH, followed by the STPP and SHMP groups.

Table 1 shows the moisture content of Chinese sausage which was affected ( $P < 0.05$ ) by packaging method and added phosphate. During storage, the sausage which was vacuum packaged (effect of storage time absorbed) had a higher ( $P < 0.05$ ) moisture content than the sausage with nonvacuum packaging.

TABLE 1.  
MOISTURE (%) OF CHINESE SAUSAGE AS AFFECTED BY PACKAGING METHOD  
AND ADDED PHOSPHATE

Phosphate <sup>a</sup>	Moisture content (%)		
	Packaging method		Mean*
	Vacuum	Non-vacuum	
TSPP	36.7	35.3	36.0 <sup>c,d</sup>
STPP	37.6	36.2	36.9 <sup>b</sup>
SHMP	36.5	36.5	36.5 <sup>b,c</sup>
Blended Phosphate	36.3	34.7	35.5 <sup>d</sup>
Means**	36.8 <sup>x</sup>	35.7 <sup>y</sup>	

<sup>a</sup>TSPP = tetrasodium pyrophosphate; STPP = sodium tripolyphosphate; SHMP = sodium hexametaphosphate; Blended phosphate = STPP:SHMP:TSPP (6:3:1).

\*Data on means within a column with different letters (b,c,d) are significantly different ( $P < 0.05$ ).

\*\*Data on means within a row with different letters (x,y) are significantly different ( $P < 0.05$ ).

Especially for the group containing blended phosphate, the differences in moisture content between vacuum and nonvacuum packaged sausages were much greater than the other groups. This seemed to indicate that Chinese sausage with added blended phosphate should be vacuum packaged in order to retain more moisture during storage. Ockerman and Kuo (1982) reported that vacuum packaged Chinese dried pork products retained more moisture during storage which should cause the tissue to maintain a greater tenderness than non-vacuum packaged samples.

The moisture content of Chinese sausage which added STPP was higher than that of the sausage containing TSPP, SHMP, or blended phosphate. However, the differences among these four groups were not all significant (Table 1). Kuo *et al.* indicated that the texture of Chinese sausage would be affected by its moisture content.

Table 2 shows the water holding capacity (WHC), expressed as percentage of free water, for Chinese sausage in different treatment groups. The higher the percentage of free water released, the lower the WHC of the sausage. Regardless of packaging method, Chinese sausage containing blended phosphate had a higher (although not always significant) WHC than that of the other three groups, as expressed by a lower percentage of free water released. Both in vacuum and nonvacuum packaging groups, the WHC for Chinese sausage

among TSPP, STPP, and SHMP were not significantly different ( $P > 0.05$ ). The differences in WHC between vacuum and nonvacuum packaged sausages were not significant, except for samples containing STPP. This phenomenon is hard to explain. It is well known that the pH value and WHC of meat products are closely related (Forrest *et al.* 1975; Kuo and Ockerman 1984). In the previous discussion, the pH of the blended phosphate group was not the highest among these four groups; however, the blended phosphate group, both in vacuum and nonvacuum treatments, was found to have a higher WHC than that of the other 3 groups. This phenomenon is probably due to the synergistic effect of blended phosphate (STPP:SHMP:TSPP = 6:3:1) which could increase the WHC of sausage more effectively. Mahon *et al.* (1970) and Shults *et al.* (1972) also reported that the blended phosphate including TSPP and SHMP could increase some of the functional properties of meat products.

TABLE 2.  
WATER HOLDING CAPACITY (WHC) OF CHINESE SAUSAGE AS AFFECTED BY ADDED PHOSPHATE AND PACKAGING METHOD

Phosphate <sup>a</sup>	WHC (% free water)*	
	Vacuum	Non-vacuum
TSPP	30.18 <sup>b,x</sup>	31.00 <sup>b,x</sup>
STPP	25.10 <sup>b,c,y</sup>	35.35 <sup>b,x</sup>
SHMP	30.07 <sup>b,x</sup>	25.23 <sup>b,c,x</sup>
Blended Phosphate	21.18 <sup>c,x</sup>	20.18 <sup>c,x</sup>

<sup>a</sup>TSPP = tetrasodium pyrophosphate; STPP = sodium tripolyphosphate; SHMP = sodium hexametaphosphate; Blended phosphate = STPP: SHMP:TSPP (6:3:1).

\*Data within columns with different letters (b,c) are significantly different ( $P < 0.05$ ). Data within rows with different letters (x,y) are significantly different ( $P < 0.05$ ).

The amount of residual nitrite in Chinese sausage was affected ( $P > 0.05$ ) by storage time and added phosphate (Table 3). In these four groups, the amount of residual nitrite in Chinese sausage, in general, decreased with increased storage time. However, the difference in amount of residual nitrite was only significant between 0 and 2 weeks of storage. This agreed with the study done by Ockerman and Kuo (1982) who found that the amount of residual nitrite of dried pork decreased with storage time (16 weeks). Cassens *et al.* (1974) pointed out that the residual nitrite of meat products might be oxidized to  $\text{NaNO}_3$  or  $\text{HNO}_3$  or reduced to NO (Kuo and Ockerman 1983). The Chinese sausage containing TSPP had the highest ( $P < 0.05$ ) amount of nitrite residue among these four

TABLE 3.  
RESIDUAL NITRITE (PPM) OF CHINESE SAUSAGE AS AFFECTED BY ADDED  
PHOSPHATES AND STORAGE TIME

Weeks	Phosphates <sup>a</sup>				Means <sup>*</sup>
	TSPP	STPP	SHMP	Blended Phosphates	
0	59.0	52.1	39.5	44.8	48.9 <sup>b</sup>
2	28.0	19.1	18.3	22.6	22.0 <sup>c</sup>
4	23.3	17.9	17.4	20.0	19.6 <sup>c</sup>
6	18.9	15.9	16.6	18.8	17.6 <sup>c</sup>
Means <sup>**</sup>	32.3 <sup>x</sup>	26.3 <sup>y</sup>	22.9 <sup>z</sup>	26.6 <sup>y</sup>	

<sup>a</sup>TSPP = tetrasodium pyrophosphate; STPP = sodium tripolyphosphate; SHMP = sodium hexametaphosphate; Blended phosphates = STPP:SHMP:TSPP = (6:3:1).

<sup>\*</sup>Data on means within a column with different letters (b,c) are significantly different ( $P < 0.05$ ).

<sup>\*\*</sup>Data on means within a row with different letters (x,y,z) are significantly different ( $P < 0.05$ ).

phosphate groups, followed by the blended phosphate and STPP groups which were similar, and then the SHMP group. This nitrite retention was probably due to the influence of pH of meat products. Cahill *et al.* (1972) and Ockerman (1980) indicated that the reaction rate of nitrite converting to other chemical substances such as NO was faster at lower pH conditions. In previous discussion, Chinese sausage with added TSPP had the highest pH (the reduction rate of nitrite was slower) and the largest amount of residual nitrite. Likewise, the group containing SHMP has the lowest pH (6.4) and the least ( $P < 0.05$ ) amount of residual nitrite among these four phosphate groups. The difference in amount of residual nitrite between the groups containing blended phosphate and STPP were not significant. From these data, it suggested that pH and the amount of residual nitrite in Chinese sausage were affected by added phosphate.

TBA values of Chinese sausage were affected significantly ( $P < 0.05$ ) by packaging method and storage time. With vacuum packaging, the TBA values (oxidative rancidity) of sausage did not change significantly ( $P > 0.05$ ) during 6 weeks of storage (Table 4). This indicated that vacuum packaging could be used to retard oxidative rancidity of this type of sausage. TBA values of nonvacuum packaged sausage increased significantly by 4 weeks of storage. However, TBA values decreased slightly after 4 weeks of storage. This is probably due to the decomposition of intermediates of oxidation which were formed during storage, thus, lowering the malonaldehyde extracted and decreasing the TBA value of this sausage.

TABLE 4.  
TBA VALUES<sup>a</sup> OF CHINESE SAUSAGE AS AFFECTED BY PACKAGING METHODS  
AND STORAGE TIME

Weeks	Packaging Methods <sup>*</sup>	
	Vacuum	Non-vacuum
0	0.35 <sup>b,x</sup>	0.37 <sup>c,x</sup>
2	0.31 <sup>b,x</sup>	0.32 <sup>c,x</sup>
4	0.28 <sup>b,y</sup>	1.60 <sup>b,x</sup>
6	0.25 <sup>b,y</sup>	1.34 <sup>b,x</sup>

<sup>a</sup>TBA value is expressed as mg malonaldehyde per Kg of Chinese sausage.

<sup>\*</sup>Data within rows with different letters (x,y) are significantly different ( $P < 0.05$ ). Data within columns with different letters (b,c) are significantly different ( $P < 0.05$ ).

After 0 and 2 weeks of storage, the TBA values of Chinese sausage between vacuum and nonvacuum packaged groups were not significantly different. However, at 4 and 6 weeks of storage, the differences in TBA values between these two groups became significantly different. This indicated that the Chinese sausage should be vacuum packaged if stored more than 2 weeks to prevent lipid oxidation. However, if this meat product was consumed within 2 weeks, then either vacuum packaging or nonvacuum packaging could be used without associated major rancidity problems. This finding agrees with Ockerman and Kuo (1982) who studied Chinese dried pork and reported that vacuum packaged dried prok had lower TBA values than nonvacuum packaged dried pork during 4–16 weeks of storage. Matlock *et al.* (1984) studied raw-frozen sausage patties and reported that the addition of phosphates or vacuum packaging both exhibited an antioxidant effect during 8 weeks of storage.

The average TBA values (pooled means over packaging methods and storage time) were also significantly ( $P < 0.05$ ) affected by type of added phosphate (Fig. 2). The sausage containing SHMP had the highest ( $P < 0.05$ ) TBA value (0.96) among these four groups and sausage containing TSPP had the lowest TBA values (0.33). The TBA values of STPP group and blended phosphate group were 0.58 and 0.54, respectively, and were not significantly different. The sausage containing SHMP had the lowest pH value (6.41) and the highest TBA value (0.96); and the TSPP group had the highest pH (6.60) and the lowest TBA value (0.33) among these four phosphate groups. This probably indicated that the lower pH value, the higher was sausage TBA value. Judge and Aberle (1980) and Dreup, *et al.* (1981) also reported that TBA values of fresh pork sausage are higher in the groups with lower pH values. Another reason for the

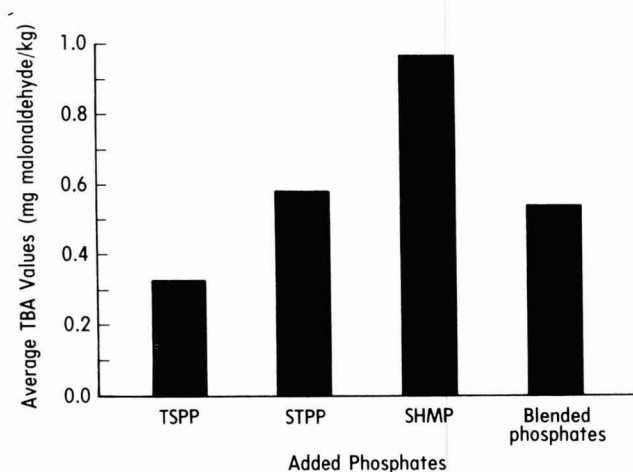


FIG. 2. EFFECT OF PHOSPHATE ON TBA VALUES OF CHINESE SAUSAGE

SHMP group having a higher TBA value may have been due to its lower amount of residual nitrite (which is usually considered an antioxidant). Nitrites have been reported to retard the rate of oxidative rancidity (TBA values) in pork products (Hadden *et al.* 1975; Freeman *et al.* 1982; and Ockerman and Kuo 1982). The SHMP group with the least amount of residual nitrite (Table 3) was also shown to have the highest ( $P < 0.05$ ) TBA values (more rancid) among these four phosphate groups.

The growth curves (log 10) of total aerobic bacteria in the four groups containing different phosphates, generally, were very similar (Fig. 3). Total aerobic plate counts at 2 weeks of storage were the highest ( $P < 0.05$ ) during storage periods (0, 2, 4 and 6 weeks).

During the 6 weeks of storage the group containing TSP had higher ( $P < 0.05$ ) aerobic plate counts than the other three phosphate groups. This probably resulted from the higher pH of TSP group. Aerobic bacteria are known to multiply more rapidly in an environment with a higher pH value (Banwart 1979). During the same storage period, the group containing SHMP had the lowest pH and contained the least (not always significant) total aerobic plate counts among these four phosphate groups. However, Molins *et al.* (1985) reported that the addition of STP, TSP, SAPP (sodium acid pyrophosphate) or SPG (sodium polyphosphate glassy) at a level of 0.5% to vacuum packaged cooked bratwurst has no significant effect on bacterial growth (total mesophilic and total anaerobic plate counts) during refrigerated storage for 7 days.

In all samples, the total anaerobic plate counts (log 10) at 2 and 4 weeks of storage were higher than those at the 0 and 6 weeks of storage (Fig. 4). At 0 and 4 weeks of storage, the group containing TSP has slightly higher anaerobic



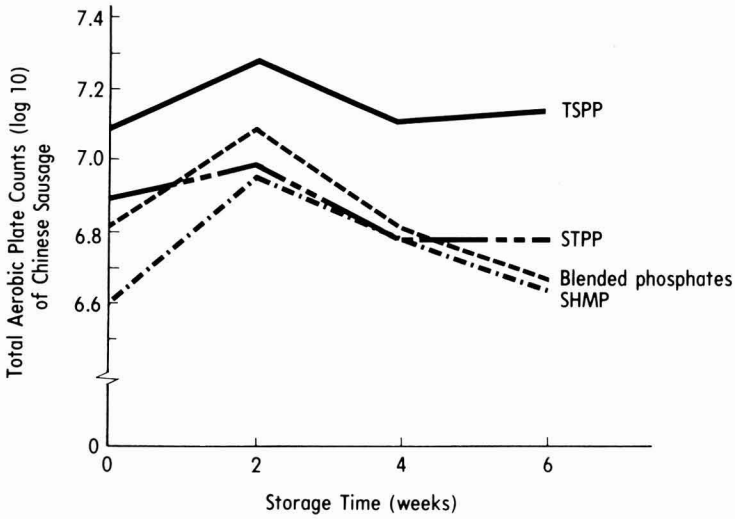


FIG. 3. EFFECT OF PHOSPHATE AND STORAGE TIME ON THE TOTAL AEROBIC PLATE COUNTS OF CHINESE SAUSAGE

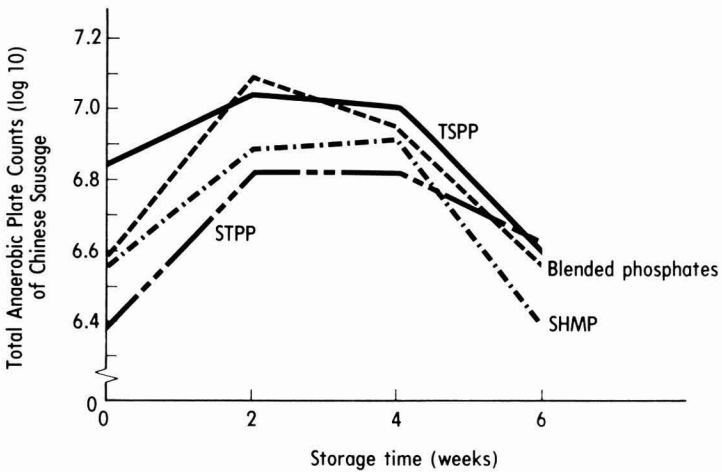


FIG. 4. EFFECT OF PHOSPHATE AND STORAGE TIME ON THE TOTAL ANAEROBIC PLATE COUNTS OF CHINESE SAUSAGE

plate count than the other three phosphate groups. Range of total anaerobic plate counts (log 10) of all treatments was from 6.38 to 7.08 during storage. This high level was probably due to the higher processing temperature (room temperature) during sample preparation since both aerobic and anaerobic plate counts of this product were relatively high at 0 time (6.40 – 7.10). Kuo *et al.* (1986) studied Chinese sausage and found the same results. Although total aerobic and anaerobic plate counts varied slightly among the phosphate groups by visual or odor observation, it seemed that all Chinese sausage had no major microbial problem during 6 weeks of storage. Banwart (1979) indicated that vacuum packaged, cured or processed meats may have microbial levels over 8 (log 10) per g and be considered satisfactory for consumption. Besides, Chinese sausage is not a ready-to-eat meat product and should be heated before consumption. This preparation method eliminates many undesirable bacteria and parasite which may be present in this meat product (Leistner *et al.* 1984).

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# COMPUTER CODES AND THEIR APPLICATION

## DEVELOPMENT OF A SAMPLING PLAN FOR SPECIFIED RISKS BY MICROCOMPUTERS

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Accepted for Publication April 13, 1987

### ABSTRACT

*The selection of a sampling plan is a very critical decision in quality control. There are standard plans available, such as MIL-STD, based on some specified AQL and corresponding "Producer's risk". However, with the widespread use of microcomputers it is possible to specify more than one point on the operational characteristic curve (OC) and obtain the sample size and acceptance number. This study involved the development of a numerical solution based on specifying two points on the OC curve. An algorithm coded in BASIC (compiled to machine language) was developed to yield the sampling plan and corresponding plot of OC curve.*

### INTRODUCTION

The incorporation of statistical methods in the manufacturing of products is probably as old as human civilization. Ancient people, when exchanging commodities, took a small sample and drew conclusions for the particular product. The quality control methods which are in practice today are based on the same principles and logical concepts even though they are far more sophisticated and effective. Although the statistical concepts utilized in quality control methods are fairly well established by many scientists, the speed of data collection and interpretation is still improving. The increased availability and low cost of microcomputers is making revolutionary changes in the quality control field. There has been considerable work done using these computers in quality control as is evident by the reports of Larson (1969), Worthman and Heinrich (1973),

<sup>1</sup>Author to whom all correspondence should be addressed.

and Montgomery (1982). The area of sampling techniques and establishment of sampling plans has also been affected by the progress in computer technology (Snyder and Stores 1972; Olorunniwo and Salas 1982; Chow *et al.* 1972; Chow *et al.* 1983).

The objective of this study was to illustrate the use of microcomputers in making quality control techniques easier to perform. A numerical solution was developed to generate the sampling plan (i.e., the sample size and the acceptance number) when two points on the operational characteristic curve are specified. The program also generates the corresponding plots of the various sampling plans, thus making the decision process quicker.

## THEORETICAL CONSIDERATIONS

The hypergeometric distribution as applied in sampling techniques is defined as the number of defectives ( $d$ ) in a sample of  $n$  items taken, without replacement, from a lot of size  $N$ , of fraction defective  $p$ . This distribution is very basic in the quality control theory, where the decision whether to accept or reject a lot depends upon the number of defectives in a sample. In order to accurately evaluate the "Producer's risk" and the "Consumer's risk" both sample size ( $n$ ) and lot size ( $N$ ) must be considered. However, since in practice the lot size is often considerably larger than the size of the sample taken for inspection, it is often possible to obtain accurate results using other distributions which are easier to use from a computational stand point. Normally when the ( $n:N$ ) ratio is less than or equal to 0.10 it is satisfactory to replace hypergeometric distribution with the binomial distribution (Cowden 1957; Duncan 1965). The probability,  $P$ , of acceptance of a lot using binomial distribution is given by the formula:

$$P = \sum_{i=0}^a \binom{n}{i} (p)^i (1-p)^{n-i} \quad (1)$$

where  $a$  = acceptance number;  $n$  = sample size;  $p$  = fraction defective.

The same formula is used to generate the operational characteristic curve (OC), an expression of the total performance of a particular sampling plan, at various fraction defectives. Each point on the curve (Fig. 1) corresponds to a particular quality level, as well as to a particular probability of acceptance. It is rather obvious that for every quality level there is one probability of acceptance and for every probability of acceptance, one quality level. Consequently every OC curve can be regarded as a set of pairs of points that identically satisfy Eq. 1. If two such pairs of points are given there is at least one OC curve that will satisfy the two pairs of points simultaneously. Two frequently specified pairs are the AQL, (Acceptable Quality Level) and its corresponding "Producer's risk" (PR) and the LTPD, (Lot Tolerance Percent Defective) and corresponding

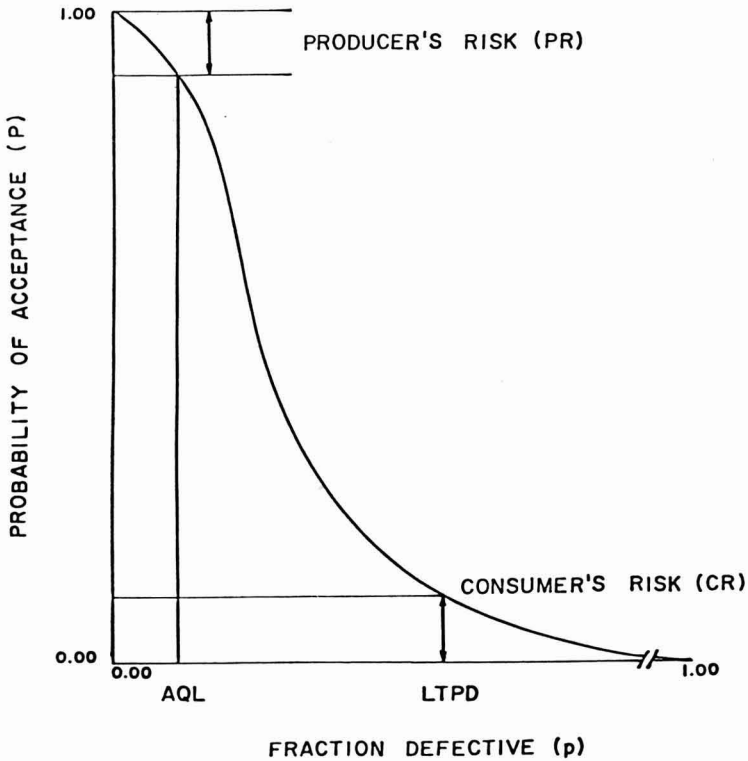


FIG. 1. AQL, LTPD, PRODUCER'S RISK AND CONSUMER'S RISK AS DEFINED ON A TYPICAL OPERATIONAL CHARACTERISTIC CURVE

"Consumer's risk" (CR). In accordance with the previous analysis at least one sampling plan and one OC curve should simultaneously satisfy these pairs. Equation 1 can be written in terms of these two pairs as follows:

$$P_1 = 1 - PR = \sum_{i=0}^a \binom{n}{i} \left(\frac{AQL}{100}\right)^i \left(1 - \frac{AQL}{100}\right)^{n-i} \quad (2)$$

$$P_2 = CR = \sum_{i=0}^a \binom{n}{i} \left(\frac{LTPD}{100}\right)^i \left(1 - \frac{LTPD}{100}\right)^{n-i} \quad (3)$$

The expressions presented by Eq. 2 and 3 can also be visualized in Fig. 1. These two relations represent a system of algebraic equations with two unknowns, the sample size,  $n$  and the acceptance number,  $a$ . Therefore, the solution should yield the parameters of the desired sampling plan. However, due to the nonlinear constraints involved, a direct solution is rather impossible (Wetherill 1969; Schilling 1982).

## METHODOLOGY

The computing power of microcomputers was used to obtain a numerical solution to Eq. 2 and 3. The input parameters required were the "AQL", the "Producer's risk", the "LTPD", the "Consumer's risk" and the size of the lot. The program actually involves four basic steps. During the first step Eq. 2 was numerically solved, to yield pairs of sample sizes and acceptance numbers using the first two parameters (AQL and PR), specified in the input statement. The procedure was written in such a way that the solutions were within an absolute error also specified in the input statement. The sample size was always smaller or equal to one tenth of the specified lot size. In the second step the pairs found from the first step were used as possible solutions to Eq. 3, which utilizes the parameters LTPD and CR, also specified in the input statement. Pairs of sample sizes and acceptance numbers that simultaneously satisfied Eq. 2 and 3 were the desired solutions. During the third step the computer searched for such solutions. If no solution is available based upon the specified criteria the program prompts the user for a change in the input criteria. The final step involved the generation of the OC curves that correspond to the solutions found in the previous steps. A flow chart of the algorithm described above is shown in Fig. 2.

## RESULTS AND DISCUSSION

In most of the cases, the computer generates a range of solutions instead of one pair. Theoretically it is expected that more than one solution will exist. Equation 1 represents a polynomial with its degree dependent upon the sample size. The highest power of this polynomial is  $n$ , and because the sample size is larger than 1, the degree of the polynomial is always greater than 1. Such a polynomial is therefore expected to have more than one solution. The probability of acceptance is not an integer and neither are the producer's and consumer's risks, although they may be input as such. Most of the existing methods use standard values for these parameters, rounding off to the closest integer. Thus when the criteria are specified in the input statement as integers, it is very unlikely that an exact solution will be found. Another reason for not obtaining an exact solution is that the



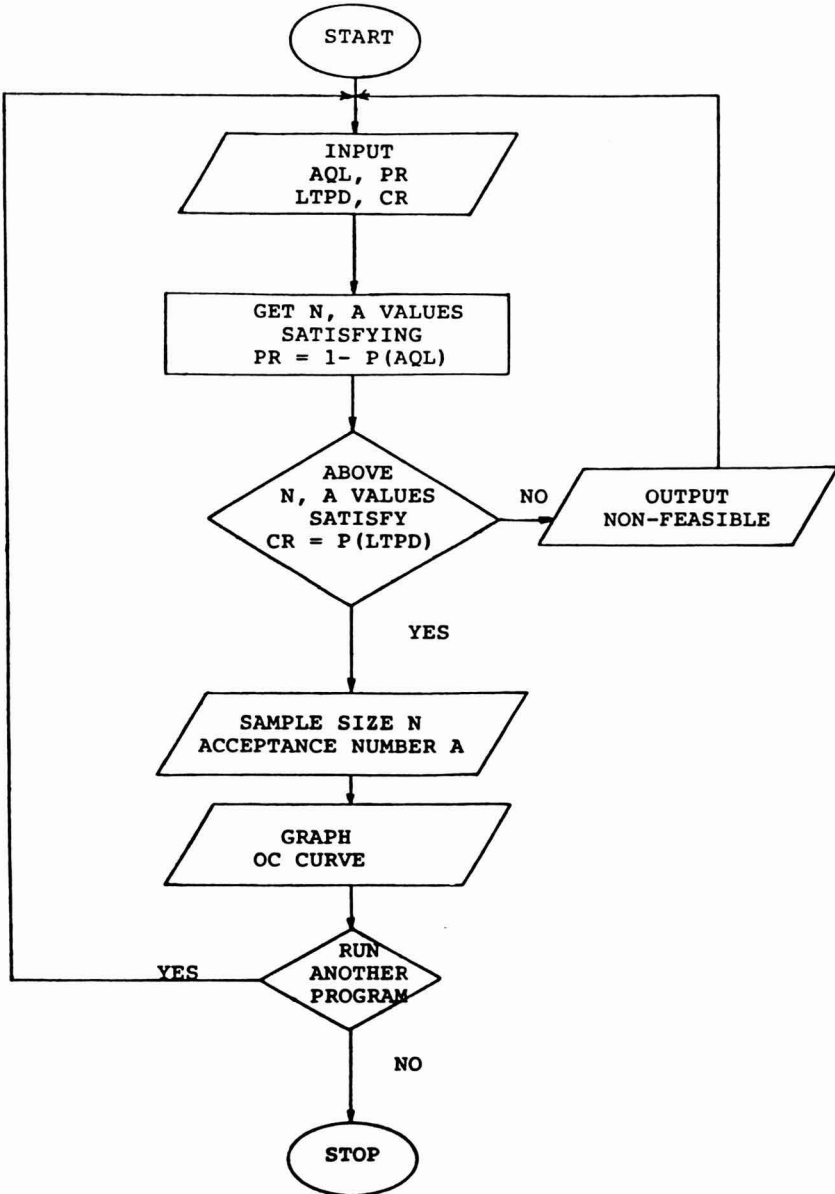


FIG. 2. FLOW CHART FOR DETERMINING SAMPLE SIZE AND ACCEPTANCE NUMBER FOR A SINGLE SAMPLING PLAN

binomial probability is a discrete function rather than a continuous one. The probability of acceptance cannot take just any value from 0 to 100. In addition the sample size and the acceptance number should be integers. These restrictions, imposed on the use of the binomial equation, made the incorporation of the error concept vital, in order to allow for small deviations which would facilitate the finding of the proper solutions. When accuracy is not required the user can specify a larger value for the error, in order to increase the number of solutions given. A numerical example is discussed below. When the following criteria were specified, AQL = 10%, Producer's risk = 40%, LTPD = 20% and Consumer's risk = 20%, no solutions were obtained when the error specified in the input statement was 1%. However, with a specified error of 2% the solution obtained was sample size = 14 and acceptance number = 1. In a second example the input parameters were AQL = 0.5%, Producer's risk = 3%, LTPD = 6% and Consumer's risk = 18%. In this example the number of solutions were 2, 4 and 7 for errors of 0.35%, 1% and 2%, respectively. A computer generated OC curve with sample size and acceptance number for an error of 0.35% is shown in Fig. 3. As the specified error becomes larger, the time required to generate solutions increases while accuracy decreases. It is suggested that the user investigate alternative input criteria rather than increase the error level.

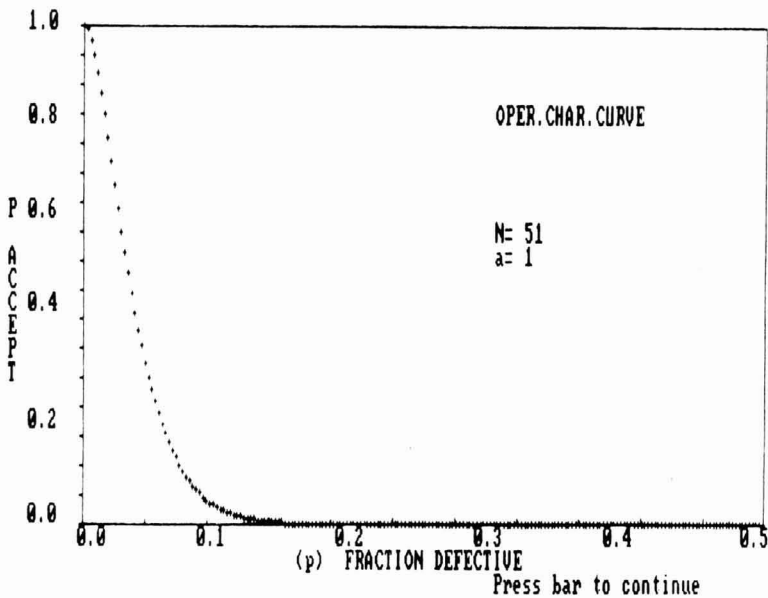


FIG. 3. A TYPICAL SOLUTION AS GENERATED BY THE ALGORITHM DEVELOPED IN THIS WORK

## CONCLUSIONS

Computers are considered a necessity for solution of most numerical problems in various fields. A suitably designed program can always save time, especially when the calculations are complicated. Some salient and new features of the procedure described in this paper are:

- (1) Use of the mathematical expressions without questionable approximations.
- (2) The percent error can be controlled by the user and primarily depends upon the criteria selected by the user.
- (3) Reduction in time factor is achieved because the program is compiled to machine language.

In conclusion, the authors believe that this program can be made part of a general procedure for establishing sampling plans and defining risks to aid the QC departments in the food industry in making more intelligent decisions regarding product acceptability.

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## BOOK REVIEW

**Food Packaging and Preservation: Theory and Practice.** M. Mathlouthi, ed., Elsevier Applied Science Publishers Ltd., pp. 402. \$79.25.

There is a world-wide explosion of interest in the use of various packaging techniques for food preservation. The enormous complexity of approaches involved in realizing such goals is amply illustrated by the contributions to this volume stemming from the First International Symposium on Food Packaging in France (ECPA '85). The book is a collection of nineteen papers presented in six sessions of the three-day symposium. These articles are not intended as a comprehensive review of the food packaging field, but they do provide valuable overviews of selected principles and practices. The first seven papers cover the fundamental aspects of mass transfer, permeability, water activity and interactions, and discuss the relationships between food and packaging materials. The next six contributions are devoted to chemical and microbiological considerations for food preservation with a major thrust on the role of water activity in prolonging the shelf-life of packaged foods. The last six articles discuss a number of other topics such as packaging materials and machines, permeation of aroma components, sensory evaluation of packaged foods and edible films. The papers are authored by many of the scientists working in the area and cover a number of disciplines.

As in most proceedings, there is unevenness in detail and lack of uniformity and coherence in this volume as well. Many of the papers describe progress to date, technical advances or individual studies. Other papers provide an overview of basic scientific principles found in text books. There is something in it for almost anyone with an interest in food preservation and packaging. A few glaring gaps do exist. For example, conspicuous by their absence are such important and widely used practices as modified atmosphere, aseptic, and retort packaging. Even the index is devoid of these key words. A rudimentary description of controlled atmosphere packaging is found in the article on "Water Interactions and Food Preservation". The title for the book is thus much broader than its contents.

Even though the book is most useful as an update for researchers in the field, it will provide others with general understanding of the multidisciplinary aspects of food packaging and preservation.

S.S.H. RIZVI



## AUTHOR INDEX

- ABBO, E. *See* GIANGIACOMO, R. *et al.*
- ADEYEMI, I.A. *See* AKINGBALA, J.O. *et al.*
- AGUILERA, J.M., ROMAN, J. and HOHLBERG, A. Engineering and Quality Aspects of Particle-To-Particle Heat Transfer to Black Beans 263
- AKINGBALA, J.O., ONOCHIE, E.U., ADEYEMI, I.A. and OGUNTOMEIN, G.B. Steeping of Whole and Dry Milled Maize Kernels in Ogi Preparation 1
- AKINGBALA, J.O. and ROONEY, L.W. Paste Properties of Sorghum Flour and Starches 13
- AMUNDSON, C.H. *See* HILL, JR., C.G. *et al.*
- BARRETT, E.L. *See* GRISIUS, R. *et al.*
- BEAUCHAT, L.R. *See* KAMENAN, A. *et al.*
- BEAUCHAT, L.R. *See* McWATTERS, H.K. *et al.*
- BECKER, R. *See* DEL VALLE, F.R. *et al.*
- BEVILACQUA, A.E. *See* LANARI, M.C. *et al.*
- BRYAN, W.D. *See* JOHNSON, C.D. *et al.*
- CHAUHAN, P. and WADHWA, B.K. Comparative Evaluation of Ghee in Tin and Polyethylene Packages During Storage 25
- CHEN, T.C. *See* WANG, S.W.
- CHINNAN, M.S. *See* KAMENAN, A. *et al.*
- CHINNAN, M.S. *See* McWATTERS, K.H. *et al.*
- DEL VALLE, F.R., MARCO, E., BECKER, R. and SAUNDERS, R.M. Development and Evaluation of a Procedure to Produce Mesquite (*prosopis* spp.) Pod Protein Concentrate 237
- DOLAN, K.D., SINGH, R.P. and HELDMAN, D.R. Prediction of Temperature in Frozen Foods Exposed to Solar Radiation 135
- EITENMILLER, R.R. *See* JOHNSON, C.D. *et al.*
- EITENMILLER, R.R. *See* RAO, V.N.M. *et al.*
- GIANGIACOMO, R., TORREGGIANI, D. and ABBO, E. Osmotic Dehydration of Fruit: Part I. Sugars Exchange Between Fruit and Extracting Syrups 183
- GILBERT, S.G. *See* KINIGAKIS, P. *et al.*
- GILL, T.A. *See* LEBLANC, E.L. *et al.*
- GRISIUS, R., WELLS, J.H., BARRETT, E.L. and SINGH, R.P. Correlation of Time-Temperature Indicator Response with Microbial Growth in Pasteurized Milk 309
- HASEGAWA, T. *See* SAKAI, Y. *et al.*
- HAYES, K.M. *See* RAMAKRISHNAN, T.V. *et al.*
- HEATH, J.L. and OWENS, S.L. Effect of Tumbling Solutions Containing Ethanol on Tenderness and Yield of Broiler Breasts 159

- HEATON, E.K. *See* KAMENAN, A. *et al.*
- HEDINSDOTTIR, R. *See* HILL, JR., C.G. *et al.*
- HELDMAN, D.R. *See* DOLAN, K.D. *et al.*
- HILL, JR., C.G., HEDINSDOTTIR, R. and AMUNDSON, C.H. Surface Active Properties and Cleaning Efficacy of Materials Derived from Lactitol 111
- HINTLIAN, C.B. and HOTCHKISS, J.H. Microbiological and Sensory Evaluation of Cooked Roast Beef Packaged in a Modified Atmosphere 171
- HOHLBERG, A. *See* AGUILERA, J.M. *et al.*
- HOHLBERG, A.I. and STANLEY, D.W. Kinetics of Bean Protein Thermal Denaturation 31
- HOTCHKISS, J.H. *See* HINTLIAN, C.B.
- HUANG, C.J. *See* KUO, J.C. *et al.*
- ISHIKAWA, M., MORI, S., WATANABE, H. and SAKAI, Y. Softening of Fish Bone. I. Relation Between Softening Rate and Solubilization Rate of Organic Matter from Fish Bone 277
- JOHNSON, C.D., BRYAN, W.D. and EITENMILLER, R.R. Proximate and Water Soluble Vitamin Composition of Grapefruit and Oranges 83
- KAMENAN, A., BEUCHAT, L.R., CHINNAN, M.S. and HEATON, E.K. Composition and Physico-Chemical Properties of Yam (*Dioscorea* Species) Flour Prepared Using Different Processes 299
- KINIGAKIS, P., MILTZ, J. and GILBERT, S.G. Partition of VCM in Plasticized PVC/Food Simulant Systems 247
- KUO, J.C., WANG, S.Y., HUANG, C.J. and OCKERMAN, H.W. Effects of Phosphate Type, Packaging Method and Storage Time on the Characteristics of Chinese Sausage 325
- LANARI, M.L., BEVILACQUA, A.E. and ZARITZKY, N.E. Changes in Tenderness During Aging of Vacuum-Packaged Beef 95
- LAPERLE, E.A. *See* RAMAKRISHNAN, T.V. *et al.*
- LEBLANC, E.L., LEBLANC, R.J. and GILL, T.A. Effects of Pressure Processing on Frozen Stored Muscle Proteins of Atlantic Cod (*Gadus morhua*) Fillets 209
- LEBLANC, R.J. *See* LEBLANC, E.L. *et al.*
- MARCO, E. *See* DEL VALLE, F.R. *et al.*
- McWATTERS, K.H., CHINNAN, M.S., WORTHINGTON, R.E. and BEAUCHAT, L.R. Influence of Storage Conditions on Quality of Cowpea Seeds and Products Processed from Stored Seeds 63
- MILTZ, J. *See* KINIGAKIS, P. *et al.*
- MORI, S. *See* ISHIKAWA, M. *et al.*
- OCKERMAN, H.W. *See* KUO, J.C., *et al.*
- OGUNTMEIN, G.B. *See* AKINGBALA, J.O. *et al.*
- ONOCHIE, E.U. *See* AKINGBALA, J.O. *et al.*
- OWENS, S.L. *See* HEATH, J.L.



- PAPPAS, G. and RAO, V.N.M. Development of a Sampling Plan for Specified Risks by Microcomputers 339
- RAMAKRISHNAN, T.V., LAPERLE, E.A. and HAYES, K.M. Comparison of Steam Canner Processing with Other Methods of Home Canning 43
- RAO, V.N.M., WEBB, K. and EITENMILLER, R.R. A Microcomputer Based Algorithm for Quantitative Determination of Vitamins from Turbidimetric Methods 77
- RAO, V.N.M. *See* PAPPAS, G.
- RAO, V.N.M. *See* WEBB, K.S.
- ROMAN, J. *See* AGUILERA, J.M.
- ROONEY, L.W. *See* AKINGBALA, J.O.
- SAKAI, Y., WATANABE, H., TAKAI, R. and HASEGAWA, T. A Kinetic Model for Oxidation of Ascorbic Acid and Beta-Carotene 197
- SAKAI, Y. *See* ISHIKAWA, M. *et al.*
- SAUNDERS, R.M. *See* DEL VALLE, F.R. *et al.*
- SINGH, R.P. *See* DOLAN, K.D. *et al.*
- SINGH, R.P. *See* GRISIUS, R. *et al.*
- STANLEY, D.W. *See* HOHLBERG, A.I.
- TAKAI, R. *See* SAKAI, Y.
- TORREGGIANI, D. *See* GIANGIACOMO, R.
- WADHWA, B.K. *See* CHAUHAN, P.
- WANG, S.W. and CHEN, T.C. Yield and Tenderness of Cured Chicken Roll Products as Affected by Water Addition 289
- WANG, S.Y. *See* KUO, J.C. *et al.*
- WATANABE, H. *See* ISHIKAWA, M. *et al.*
- WATANABE, H. *See* SAKAI, Y. *et al.*
- WEBB, K.S. and RAO, V.N.M. Algorithm for Quality Control Charts for Variables Using Microcomputers 257
- WEBB, K. *See*, RAO, V.N.M. *et al.*
- WELLS, J.H. *See*, GRISIUS, R. *et al.*
- WORTHINGTON, R.E. *See* McWATTERS, K.H. *et al.*
- ZARITZKY, N.E. *See* LANARI, M.C. *et al.*



## SUBJECT INDEX

- Ascorbic acid, kinetic model for oxidation, 197-207
- Beans, particle to particle heat transfer, 263-276
- Beef, cooked roast in modified atmosphere, 171-179  
tenderness of vacuum-packaged, 95-109
- Beta-carotene, kinetic model for oxidation, 197-207
- Canning, home, 43-61
- Chicken, tenderness and yield during tumbling with ethanol, 159-169
- Cleaning, efficacy of lactitol, 111-134
- Cod, protein, effects of pressure, 209-235
- Computer code, algorithm for quality control charts, 257-262  
determination of vitamins, 77-82  
sampling plan for quality control, 339-445
- Cowpea seeds, storage, 63-76
- Dehydration, osmotic, of fruit, 183-195
- Denaturation, bean protein, 31-42
- Fishbone, softening, 277-287
- Flour, yam, 299-308
- Frozen foods, effect of solar radiation on temperature, 135-158
- Fruit, osmotic dehydration, 183-195
- Ghee, packaging, 25-30
- Grapefruit, proximate and vitamin composition, 83-89
- Heat transfer, particle to particle, 263-276
- Kinetics, oxidation of ascorbic acid and  $\beta$ -carotene, 197-207
- Kinetics of denaturation, 31-42
- Maize kernels, steeping, 1-11
- Microbiology, cooked roast beef in modified atmosphere, 171-179
- Milk, time-temperature indicators, 309-324
- Modified atmospheres, storage of cooked roast beef, 171-179
- Muscle proteins, fish, effects of pressure, 209-235
- Oranges, proximate and vitamin composition, 83-89
- Packaging, chinese sausage, 325-338  
partitioning of vinyl chloride monitor, 247-255  
vacuum-treatment of beef, 95-109
- Phosphate, effect on chinese sausage, 325-338
- Polyethylene, packaging of ghee, 25-30
- Poultry, yield and tenderness of cured chicken roll, 289-298

- Protein, fish muscle, effects of  
  pressure, 209-235  
  thermal denaturation of bean,  
    31-42
- Protein concentrate, mesquite pod,  
  237-246
- Proximate composition, grapefruit  
  and oranges, 83-89
- Quality control, algorithm for  
  charts, 257-262  
  sampling plan, 339-445
- Sausages, packaging of chinese  
  sausage, 325-338
- Solar radiation, influence on tem-  
  perature of frozen foods,  
    135-158
- Sorghum flour and starches, paste  
  properties, 13-24
- Surface active properties, lactitol,  
  111-134
- Texture, of vacuum-packaged beef,  
  95-109
- Time-temperature indicators, for  
  pasteurized milk, 309-324
- Turbidimetric methods, determina-  
  tion of vitamins, 77-82
- Vinyl chloride monomer, partition-  
  ing, 247-255
- Vitamins, quantitative determina-  
  tion, 77-82.  
  water soluble in grapefruit and  
    oranges, 83-89
- Yam flour, 299-308

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HUMAN NUTRITION, 3RD ED., F.R. Mottram  
FOOD POISONING AND FOOD HYGIENE, 4TH ED., B.C. Hobbs *et al.*  
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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 References 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) and 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.

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EDITORIAL OFFICE: Dr. D.B. Lund, Editor, *Journal of Food Processing and Preservation*, University of Wisconsin-Delaware, Newark, Delaware 19713.

CONTENTS

Editorial .....v

Engineering and Quality Aspects of Particle-To-Particle Heat Transfer to Black Beans  
**J.M. AGUILERA, J. ROMAN and A. HOHLBERG** ..... 263

Softening of Fish Bone. I. Relation Between Softening Rate and Solubilization Rate of Organic Matter from Fish Bone  
**M. ISHIKAWA, S. MORI, H. WATANABE and Y. SAKAI** ..... 277

Yield and Tenderness of Cured Chicken Roll Products as Affected by Water Addition  
**S.W. WANG and T.C. CHEN** ..... 289

Composition and Physico-Chemical Properties of Yam (*Dioscorea* Species) Flour Prepared Using Different Processes  
**A. KAMENAN, L.R. BEUCHAT, M.S. CHINNAN and E.K. HEATON** ..... 299

Correlation of Time-Temperature Indicator Response with Microbial Growth in Pasteurized Milk  
**R. GRISIUS, J.H. WELLS, E.L. BARRETT and R.P. SINGH** .... 309

Effects of Phosphate Type, Packaging Method and Storage Time on the Characteristics of Chinese Sausage  
**J.C. KUO, S.Y. WANG, C.J. HUANG and H.W. OCKERMAN** ... 325

Computer Codes and Their Application  
Development of a Sampling Plan for Specified Risks by Microcomputers  
**G. PAPPAS and V.N.M. RAO** ..... 339

Book Review ..... 347

Author Index ..... 349

Subject Index ..... 353



