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D.B. LUND EDITOR

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# Journal of FOOD PROCESSING and PRESERVATION

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# EDITORIAL

1988 was the 12th year of the *Journal of Food Processing and Preservation* and I can report that it has been an extremely successful year. We had some additional papers in two unique sections: (1) Computer Codes and Their Applications, and (2) Databank. I would like to encourage our readership to submit papers appropriate for both of 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 last year. I especially want to thank Dr. Ricardo Villota who has served on the Board for a three year term and to welcome Dr. J.H. von Elbe who will begin a three year term. Professor Breene, Cash and Swanson will continue with another three year term.

In addition I would like to thank all who served as reviewers (listed below) for 1988. Their contribution is 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 look forward to receiving other quality manuscripts from our past authors and encourage others to submit quality manuscripts.

For Volume 13, the Journal is going to six issues per year. This will greatly reduce time between acceptance of papers and actual publication and will allow us to bring more research to the public sector. Thank you for your support.

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# FILTRATE FLOW THROUGH FILTER CAKES WHICH EXHIBIT TIME DEPENDENT RESISTANCE

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# ABSTRACT

Filtrate flow through filter cakes which exhibits time dependent resistance was studied by measuring filtration rates through four types of perlite filter aids using a laboratory test system. Poultry chiller overflow water which has the potential for significant savings if recycled was used as the test suspension. Assumption of constant specific cake resistance and determination of its value from a plot of filtration rate against cummulative filtrate volume resulted in negative values for the medium resistance and large deviation between predicted and experimental values of filtrate volume particularly in the early stages of the filtration cycle. Equations were derived for independent determination of filtration medium resistance, which enabled determination of time dependent cake resistance factor. An equation which considered a time dependent cake resistance predicted values for filtrate volume which agreed well with experimental data. Data show that batch filtration of chiller overflow water will require very short cycle times for optimum filtrate flow.

#### INTRODUCTION

Filtration is a common operation for clarifying liquids containing suspended solids. Studies illucidating filtration behavior through filter cakes which exhibit time dependent specific cake resistance will have wide applications in the food industry. Data collected in the present study are specifically for determination of requirements for clarifying poultry chiller water overflow for recycling. This practice is allowed by the Food Safety and Inspection Service (FSIS) of the United States Department of Agriculture USDA (CFR, 1987). Poultry processors are interested in this process for reducing ice and water usage and liquid waste effluent. Lillard (1978) and Chang *et al.* (1986) reported that filtration produces the filtrate clarity and microbial reduction which meets FSIS guidelines for recycling. Not addressed in these studies is the problem of filtrate flow as a function of length of filtrate cycles.

In the process of filtration, fluid is passed through a porous medium which retains the suspended solids. The porous medium is formed by a filter cloth which is usually not fine enough to retain the suspended solids by itself, a precoat of filter aid which provides a porous barrier for filter cake retention, and finally and filter cake itself which consists of the suspended solids and filter aid added to the feed. Several articles were previously published regarding development of equations for prediction of filtrate flow. Some of the more recent are those of Kottwitz and Boylan 1958; Baumann *et al.* 1962; Baumann and Oulman 1964; and Gale 1967. In developing prediction equations, pore size was assumed to be constant and filter cakes were assumed to be incompressible. There is very little information on resistance to flow through cakes which exhibit increasing specific resistance with time of filtration.

The Sperry equation which is widely used to predict filtration behavior, (Dickey 1961) is expressed below:

$$\frac{dt}{dv} = \frac{u}{\Delta P A} \left( \frac{\alpha c V}{A} + R_m \right) \qquad \text{Eq. 1}$$

 $R_m$  is the medium resistance defined by Eq. 2.  $R_m$  is attributed to the resistance of the filter support and the precoat of filter aid to filtrate flow.  $\alpha$  is the specific cake resistance defined by Eq. 3.

$$R_{m} = \frac{\Delta P_{m}}{\mu U} \qquad \text{Eq. 2}$$

$$\alpha = \frac{\Delta P_c A}{\mu U M c}$$
 Eq. 3

 $\alpha$  is attributed to the resistance to filtrate flow of filter cake which is formed when suspended solids and filter aid in the feed, accumulate on the filter. At constant pressure, if the cake is incompressible and solids do not migrate through the cake, then  $\alpha$  is constant even though the filtrate velocity decreases as solids accumulate in the cake.

Equation 1 can be integrated for constant pressure filtration to yield Eq. 4.

$$t = \frac{\frac{\mu \alpha c}{2 \Delta P A^2}}{\frac{\lambda P A^2}{2 \Delta P A}} \frac{v^2}{v^2} + \frac{\frac{\mu R_m}{\Delta P A}}{\frac{\lambda P A}{2 \Delta P A}} v \qquad Eq. 4$$

Equation 4 shows that if  $R_m \ll \alpha c$ , at large values of V the second term in Eq. 4 may be neglected. "t" when plotted against  $(A/V)^2$  will be linear and  $\alpha$  can be calculated from the slope.  $R_m$  can also be calculated by plotting the reciprocal of the rate of filtration as a function of cummulative filtrate volume according to Eq. 1. These procedures however, cannot be used if  $\alpha$  is not constant.

This study was conducted to determine specific resistance of filter cakes formed during constant pressure filtration of poultry chiller water overflow containing perlite as the filter aid and to develop equations that best describe filtrate flow through filter cakes that exhibit time dependent specific resistance.

# MATERIALS AND METHODS

#### Water Samples for Filtration

Chiller overflow water was obtained from a poultry broiler processing plant in Athens, GA. The water was collected after at least 4 h of steady state operation from start-up, transported to the laboratory (ca. 15 min), and used within a 2 h period for the experiments. To prevent variations in temperature, the plastic jugs containing the sample was stored under refrigeration at 2 °C. The content of the jugs was thoroughly mixed prior to drawing subsamples of 7.5 L at one time. The samples were passed through an 80 mesh sieve to remove large solids and congealed fat, and immediately used in the filtration experiments. A small volume was saved for analysis of suspended solids using the procedures for analysis of water and waste water (APHA, 1976).

# Filtration

Extensive analysis of suspended solids present in chiller water overflow established a mean solids content of 535 mg/L. Preliminary experiments were also conducted on the influence of amount of filter aid added to the water on filtration rate. Results indicated that although filtrate flow increased with increasing filter aid concentration early in the filtration cycle, the cummulative volume curves cross over because of faster accumulation of cake thickness at higher concentrations. The best compromise on filtrate flow and quantity of filter aid utilized a 1:1 filter aid to suspended solids concentration ratio. However, it was not possible to keep this ratio exact in all filtration experiments since it took at least 8 h to complete the solids analysis. Thus, based on a mean solids content of 535 mg/mL, a constant 0.05% filter aid concentration was used in all tests. A small sample of the unfiltered water was saved prior to each test and suspended solids analyzed. Twenty minute filtration tests were used early in the study and duration was later extended to 60 min. In a companion study

(Chang *et al.* 1986) it was shown that total aerobic counts in the unfiltered water did not change during this period. Data for each batch was analyzed separately since significant batch to batch variation prevented pooling of data. There was no control of suspended solids in the samples obtained from the poultry processing plant and concentration varied from 280 to 600 mg/L.

# **Filter Aids**

Perlite, an amorphous fused sodium potassium aluminum silicate mineral (GREFCO, Inc., Los Angeles, CA) was used as filter aid. The four types used, designated by the manufacturer with numbers which increased with increasing particle size were: 416, 436, 476, and 4106. These types have rated pure water permeabilities, respectively, of 0.13, 0.40, 0.93, and 1.5 mL/s(cm<sup>2</sup>) through a cake 1 cm thick and a pressure drop of 1 atm.

#### **Filtration System**

The filtration system shown schematically in Fig. 1, was constructed at the University machine shop. The design is similar to the standard filtration test device used in industry except that a mixer was added to the present system to prevent settling of the filter aid in prolonged filtration cycles. A 7.5 L pressure vessel (h) held the fluid and filter aid. A 60 RPM gear motor (b) Model 2Z810, (Dayton Motors, Chicago, IL) Dayton Motors, Model 2Z810, Chicago, IL) mounted on a support bracket bolted to the cover turns a stirrer shaft that enters the vessels through a mechanical seal. Two stirrer elements (i) were mounted on the shaft. The motor, stirrer shaft and top cover remained as one unit after assembly. The gas pressurization assembly which consisted of a stainless steel tube (f) fitted with a pressure gage (a), a needless valve (e), and a pressure regulator (g) was connected to the cover. This assembly was disconnected from the cover when disassembly the unit after a test. Another fitting on the cover provided connections for the filtrate exit tube (c) and an extension towards the bottom of the pressure vessel which terminated in a filtration cell assembly (x). The extension tube with the filtration cell can be detached from the cover when needed. The filtration area of the test cell and the thickness of cake deposited in one filtration test can be adjusted using a replacable ring (j) on the base of the filtration cell. A filtration area of 20.27 cm<sup>2</sup> was used in all experiments. A needle valve (d) at the filtrate exit tube remained closed while preparing to start filtration, and timing of filtration was started when the needle valve resulted in the deposition of 2.20 g of filter aid  $(1 \text{ kg } (dry)/m^2)$  filter area.

After precoating, 7.5 L of chiller overflow water and 0.05% (w/w, dry) filter aid were mixed thoroughly. The pressure vessel was opened and the suspension was siphoned into the vessel to prevent the incoming fluid from dropping directly on the precoated filtration test cell. The vessel was then closed, agitation



FIG. 1. SCHEMATIC DIAGRAM OF TEST FILTRATION SYSTEM:
a. pressure gage; b. gear motor; c. filtrate exit tube; d. needle valve; e. needle valve;
f. compressed air inlet tube; g. pressure regulator; h. pressure vessel; i. stirrer element;
j. replaceable ring; k. filtration cell assembly.

started, and the filtrate exit control valve closed. The vessel was then pressurized to 172.3 kPa. When pressure stabilized, the needle valve on the exit tube was opened and timing was started immediately. Cummulative filtrate volume was measured using a graduated cylinder, at two minute intervals. In a companion study (Chang *et al.* 1986) turbidity and microbial counts were also determined on the feed and filtrates. Results of this study showed that turbidity of filtrates approached that of tap water and microbial count reduction was in the order of 95%. Thus for most practical purposes filtrates were free of suspended solids greater than 0.5  $\mu$ m in size.

#### **RESULTS AND DISCUSSION**

#### **Constant Specific Cake Resistance Assumed**

Analysis of filtration data is often done (McCabe and Smith 1956) by plotting time (t) against cummulative filtrate volume (V), drawing tangents and determining slopes (dt/dV) at different cummulative volume. A linear plot is then made of dt/dV against V according to Eq. 1.  $\alpha$  and R<sub>m</sub> are determined from the slope and intercept, respectively. This manual manipulation of tangents to determine dt/dV, often resulted in negative values for R<sub>m</sub>, which is physically impossible. To ensure positive values for R<sub>m</sub>, polynomial regression may be used to fit the data based on Eq. 4. Using data from a typical filtration experiment, the resulting best fit equation was:

$$t = 0.0262 V^2 + 0.4589 V (r^2 = 0.999) Eq. (5)$$

Curve (A) in Fig. 2 shows calculated values of V using Eq. 5. the curve shows a good fit to experimental data ( $r^2=0.999$ ) but filtrate flow in the first 4 min of the filtration cycle was underestimated, indicating that the second term in Eq. 5 which accounts for  $R_m$  may be too high. On the assumption that  $R_m$  may be negligible compared to c, the second term in Eq. 4 may be dropped to yield Eq. 6.

$$t = \frac{\mu \alpha c}{2 \Delta P A^2} V^2 \qquad Eq. (6)$$

Equation 6 suggests that t will be linear against  $V^2$ , and regression analysis resulted in Eq. 7.

t = 
$$0.001940 V^2$$
 (r<sup>2</sup> = 0.995) Eq. (7)

Equation 7 was used to generate curve B, Fig. 1, which represented the data better than Eq. 5 in the first minute of filtration. However, considerable deviation occurred later in the filtration cycle. The  $r^2$  of 0.995 is high, but calculated filtrate flow with prolonged filtration was higher than experimental data.

A procedure was developed to correctly assess the contribution of the medium resistance to the total resistance against filtrate flow. The procedure involved measurement of filtrate flow (q) through a precoated filtration cell using water (no filter aid) in the pressure vessel. The pressure drop  $\triangle P_m$  across the filter medium and through the filtrate exit tube, will be the applied pressure  $\triangle P$ . Substituting Eq. 2 in Eq. 4 and considering that UA is the volumetric rate of



FIG. 2. PREDICTED CUMMULATIVE FILTRATE VOLUME ASSUMING CONSTANT SPECIFIC CAKE RESISTANCE (Curve A - constants derived using Sperry equation; Curve B -constants derived by regression assuming no medium resistance; Curve C - constants derived by regression and separate measurement of medium resistance). Points represent experimental data

flow of filtratre (q) when only the filter and precoat of filter aid are in place. Eq. 8 is obtained. Under these conditions filter cake thickness is constant, therefore, q is constant with time.

$$t = \frac{\mu \alpha c}{2\Delta P A^2} V^2 + V/q \qquad Eq. (8)$$

 TABLE 1.

 MEDIUM RESISTANCE OF THE FILTRATION SYSTEM USING DIFFERENT FILTER AIDS

and a second s				
Filter aid (Perlite)	416	436	476	4106
Flow rate q (ml/sec)	7.1	25.5	28.8	26.8
Rm ×1 <sup>107</sup> (cm <sup>-1</sup> )	44.2	12.3	10.9	8.6

Table 1 shows the volume (q) of the filtrate flowing through the precoated filtration cell and the calculated values of  $R_m$ . Since in filtration systems the resistance of the filter cloth and support is designed to have a small value,  $R_m$  is attributed primarily to the resistance of filter aid used to precoat the filter. Therefore values for  $R_m$  should be applicable for any system which uses the same type of filter aid. Data for filtration through type 416 filter analyzed by polynomial regression based on Eq. 8 resulted in Eq. 9.

t = 
$$0.001728 V^2$$
 +  $0.1402 V$  (r<sup>2</sup> = 0.990) Eq. (9)

Cummulative filtrate volume calculated using Eq. 9 (curve C, Fig. 2), did not improve the fit compared with Eq. 7. Thus, even with the improvements in methods for analyzing filtration data introduced above, a satisfactory fit to experimental data was not possible when constant specific cake resistance was assumed. Other filtrate aids were also used in the filtration experiments and similar results were observed.

#### **Time Dependent Cake Resistance**

The reason for the poor fit of filtrate flow equations (Eq. 5, 7, and 9) to experimental data may be the time dependent nature of cake resistance. An empirical equation (Eq. 10) was assumed to represent filtrate flow. The coefficient of  $V^2$  in Eq. 10 was assumed to be a time dependent function f(t).

$$t - (V/q) = f(t)(V^2)$$
 Eq. (10)

Since V/q has units of time, the left hand side of Eq. 10 can be designated as a corrected filtration time, t', and the right hand side represents a time dependent function which affects filtrate flow. f(t) may be designated by a parameter K. As a first approximation, K may be assumed to be linearly time dependent according to Eq. 11:

$$K = \beta t + \gamma \qquad Eq. (11)$$

Substituting K for f(t) in Eq. 10 will allow calculation of K at different times of filtration. For the linear approximation, regression of (t '/V<sup>2</sup>) against K will give a slope equal to  $\beta$  and an intercept equal to  $\gamma$ . Values of K calculated using filtration test data on filter aid type 416 is shown by the data points in Fig. 3. Curve A represents the regression line assuming a linear dependence of K with time. The data points do not all lie on the line and a definite curving trend is exhibited. Linear regression equations for K as a function of time, for filtration of water containing various solids content are shown in Tables 2, 3, 4, and 5 for filter aid types 416, 436, 476, and 4016, respectively. The data shows variation in K from one batch of poultry chiller water to another. There is no definite trend relating suspended solids content to the magnitude of K. The variability may be attributed to the difference in the type (e.g., grease, fat-protein complex) and particle size distribution of suspended solids in the different batches of chiller water. Thus, filtration performance estimates on scale-up may be made only on a "worst case" basis. In Table 2, the sample which contained 515 mg/mL suspended solids exhibited the highest resistance to filtrate flow since it had the highest value for K.

Filtrate flow can be easily calculated using the time dependent expression for K. Cummulative filtrate volume against time can be calculated using values of  $\beta$  and  $\gamma$  (from Tables 2–5) and flow rate q of filtrate through the precoated filtration test cell (from Table 1) using the positive root of Eq. 12.

$$V = \frac{-\frac{1}{q} + \sqrt{(\frac{1}{q})^2 + 4(\beta t + \gamma) t}}{2(\beta t + \gamma)} \qquad \text{Eq. (12)}$$



FIG. 3. LINEAR PLOT OF CAKE RESISTANCE FACTOR K AS A FUNCTION OF FILTRATION TIME

Figure 4 shows experimental data for the "worst case" sample which exhibited the highest values for K when using filter aid type 416. Curve A represents calculated values using Eq. 10 and 11 and the parameter estimates for  $\beta$  and  $\gamma$  for this batch shown in Table 2. The assumption of a linear change in K with time, resulted in a better fit to the data compared to that for the standard filtration equation (Eq. 4) with constant values for  $\alpha$  previously shown in Fig. 2. A slight deviation is exhibited but for most practical purposes, the calculated and experimental values are similar.

Solid Content (mg/1)	k(x 10 <sup>7</sup> S/cm <sup>6</sup> )	R <sup>2</sup>
474	4.30 t + 7700	0.953
608	4.39 t + 12000	0.908
515	7.38 t + 10300	0.985
478	4.11 t + 6700	0.950
555	2.47 t + 8460	0.994
555	2.04 t + 9320	0.989
280	1.47 t + 6400	0.998
280	1.04 t + 6700	0.982
419	1.22 t + 3240	0.970

TABLE 2.CAKE RESISTANCE FACTOR AT 0.05% PERLITE 416

TABLE 3.	
CAKE RESISTANCE FACTOR FOR POULTRY CHILLER WA	TER
CONTAINING 0.05% PERLITE 436	

Solid Content (mg/1)	k(x 10 <sup>7</sup> S/cm <sup>6</sup> )	R <sup>2</sup>
500	1.11 t + 1800	0.985
608	2.70 t + 2910	0.968
515	2.75 t + 2360	0.983
478	2.56 t + 3130	0.983
280	0.56 t + 1400	0.968
419	0.50 t + 1258	0.998

Solid Content (mg/1)	k(x 10 <sup>7</sup> S/cm <sup>6</sup> )	R <sup>2</sup>
500	1.11 t + 680	0.927
608	2.67 t + 1770	0.974
515	2.25 t + 2100	0.983
478	2.57 t + 2280	0.990
538	0.66 t + 1380	0.985
538	0.39 t + 1290	0.995
405	0.42 t + 1300	0.993
405	0.57 t + 1130	0.998
555	2.08 t + 1420	1.000
555	2.15 t + 1500	0.999
360	0.17 t + 800	0.977

TABLE 4. CAKE RESISTANCE FACTOR FOR POULTRY CHILLER WATER CONTAINING 0.05% PERLITE 476

The plot of K against time (Fig. 3, curve B) shows a better fit for K when an exponential expression for K was assumed (Eq. 13):

$$K = a + b (1 - e^{-c't})$$
 Eq. (13)

Equation 13 implies a limiting value of K when filtrate flow is completely impeded, at which point no additional solids deposition will occur. Values of a and b were calculated from K at different filtration times. Rearranging Eq. 13:

$$\ln(1 - \frac{k-a}{b}) = -ct$$
 Eq. (14)

The constant, a, is the value K at time 0. The value of K after 20 min of filtration was used as an assumed value of b and the correct value was determined by trial and error. A regression analysis was performed on the left hand side of Eq. 14 against t using an assumed values of b. By incremenating b and repeating the regression, a value of b can be obtained which gave a maximium  $r^2$  value for the regression. The corresponding value of , c', was obtained from the slope of the regression line.

Solid Content (mg/1)	k(x 10 <sup>7</sup> S/cm <sup>6</sup> )	R <sup>2</sup>
608	0.56 t + 376	0.994
515	1.04 t + 591	0.978
478	1.13 t + 733	0.979
560	0.69 t + 833	0.999
500	0.48 t + 904	0.997
680	0.35 t + 709	0.998
680	0.31 t + 605	0.999
495	0.51 t + 462	1.000
495	0.42 t + 508	0.996
750	0.44 t + 548	0.993
750	0.42 t + 541	0.988

TABLE 5. CAKE RESISTANCE FACTOR FOR POULTRY CHILLER WATER CONTAINING 0.05% PERLITE 4106.

For the data set on filter aid type 416, the values of a, b, and c' are respectively, 0.00103, 0.0024, and 0.000366. The  $r^2$  value was 0.998 indicating a very good fit.

When K is expressed by Eq. 13, the filtrate volume may be calculated by Eq. 15:

$$V = \frac{-1/q + \sqrt{(1/q)^2 + 4 a t + 4 b t (1 - e^{-C't})}}{2(a + b(1 - e^{-C't})} \quad \text{Eq. (15)}$$

Curve B in Fig. 4 shows that the cumulative volume generated using the expression for K in Eq. 13, exactly coincides with the experimental data. However, use of Eq. 13 resulted in only a very slight improvement in the fit of the predicted cumulative filtrate flow to experimental data compared to Eq. 11, therefore the linear expression for K (Eq. 11) is adequate to use for most practical purposes.



 FIG. 4. PREDICTED CUMMULATIVE FILTRATE VOLUME UTILIZING DERIVED EQUATIONS FOR TIME DEPENDENT SPECIFIC CAKE RESISTANCE
 (Curve A - linear dependence of specific cake resistance with time; Curve B - exponential dependence of specific cake resistance with time). Points represent experimental data.

#### FILTRATE FLOW THROUGH FILTER CAKES

#### Optimization

Filtration data using the filtration test cell is often used to determine optimum filtration cycles and obtain estimates of filtration area needed for larger scale filtrations. Filtration cycles are optimized generally on the basis of maximal filtration volume per unit time based on actual filtration time and time for backflushing and precoating the filter with new filter aid.

If a cleaning and set-up time of 5 min between cycles is assumed, a maximum average filtration rate of 0.72 mL/s can be obtained at a cycle time of 5 min when the volume of filtrate/cycle calculated using Eq. 15 is 432 mL. On the other hand, when the cleaning and set-up time is increased to 10 min, the maximum average filtration rate is reduced to 0.49 mL/s at a filtration cycle of 8.3 min when the volume of filtrate cycle is 520 mL. The same values for the average rate and optimum filtration time/cycle will be obtained using Eq. 12. These results indicate that rapid increase in resistance to filtrate flow by the filter cake with time of filtration would favor very short filtration cycles, and therefore a batch type filtration system would not be recommended for the types of filter cakes that were studied. Since each cycle will require a fresh precoat of filter aid, the frequent cycling necessary with these high resistance filter cakes will result in excessive consumption of filter aid and may make the process uneconomically feasible. Since precoat filter aid is 2.2g/cycle in the test system, the amount used will be 5 mg/mL of filtrate for precoat and 0.5 mg/mL added directly to the water for a total of 5.5 mg filter aid/mL of filtrate. Increasing the cycle time to 10 min when cleaning and set-up time is 5 min will result in an average filtrate flow of 0.66 mL/s and a total filter aid use of 4.2g/mL of filtrate.

#### CONCLUSION

The use of equations which utilizes constant filter cake specific resistance in the analysis of filtration where specific cake resistance increases with time of filtration, results in very poor agreement between calculated and experimental values. Filter medium resistance can be determined accurately by measuring filtrate flow through the precoated filter medium. An empirical equation which assumes linearly increasing filter cake resistance with time, resulted in good agreement between experimental and calculated filtrate volumes. Best agreement was obtained when K was assumed to vary exponentially with time, but the small improvement in fit may not justify the complexity of the process for determining the parameters for the exponential expression. Results from filtration tests on poultry chiller water and perlite filter aids suggest that constant pressure filtration is not recommended for this application.

# LIST OF SYMBOLS

Α	=	area of filtrate flow, cm <sup>2</sup>
Κ	=	time dependent factor for cake resistance, cm <sup>-6</sup>
M <sub>c</sub>	=	mass of solids in filter cake, g
Ρ	=	pressure drop across filtration cell, dynes/cm <sup>2</sup>
P <sub>c</sub>	=	pressure drop across filter cake, dynes/cm <sup>2</sup>
Pm	=	pressure drop across filter medium, dynes/cm <sup>2</sup>
R <sub>m</sub>	=	filter medium resistance, cm <sup>-1</sup>
U	=	filtrate flow velocity through cake, cm/s
V	=	cumulative filtrate volume, cm <sup>3</sup>
a	=	constant in exponential expression for K (initial value of K)
b	=	constant in exponential expression for K (limiting value of K)
с	=	concentration of suspended solids and filter aid in water, g/cm <sup>3</sup>
c ′	=	time constant in exponential expression for K, s <sup>-1</sup>
q	=	filtrate flow through precoated filtration cell, L/s
t	=	filtration time, s
t'	=	modified filtration time, s
α	=	specific cake resistance, cm/g
β	=	constant in linear expression for K, s/cm <sup>6</sup>
γ	=	constant in linear expression for K, (initial value of K)
μ	=	filtrate viscosity, g/cm(s)

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# A QUALITY-BASED INVENTORY ISSUE POLICY FOR PERISHABLE FOODS

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# ABSTRACT

Inventory issue policies that are based on elapsed storage time do not account for nonuniform deterioration within a perishable inventory stockpile. The literature on keeping quality of perishable food provides ample evidence of the direct relationship between storage temperature and quality deterioration. Because varying temperature condition may be encountered during storage and distribution of perishable foods, each item in an inventory stockpile may have a different deterioration history. The time-based first-in first-out (FIFO) and lastin first-out (LIFO) issue policies, in additional, to a quality-based issue criterion, the shortest remaining shelf-life (SRSL) issue policy were examined with a computer simulation of frozen broccoli stored in temperature varying conditions. The SRSL issue policy showed advantages over both the FIFO and LIFO policies in that items issued under the SRSL policy were of equal or higher average quality, with less quality variation at time of issue. The use of the SRSL issue policy in conjunction with full-history time-temperature indicators to predict temperature dependent changes in food quality is discussed.

#### **INTRODUCTION**

Perishable inventory management, the problem of determining the order in which items are dispersed from an inventory stockpile (e.g., inventory issue policy), has been addressed in the operations research literature. Earliest work in perishable inventory management was conducted without regard to specific applications, however, more recent investigations have focused on the class of

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problems related to the issuance of whole blood from a central blood bank (Pegels and Jelmert 1970; Brodheim *et al.* 1975; Nahmias 1982; Prastacos 1984). The issue policies that resulted from these investigations have been formulated based on the product age or elapsed time in storage, and have assumed that perishable inventory has a fixed storage life and that all items in the stockpile decay with the same uniform deterioration function.

The literature on the keeping quality of perishable foods provides ample evidence of a direct relationship between storage temperature and the rate at which quality changes occur (Van Arsdel *et al.* 1969; Labuza 1982; Jul 1985). Except for those products which exhibit the phenomena of reverse stability, most perishable foods deteriorate more rapidly at higher temperatures (Fennema *et al.* 1973). Thus, perishable foods that are exposed to variable temperature conditions will have deterioration functions that are dissimilar to those of items stored at a constant temperature. In situations where items within an inventory stockpile have nonuniform deterioration functions, the use of time-based issue policies may be inappropriate. An alternative issue policy would be to determine issue priority based on observed (or estimated) food quality rather than elapsed time in storage.

In this paper, the authors propose an inventory issue policy for perishable foods based on the remaining shelf-life calculated from the estimated food quality. Three explicit objectives are covered: (1) establishment of the objectives of an appropriate inventory issue policy for perishable foods; (2) development of an inventory issue policy based on food quality; and (3) comparison of the time-based and quality-based inventory issue policies for perishable foods.

# THEORETICAL AND EMPIRICAL CONSIDERATIONS

#### **Time-Based Inventory Issue Criterion**

The general approach to the solution of the inventory depletion problem has been to determine the mathematical constraints on the deterioration and field-life conditions such that either a last-in first-out (LIFO) or first-in first-out (FIFO) policy would yield the issue sequence that maximized the total life of the stockpile (Derman and Klein 1959; Bomberger 1961; Pierskalla 1967a and 1967b; Nahmias 1974; Albright 1976). A FIFO policy implies that items be issued in the order of decreasing age beginning with the oldest item in supply, whereas a LIFO policy implies that items be issued in the order of increasing age beginning with the youngest item on hand. Both the FIFO and LIFO issue policies are time-based management strategies in that items held in inventory are issued according to the total elapsed time that an item has been in storage (i.e. product age). The LIFO and FIFO issue policies have been shown to be optimal for perishable inventories that exhibit deteriorations functions (a utility function of a measure of deterioration verses storage time) of specific mathematical form (Derman and Klein 1958). Items that have utility functions which are concave are optimally issued under a LIFO policy, and items with functions that are convex are optimally issued with the FIFO policy (Pierskalla and Roach 1972). The optimal use of these policies hold only for stockpiles in which items deterioate with the same, uniform deterioration function. Neither the FIFO nor LIFO issue policies compensate for inventory items which undergo different rates of deterioration.

Traditionally, the FIFO issue policy is used to determine the sequence in which frozen and refrigerated inventories are dispersed from storage. However, since food quality has been observed to deteriorate along either a decreasing linear or decreasing concave function of time (i.e. zero- or first-order kinetic model), the LIFO policy would be considered optimal for the classic objective of maximizing the total life of the stockpile. The conflict between the widely used FIFO policy and the theoretically optimal LIFO policy suggests that maximizing the life of the stockpile may be an appropriate criterion for determing issue policy for perishable foods.

#### **Appropriate Issue Criterion for Perishable Foods**

Within the food distribution system, adherence to the FIFO issue policy would always require issue of the oldest items as measured from time of manufacture, while adherence to the LIFO policy would require issue of the most recently processed items. If a LIFO issue policy (the theoretically optimal policy) was placed into effect, it is likely that a portion of the accumulated product within the stockpile (the perpertual inventory) would not be distributed since any recently manufactured product would preempt the issue of any older item. The perpetual inventory would eventually become unsuitable for consumption and require disposal. The FIFO issue policy does not have the problem of stockpile obsolescence since the oldest items within the inventory would take priority for the first issue.

The use of the FIFO issue policy, as compared to the LIFO policy, will give rise to the issuance of items with a more consistent level of quality. One can argue that uniform product quality is substantially important in determining the issue priority of perishable foods. Given that a food item does not deteriorate to an unacceptable level of quality, a product of consistent quality (from one purchase to the next) would be preferred to a product with inconsistent quality. Certainly consumers of brand name foods continue to purchase these products because of the reasonable surety of consistent quality. While consistent product quality is often thought of as uniform manufacturing standards and careful selection of raw materials, the choice of which item is issue from inventory could negate whatever quality consistency has been attributed to a particular product brand. An inappropriate issue policy could inadvertently retain an item, that with an additional period of storage, would result in a product of grossly inferior quality when compared to quality of the item that was issued had the issued product remained in storage for the additional period of time.

A time-based issue policy (such as FIFO or LIFO) is unable to compensate for the increased deterioration due to elevated or irregular temperature exposures within the distribution system. Thus, the uniformity in the quality of the product distributed from the stockpile may be compromised. Ideally, an appropriate inventory policy for perishable foods would seek to management the quality of the inventory in such a way that the product is delivered to the consumer with the most consistent quality possible.

#### Food Quality Management and Time-Temperature Indicators

Product shelf-life is defined as the length of time a perishable product can be stored before the quality changes that accrue during storage result in an unacceptable product (International Institute of Refrigeration 1972). During the storage and distribution of perishable foods, temperature conditions may be encountered which give rise to increased (or decreased) rates of quality change. Thus, the threshold quality which defines a product's shelf-life would be achieved at an elapsed storage time dependent on the temperature history to which the product had been stored. Wells and Singh (1988) noted that for any level of quality between the initial and threshold quality levels, shelf-life,  $t_{Q,ref}$ , may be denoted as:

$$t_{Q,ref} = \lambda_{n,ref} + \omega_{n,ref}$$
(1)

where  $\lambda_{n,ref}$  and  $\omega_{n,ref}$  are the equivalent age and remaining shelf-life, respectively.

The equivalent age,  $\lambda_{n,ref}$ , represents the length of time that would be necessary to bring about the same level of quality if the product had been stored at an isothermal reference temperature; and the remaining shelf-life  $\omega_{n,ref}$ , represents the length of time for food quality to change from the observed level to the threshold level if the product is stored at the same reference temperature. The remaining shelf-life and equivalent age functions are complementary, and are both functions of quality.

Recent studies have confirmed that the response of full-history timetemperature indicators is correlated to storage related quality changes in several frozen and refrigerated foods (Singh *et al.* 1984; 1986; Singh and Wells 1986; Campbell *et al.* 1986). Furthermore, an explicit mathematical relationship, based on chemical kinetic theory, has been derived to predict changes in food quality from time-temperature indicator response (Singh and Wells 1987; Wells and Singh 1988). The quality-based interpretation of the shelf-life of perishable foods, combined with the general mathematical form of quality prediction from time-temperature indicator response provides a means by which product inventory could be issued based on the estimated quality rather than the elapsed storage time.

#### Shortest Remaining Shelf-Life (SRSL) Issue Policy

An alternative to the time-based issue policy would be to determine issue priority on the maximum expected remaining shelf-life as calculated from the estimated quality change. The precise life of a perishable food is unknown since the product may be consumed (eaten) at any time subsequent to issue. However, because food quality continues to change until the product is consumed, the maximum expected remaining shelf-life can be derived in terms of the amount of quality change that is acceptable prior to final consumption.

Establishing issue priority in the sequence from shortest to longest remaining shelf-life, the shortest remaining shelf-life (SRSL) issue policy, would retain the items with the greatest amount of quality reserve within the stockpile and expedite issue of the items which are less tolerant to additional, possibly abusive storage. In effect, the SRSL issue policy would allow inventory items that have undergone the greatest amount of quality change to move most rapidly through the remainder of the food distribution system. A qualitative comparison of the SRSL inventory issue policy to the FIFO and LIFO policies is given in the Appendix. The appended discussion is meant to further the reader's understanding on the relationship between issue priority and food quality.

# MATERIALS AND METHODS

#### **Issue Policy Evaluation and Computer Simulation Methods**

The inventory transaction records for frozen broccoli packed in cheese sauce were obtained from a cooperating cold storage warehouse in Central California. The transaction records covered the period of time from January 1, 1987 to May 1, 1987, and included the date and quantity of cases shipped and received, and the disposition of the inventory stockpile at the beginning of the investigation period. Computer simulation was used to determine the quality change during storage and to assign an initial quality level to the incoming replenishment inventory. Quality changes were estimated with a first-order kinetic model for a randomly fluctuating period-wise constant temperature (Labuza 1984). The kinetic parameters (activation energy of 16,120 cal/mole, and rate constant of 0.002639 day<sup>-1</sup> at -18 °C) were determined from shelf-life data for frozen broccoli

presented by Lai and Heldman (1982) assuming a shelf-life of 379 days at -18 °C. Quality score was indicated for a nonspecific attribute on a centesimal scale, with 100 indicating maximum quality.

The simulation program was written in Microsoft BASIC 3.0 and executed on an Apple Macintosh Plus microcomputer. A flow chart of the simulation algorithm is shown in Fig. 1. As shown, at the beginning of each simulated storage period the age of the stock on hand was increased by an increment of one day, and the quality change of the food inventory was calculated given the assumed temperature for previous period. Replenishment inventory was entered into on-hand stock and entries of product age (age zero upon receipt) and initial product quality (a random variable of specific variance) were assigned. The inventory was then prioritized for issue according to the FIFO, LIFO, or SRSL issue policy, and complete and/or partial lots were supplied until the demand for that period had been satisfied. The quantity of items demanded for shipment (or available for replenishment) were based on the transaction records from the warehouse. At the end of each period in which inventory transactions occurred, the simulation printed a record of the activity and recorded the storage age, quality level, and remaining shelf-life of the issued products.

At the end of each 120 day simulation trial, a simulation summary was printed that allowed comparison of the performance of the different issue policies. The performance parameters studied were the average and standard deviation of age, quality, and remaining shelf-life of the product at time of issue. The policy evaluation study consisted of examining the influence of variable temperature history and initial quality variation for replenishment stock on the performance of the FIFO, LIFO, and SRSL inventory issue policies. The initial quality of the replenishment stock was assigned as a random variable of specified variance. The variance of the initial quality assignments ranged from 0 to 40% of the centesimal quality scale in 5% increments. Each simulation trial used the same random number "seed" to assure that initial quality levels and temperature conditions would be the same.

# **RESULTS AND DISCUSSION**

#### Performance of SRSL Policy to Simulated Storage Conditions

For the 120 day period studied, there were 42 periods which required inventory shipment and 6 periods for which replenishment inventory was received into the warehouse. The inventory position (the number of cases in storage) after the daily shipping and receiving transactions is shown in Fig. 2. The number of cases stored in the stockpile ranged from 700 to 5400, and contained at least 3 to 5 manufacturing lots from which inventory could be supplied to satisfy shipment demand. The variable (daily, period-wise constant) temperature for the 120 day study is shown in Fig. 3.



FIG. 1. FLOWCHART OF THE DESIGN STRUCTURE OF THE SIMULATION ALGORITHM USED TO EVALUATE THE PERFORMANCE OF DIFFERENT POLICIES



FIG. 2. ON-HAND INVENTORY DURING THE 120 DAY SIMULATION OF THE FROZEN BROCCOLI STOCKPILE



FIG. 3. PERIOD-WISE CONSTANT, RANDOM TEMPERATURE FUNCTION USED TO SIMULATE A VARIABLE TEMPERATURE STORAGE FOR THE FROZEN BROCCOLI STOCKPILE

The average quality at time of issue as influenced by changes in initial product quality for fluctuating storage temperature is shown in Fig. 4. Changes in the initial quality of the product received into the stockpile have an effect on the average quality at time of issue. An analysis of variance classification of average quality as a factor of issue policy revealed no significant differences between average product quality at issue for the SRSL and FIFO policies. That is the choice of the SRSL issue policy showed no improvement in average issue quality as compared to the quality of product issued with the FIFO policy.



FIG. 4. AVERAGE QUALITY OF FROZEN BROCCOLI AT TIME OF ISSUE FOR THE SRSL, FIFO, AND LIFO ISSUE POLICIES AS INFLUENCED BY VARIATIONS IN QUALITY OF REPLENISHMENT STOCK (VARIABLE TEMPERATURE STORAGE)

The LIFO policy gave rise to issuance of products of significantly higher average quality than either of FIFO or SRSL policies. The slight improvement in average product quality for the LIFO issue policy was less noticeable under conditions of increasing initial quality variation. It would appear that it situations where initial product quality varied widely, the policy chosen for issue priority has little influence on the quality of the product which is dispersed from the stockpile. That is if an inferior product is received into storage no issue policy alone can remedy the issuance of poor quality product.

The variation in quality at time of issue as influenced by changes in initial quality level is shown in Fig. 5. The choice of inventory issue policy is seen to have an effect on quality variation of the issued products. The LIFO issue policy gives rise to the largest standard deviation of average quality for all conditions of initial product quality, and the SRSL issue policy gave the smallest issue quality variation. Both the SRSL and FIFO issue policies give rise to equal variations in quality at issue for initial quality variation up to 10% of the centesimal quality scale. For initial quality variations above 10%, the SRSL policy yielded decreased variation in average quality at issue.



FIG. 5. STANDARD DEVIATION OF THE AVERAGE QUALITY OF FROZEN BROCCOLI AT TIME OF ISSUE FOR THE SRSL, FIFO, AND LIFO ISSUE POLICIES AS INFLUENCED BY VARIATIONS IN QUALITY OF REPLENISHMENT STOCK (VARIABLE TEMPERATURE STORAGE)

The average remaining shelf-life for the frozen broccoli inventory at time of issue as influenced by initial quality variation for the variable temperature storage is shown in Fig. 6. The remaining shelf-life of a product represents the maximum length of time a product can be stored in the remainder of the distribution chain and consumer storage if the recommended reference storage temperature is maintained. Once an item has been issued from the stockpile, the remaining shelf-life for the product will continue to decrease to a greater or lesser extent in relation to the temperature during handling and storage in the remainder of the distribution chain. Storage temperatures above the reference conditions will cause an acceleration in the rate at which remaining shelf-life decreases, while temperatures below the reference will decrease the rate of change and actually extend the remaining shelf-life. Similar to the analysis of variance for issue quality, no significant difference in remaining shelf-life was found for the items that were issued under the SRSL and FIFO issue policies. This result would be expected as remaining shelf-life is a function of product quality.


FIG. 6. AVERAGE REMAINING SHELF-LIFE OF FROZEN BROCCOLI AT TIME OF ISSUE FOR THE SRSL, FIFO, AND LIFO ISSUE POLICIES AS INFLUENCED BY VARIATIONS IN QUALITY OF REPLENISHMENT STOCK (VARIABLE TEMPERATURE STORAGE)

The variation for the average remaining shelf-life at time of issue for the simulated temperature scenario is shown in Fig. 7. The variation in remaining shelf-life is seen to be strongly influenced by inventory issue policy. In the range of initial quality variation between 0 and 35% the LIFO policy gives rise to the greatest variation, and at initial quality variation above 35% the FIFO policy gave results in the largest variation in remaining shelf-life.

The difference between the remaining shelf-life variation for the LIFO and FIFO issue policies can be explained by examining the issue priority for each policy. The LIFO policy will issue the items most recently placed into the stockpile, whereas the FIFO policy will issue the items which have remained in the stockpile for the longest duration. When products are issued under a LIFO policy a small quantity of product will remain in storage (perpetual inventory) and will not be issued as long as shipment demand does not exceed the total quantity available in the stockpile. In periods of high demand, however, when all recent products have been issued a LIFO policy will issue items from the perpetual inventory. These items will have deteriorated considerably compared to the recently replenished inventory because of the length of time they have been in storage. Thus the variation in remaining shelf-life would be increased for a LIFO policy since products are being issued alternatively between recent and perpetual inventory items.

In situations where items received into the stockpile have a large variation in initial quality (advanced quality deterioration more likely), retaining these items in the stockpile for the time necessary for them to become the oldest items in the



FIG. 7. STANDARD DEVIATION OF THE AVERAGE REMAINING SHELF-LIFE OF FROZEN BROCCOLI AT TIME OF ISSUE FOR THE SRSL, FIFO, AND LIFO ISSUE POLICIES AS INFLUENCED BY VARIATIONS IN QUALITY OF REPLENISHMENT STOCK (VARIABLE TEMPERATURE STORAGE)

stockpile (as would occur in the case of a FIFO policy) would further increase the deterioration and exaggerate any variations in quality or remaining shelf-life which already exists. For the FIFO and LIFO issue policies there appears to be a point in the quality change of a product beyond which quality variation would not be reduced by the choice of one issue policy over the other.

Additional simulation trials were conducted for situations where the inventory was stored at -18 °C constant temperature. The discussion above applies to these results as the findings for average and variation of average for quality and remaining shelf-life were similar to the variable temperature storage condition.

Table 1 shows the mean values for the standard deviation of remaining shelflife of the simulation trials conducted for both the constant (-18 °C) and variable (Fig. 3) storage temperatures. An analysis of variance for the issue policy classification at each level of initial quality variation shows a significant difference (p < 0.05) between the variation of remaining shelf-life for the SRSL and FIFO policies in cases where initial quality variation was greater than 25%. At initial quality variation of 30%, the least significant difference (LSD) is 5.33, which is less than the difference between the standard deviation of average remaining shelf-life for the SRSL and FIFO issue policies (i.e. |32.91 - 38.98| >5.33), but at an initial quality variation of 25% the difference between policies was not greater than the LSD (i.e. |28.70 - 32.60| < 5.96). This would indicate that in situations where the initial quality of replenishment inventory varied more than 25% there would be a statistically significant reduction in the variability of the remaining shelf-life at the time the item was shipped from storage if the SRSL issue policy was used instead of the FIFO policy.

FFERENCES (p OF VA	< 0.05) BET RIATION IN	WEEN POLIO REPLENISHN	CIES AT DIFF	ERENT LE
Initial Quality	Standard D	eviation of Av	erage Remaing S	Shelf Life
Variation	SRSL	FIFO	LIFO	LSD
0a	22.50b	22.50	44.06	5.46c
5	20.84	20.84	41.24	5.81
10	20.97	21.03	30.12	6.01
15	22.62	23.25	37.94	6.02
20	25.78	27.25	37.96	6.07

TABLE 1.
TABULATION OF THE STANDARD DEVIATION OF AVERAGE REMAINING SHELF-LIFE
FOR THE SRSL, FIFO, and LIFO ISSUE POLICIES, AND THE LEAST SIGNIFICANT

a Percent variation in initial quality of replenishment inventory

32.60

38.98

46.20

54.18

39.34

42.18

46.40

51.90

5.96

5.33

4.64

4.04

<sup>b</sup> Standard deviation of average remaining shelf life at issue

28.70

32.91

38.53

45.10

25

30

35

40

<sup>c</sup> Fisher least significant difference (LSD) at 95% confidence

In situations where the initial quality variation ranges to some maximum level, the conditions under which the SRSL issue policy offers a reduction in the variation of remaining shelf-life can be expressed in terms of the probability plot shown in Fig. 8. The intersection of the probability curve and the line at the 50% significance level (Fig. 8) indicates that in situations where initial quality varies up to 25%, the use of the SRSL policy would yield a significant reduction in remaining shelf-life variation in about half the shipments as compared to the FIFO policy. From such a relationship, a warehouse manager could begin to assess importance of issue policy as it relates to the dissatisfaction a consumer would experience from products with inconsistent remaining shelf-life and quality. Development of the operating characteristic curves that assess the consumer's and producer's (warehouse manager's) risk is left for future researchers.

For this simulation study, the FIFO and SRSL policies issued products of about the same quality in all cases of initial quality variation. The SRSL policy has the advantage over the FIFO or LIFO issue policies in that the SRSL policy



FIG. 8. PROBABILITY OF A SIGNIFICANT DIFFERENCE BETWEEN THE STANDARD DEVIATION OF THE AVERAGE REMAINING SHELF-LIFE FOR FROZEN BROCCOLI ISSUE UNDER THE SRSL AND FIFO ISSUE POLICIES

gave rise to the issue of items with more consistent quality. It would appear that for a perishable inventory such as that modeled in this simulation the quality based SRSL issue policy would give rise to issuance of items with more consistent quality when compared to the items distributed with a time-based issue policy. The SRSL issue policy, thus, would be considered the most appropriate issue policy for perishable foods.

## SRSL Issue Policy and Shelf-Life Dating

A crucial decision in perishable food management is the removal of a product from inventory. Currently, manufactured product are stamped with a "pull date" to reflect a conservative estimate of the product's shelf-life. Procedures require that items stored beyond their "pull date" be removed from inventory and destroyed or otherwise eliminated from their originally intended use. As a result of the conservative "pull dates" significant amounts of high quality product are disposed without having quality changes sufficient to deem the product unacceptable by a consumer.

Because the SRSL issue policy bases decisions of issue priorty on the remaining shelf-life, the SRSL policy could also be used to determine when an item is to be removed from inventory. Remaining shelf-life is calculated as the difference between the current estimate of quality and an unacceptable quality threshold. In principle, this means that the date to remove product from inventory (the "removal date") would be reached (or have passed) when the remaining shelflife of an item was zero (or negative). A remaining shelf-life of zero would imply that there is no difference between the quality of a product in its current state and the threshold quality level that is unacceptable to a consumer, and a negative remaining shelf-life means that a product has quality changes that are in excess of what is considered to be tolerable. Use of the SRSL policy would base the "removal date" on the estimated quality changes that have occurred within the food, rather than conservative estimate of shelf-life as does the "pull date". The "removal date" method would not give rise to inventory removal unless warranted by the magnitude of the estimated quality change.

# CONCLUSION

Full-history time-temperature indicators can be used to provide useful information regarding irregular and time-varying temperature conditions during storage and distribution of perishable foods. Methods have been established which utilize indicator response to predict changes in food quality thus allowing inventory management decisions to be based on food quality rather than elapsed storage time. A quality-based inventory management strategy, such as the SRSL issue policy, has the potential to enhance the consistency in the quality of perishable foods delivered to the consumer without compromising the overall level of quality. Improvements in consistency of perishable food quality by implementing a quality-based issue policy could heighten the reputation products which have established consistent quality standards in raw material selection and process quality control.

# APPENDIX

## **Qualitative Comparison of Different Issue Policies**

The following examples illustrate the use of different inventory issue policies in the management of an inventory stockpile with uniform and nonuniform deterioration. The example of an inventory stockpile with nonuniform deterioration calls attention to the inability of a time-based inventory management scheme to compensate for differences in quality deterioration between items within the same supply. The examples are meant to further the reader's understanding of the relationship between issue priority (issue policy) and the quality and quality variation of items at time of issue.

**Perishable Inventory Example** — Uniform Deterioration. Consider an exmaple of a perishable inventory in which all items deteriorate according to the single deterioration function shown in Fig. A1. The function of quality score versus time could represent the changes in a sensory attribute monitored during isothermal storage. All items were received into the stockpile with a quality score 10 to age 0, and since the items are stored at the same isothermal condition, the quality score changes according to the deterioration function (shown in Fig. A1) as each item ages within the stockpile.



FIG. A1. EXAMPLE OF A UNIFORM QUALITY DETERIORATION FUNCTION FOR ITEMS IN A PERISHABLE INVENTORY STOCKPILE

The initial disposition of the inventory stockpile consists of four items (A,B,C,D) with ages 0, 1, 2 and 3 periods, respectively. That is, item A has just been manufactured and received into the stockpile (age 0 and quality score 10), and item D had been placed in storage three periods earlier (now age 3 and quality score 3). Demand for the inventory is assumed as one item per period, with no stockpile replenishment. Thus, in four consecutive periods all items in the stockpile will have been issued. Once an item has been issued from the stockpile it is unavailable for issue in subsequent periods.

Table A1 shows a complete tabulation of ages and quality levels for each of the four items during the life of the stockpile. If items are prioritized according to the FIFO policy (oldest item available in the stockpile issued) the issue order for the consecutive periods is D, C, B, A. In the case when all items have the same uniform deterioration function, the SRSL issue policy has the same issue order as the FIFO policy. When the stockpile items are issued according to the LIFO policy (youngest item available in the stockpile issued) the issue order is A, B, C, D. The average quality and range of quality at issue for the FIFO, LIFO, and SRSL issue policies are given in Table A2.

Earlier researchers (cf. Bomberger 1961) considered the LIFO policy to be the optimal issue policy for items which deteriorate with a concave deterioration

	Age and Quality of Items at Issue									
	Per	iod 0	Per	iod 1	Per	iod 2	Per	iod 3		
Item	Age	Quality	Age	Quality	Age	Quality	Age	Quality		
Α	0	10.00	1	6.00	2	4.00	3	3.00		
В	1	6.00	2	4.00	3	3.00	4	2.25		
C	2	4.00	3	3.00	4	2.25	5	1.75		
D	3	3.00	4	2.25	5	1.75	6	1.25		

#### TABLE A1. TABULATION OF AGE AND QUALITY FOR ITEMS IN A PERISHABLE STOCKPILE WITH UNIFORM DETERIORATION

TABLE A2. COMPARISON OF AVERAGE QUALITY AND RANGE OF QUALITY OF ITEMS ISSUED FORM A STOCKPILE WITH UNIFORM DETERIORATION USING DIFFERENT ISSUE POLICIES

		Inven	tory	Issue Po	olicy	
FIF		FO	LI	FO	SF	RSL
Period	Item	Quality	Item	Quality	Item	Quality
0	D	3.00	Α	10.00	D	3.00
1	С	3.00	В	4.00	С	3.00
2	В	3.00	С	2.25	В	3.00
3	А	3.00	D	1.25	Α	3.00
Avg. Q	Quality	y 3.00		4.375	5	3.00
Quality	y Rang	ge O		8.75		0

function (Fig. A1), the optimal policy was defined as one that maximized and total field life of a stockpile. Maximizing the field life of a stockpile amounts to maximizing the average quality of a product at issue, as a higher average product quality at issuance infers (on the average) the longer period of time the item can be stored once it has been issued (a longer field life). The average quality upon issue for the items issued with a LIFO issue policy is 4.375, the highest average quality upon issue for any of the compared policies. This policy, however, is undesirable because the range of quality score at issue is 8.75, the widest fluctuation observed. The FIFO issue policy yields an issue order with an average quality of 3.00 for the entire stockpile, but there is no fluctuation in the quality score at time of issue. In the case of uniform deterioration, the SRSL issue policy yielded the same results as the FIFO policy. Thus, either the FIFO or SRSL issue policy is preferred for issuance of perishable inventories in which all items

deteriorate with the same concave deterioration function since these policies give rise to issuance of products with the most consistent quality.

**Perishable Inventory Example – Nonuniform Deterioration.** Consider the example of the same perishable inventory as discussed above, where one of the items (item B) deteriorates with a slightly different deterioration function than the remainder of the items (Fig. A2). Again the initial inventory stockpile consists of four items (A, B, C, D) of ages 0, 1, 2 and 3 periods, but item B deteriorates with a function dissimilar to that of items A, C, and D (see Fig. A2). The nonuniform deterioration may have resulted from an increase in storage temperature for item B during the interval between the periods 1 and 2.



FIG. A2. EXAMPLE OF A NONUNIFORM QUALITY DETERIORATION FUNCTION FOR ITEMS IN A PERISHABLE INVENTORY STOCKPILE

Table A3 shows the tabulation of the ages and quality of each item during the life of the stockpile, and Table A4 shows the issue order and quality comparison for the different issue policies. The FIFO and LIFO issue policies produce the same issue order as seen in the case of uniform deterioration, but the average quality at issue and range of quality at issue is different because of the accelerated rate of quality change for item B. The average quality at issue for all the policies was lower compared to the case of uniform deterioration, but since the FIFO and LIFO issue policies are based on storage time, they could not compensate issue order for the nonuniform deterioration experienced by item B. The SRSL issue policy, however, recognized the nonuniformity and altered issue priority so that item B was the second item dispersed from the stockpile.

		Age	and	Quality	of Ite	ms at Is	sue	
	Per	iod 0	Per	iod 1	Per	iod 2	Per	iod 3
Item	Age	Quality	Age	Quality	Age	Quality	Age	Quality
Α	0	10.00	1	6.00	2	4.00	3	3.00
В	1	6.00	2	2.75	3	1.75	4	1.00
С	2	4.00	3	3.00	4	2.25	5	1.75
D	3	3.00	4	2.25	5	1.75	6	1.25

#### TABLE A3. TABULATION OF AGE AND QUALITY FOR ITEMS IN A PERISHABLE STOCKPILE WITH NONUNIFORM DETERIORATION

TABLE A4. COMPARISON OF AVERAGE QUALITY AND RANGE OF QUALITY OF ITEMS ISSUED FORM A STOCKPILE WITH NONUNIFORM DETERIORATION USING DIFFERENT ISSUE POLICIES

		Inver	tory	Issue Po	olicy	
	FI	FO	LI	FO	SF	RSL
Period	Item	Quality	Item	Quality	Item	Quality
0	D	3.00	Α	10.00	D	3.00
1	С	3.00	В	2.75	В	2.75
2	В	1.75	С	2.25	С	2.25
3	Α	3.00	D	1.25	Α	3.00
Avg. Q	Quality	2.69		4.06		2.75
Quality	y Rang	ge 1.25		8.75		0.75

The reason for issuance of item B as the second item under the SRSL issue policy can be understood by examining the quality of the items in the stockpile at each period of issue demand (every period in this example). The item that has the lowest quality value would be the item with the shortest remaining shelf-life and would take highest priority for issue. Initial examination of the stockpile (period 0) revealed item D had the lowest quality score and thus was issued first since its remaining shelf-life was shortest (see Table A3 to compare quality scores of items A, B, C, and D at period 0). In period 1 the items remaining in the stockpile (items A, B, and C) were surveyed to determine which item had experienced the greatest cumulative quality change. Item B had a quality level of 2.75, compared to quality levels of 3.00 and 6.00 for items C and A, respectively. Thus item B was chosen for issue because it had the shortest remaining shelf-life of all the items within the stockpile. By similar reasoning item C was chosen for issue in period 2.

As shown in Table A4, the SRSL policy performed better in the case of nonuniform deterioration than either the FIFO or LIFO policies. The SRSL policy gave rise to the product with the highest average quality and the smallest range of quality fluctuation at issue. The quality based SRSL issue policy called attention to the observation of the nonuniform deterioration in item B and expedited issue by altering the sequence in which stockpile items were issued. As a result of the SRSL issue policy, item B was disbursed from storage more rapidly and the variation in the range of quality at issue was reduced.

A characteristic of the SRSL policy is that it retains the same issue order as the FIFO policy when all items deteriorate in the same manner, but compensates for nonuniform deterioration when necessary. Time-based inventory issue policies, such as FIFO or LIFO, are unable to distinguish quality differences between items that deteriorate with uniform and nonuniform deterioration functions. When the scenarios of these two examples are compared, use of the FIFO or LIFO issue policy results in a wider variation of product quality, with at best the same average quality at time of issue compared to the SRSL policy. An issue policy based on the shortest remaining shelf-life criterion has the potential to enhance consistency in the quality of perishable foods delivered to the consumer.

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# STORAGE STABILITY OF MODEL SUCROSE OR SALT ADDED O/W EMULSIONS THROUGH STEADY SHEAR AND CREEP RHEOLOGICAL MEASUREMENTS

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## ABSTRACT

Model oil-in-water emulsions prestabilized with xanthan gum and propylene glycol alginate have been studied in the presence of salt and sucrose. Emulsions to which sucrose was added appeared to develop a network structure due to the high effective xanthan gum concentration at the interface and the high continuous phase viscosity caused by sucrose. Salt emulsions appeared to have enhanced stability due to electrostatic effects. Both the sucrose and the salt emulsions showed an increase in stability with an increase in concentration.

## INTRODUCTION

The formulation of commercial oil-in-water salad dressings involves the use of many ingredients whose primary function include flavoring and texturization, rather than stabilization. For example vinegar, sugar, salt, mustard flour are largely added for microbiological stability or as flavoring agents. Gums such as xanthan and propylene glycol alginate on the other hand are included to stabilize these emulsions (Gladwell *et al.* 1985; Glicksman 1982).

Emulsions are thermodynamically unstable or metastable (Tadros and Vincent 1983). Breakdown processes of emulsions include flocculation and coalescence. Flocculation depends on the sum of attractive forces and repulsive forces. Attractive forces are caused by London-van der waals interactions and repulsive forces are generated from electrical double-layer interactions as well as steric mechanisms. Repulsive forces in oil-in-water emulsions arise due to adsorption of surface active agents at the interface between oil and water (Friberg 1976). Proteins, phospholipids and polysaccharides can be the sources of these ions. In the presence of dissolved electrolytes, as in the case of NaCl, an electrical double layer develops around each droplet.

Journal of Food Processing and Preservation 12(1989) 293-308. All Rights Reserved. ©Copyright 1989 by Food & Nutrition Press, Inc., Trumbull, Connecticut. The adsorption of surface active macromolecules at the liquid/liquid interface is a slow process and equilibrium is only reached after long times of the order of several hours or days. At equilibrium, adsorbed polymer molecules form loops and tails extending into the solution phase. Thus, when two oil droplets approach each other, through Brownian diffusion, a barrier is generated due to the presence of these adsorbed layers. This steric stabilization can act to prevent flocculation and provides a mechanial barrier to coalescence. The latter case occurs in particular with many adsorbed protein molecules, which denature upon adsorption followed by collapse at the interface leading to a rigid film formation (Phillips 1981).

The presence of adsorbed polymer layers at the oil-water interface that generate the steric interaction can also be of sufficient dimension to affect the van der Waals and electrostatic contributions (Tadros and Vincent 1983). The adsorption of macromolecules leads to the redistribution of ions in the double layer around a droplet which can effect the range and magnitude of the repulsion forces around the droplet. Also, the spatial extensions of polymer molecules of modest molecular weights are usually comparable to, or greater than, the range of the London-van der Waals attraction between droplets (Napper 1983).

One common biopolymer used as a stabilizer is propylene glycol alginate. Propylene glycol alginate consists of blocks of mannuronic and guluronic acid where a large fraction of the carboxyl groups contain propylene glycol groups. The propylene glycol groups are hydrophobic so, this organic derivative produces a molecule having both hydrophobic and hydrophilic portions making them good surface active agents. Propylene glycol alginate does not appear to act as a film former (Shotton and White 1963 and Acton and Saffle 1972). Propylene glycol alginate is most frequently used in combination with xanthan gum, rather than alone, and has been shown to give long term stability (Glicksman 1982).

Xanthan is an anionic gum composed of a cellulose backbone with trisaccharide branches on alternating anhydroglucose units. Intermolecular associations among these polymer chains result in the formation of a complex network of entangled rod-like molecules (Glicksman 1982). Xanthan gum molecules get adsorbed at the oil-water interface by forming a microgel and partly by acting as an emulsifier. Prud'homme and Long (1983), have also shown evidence which suggests that xanthan gum molecules get adsorbed at the oil-water interface in the presence of a surfactant.

The presence of electrolytes such as salt has been shown to affect the charge stabilization between droplets in an oil-in-water emulsion, (van den Tempel 1963) and resulted an increase in emulsion viscosity with salt addition. Above xanthan gum concentration of 0.15%, the addition of salt in the concentration range of 0.01-0.07%, increases the viscosity of xanthan gum solutions. The

electrolyte appears to stabilize the helical conformation of xanthan gum, reducing the repulsion between carboxylate ions on the triasaccharide side chains. Above a concentration of 0.07% salt, however, no further increase in viscosity is observed (Glicksman 1982).

Elfak *et al.* (1980), studied the effect of sucrose and milk on several types of gums. They found that sucrose limited hydration of nonionic gums, guar and locust bean, which resulted in reduced viscosity.

The objective of this study is to use steady shear and creep rheological techniques to study the stability of a model salad dressing formula consisting of oil, water, vinegar, and the gums xanthan and propylene glycol alginate to which either sucrose or salt, in various concentrations was added.

# **MATERIALS AND METHODS**

All of the ingredients used in this study were supplied by Thomas J. Lipton Inc. Xanthan and propylene glycol alginate (P.G.A.), were from Kelco, Division of Merck & Co.; sucrose came from the Imperial Sugar Co.; salt (NaCl), Culinox 999, from the Morton Salt Co.; soybean oil, Admiration Salad Oil, was purchased from the Supreme Oil Co. and vinegar, white distilled 100 grain, was purchased from Speaco Corporation. Deionized water was used for the aqueous phase. All formulations contained 38% soybean oil, 9% vinegar, 0.2% xanthan and propylene glycol alginate gums. Sucrose varied from 6% to 20% at five levels and salt varied from 0.5% to 3% also at five different levels. The amount of water was adjusted depending on the amount of sucrose and salt.

The preparation of the emulsions proceeded as follows: dry ingredients, xanthan gum, propylene glycol alginate and the appropriate variable ingredient (sucrose or salt) were mixed for 15 min with a propeller mixer after the proper amount of each one was slowly added to the aqueous phase of water and vinegar. For maximum hydration of the gums, the aqueous solution was left shaking 24 h, on a shaker. Soybean oil was added in the proper amount and the mixing of the two phases was done with a Hobart mixer for 10 min. Emulsification was induced with a horizontal Charlotte Colloid Mill with gap setting of 0.008 in and by running the sample through the mill three times. The emulsions were stored in 500 mL salad dressing jars at room temperature, which varied between  $23^{\circ}-27^{\circ}C$ .

Steady shear rate experiments were conducted using the cone and plate geometry of the Rheometrics Fluids Rheometer with a cone angle of 0.0198 radians and a gap of 50 microns. Shear rates used in the experiments were 0.35, 1.0, 10, 100, and  $350 \text{ s}^{-1}$ . Creep experiments were conducted using Rheometrics Stress Rheometer equipped with 50 mm parallel plates. A stress of 5 dynes/cm<sup>2</sup> was chosen for the sucrose and salt-added emulsions.

During creep testing when the emulsion behaves as a viscoelastic liquid there is an instantaneous elastic compliance and a newtonian compliance. In terms of these compliances, a typical creep curve for a viscoelastic liquid can be represented as: (Sherman 1983)

$$J(t) = J_{0} + \Sigma J_{i} (1 - e^{-t/\tau_{i}}) + J_{N}$$
(1)

where  $J_0$  and  $J_N$  represent the instantaneous compliance and the newtonian compliance, respectively.  $\tau_i$  in this equation is a retardation time and a viscosity can be determined using  $\eta_i = \tau_i/J_i$ . The rheological parameters can be obtained considering that the total compliance is equal to (Sherman 1963, 1966, 1983);

$$J(t) = J_{0} + \Sigma J_{i} (1 - e^{-t/\tau_{i}}) + t/\eta_{N}$$
(2)

where  $t/\eta_N = J_N$ .

Rearranging Eq. (2) and taking the natural logarithms of both sides gives:

$$J_2 e^{-t/\tau_2} + J_3^{-t/\tau_3} + \dots \ll J_1 e^{-t/\tau_1}$$
 (4)

then Eq. (3) reduces to:

$$\ln [J_0 + \Sigma J_1 + t/\eta N - J(t)] = \ln J_1 - e^{-t/\tau_1}$$
(5)

Equation (5) would allow the determination of  $J_1$  and  $\tau_1$  and subsequently  $E_1$  and  $\eta_1$ .

It is possible to define:

$$Q = [J_0 + J_i + t/\eta N - J(t)]$$
(6)

A plot of ln Q vs t on semi-log coordinates will yield an intercept of ln  $J_1$  and a slope of  $-1/\tau_1$  using the linear portion of the curve. When curvature is observed the analysis is continued to fit the rest of the data by replotting ln  $[Q - J_1e^{-t/\tau_1}]$  vs time. This time the linear region will have a slope of  $-1/\tau_2$  and an intercept of ln  $J_2$ .

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# **RESULTS AND DISCUSSION**

# Analysis of Steady Shear Data

Changes in viscosity with storage time observed for emulsions to which sucrose and salt were added, at shear rates of  $.35 \text{ s}^{-1}$ ,  $10 \text{ s}^{-1}$  and  $100 \text{ s}^{-1}$ . are show in Fig. 1 and 2, respectively. The low shear rate of  $.35 \text{ s}^{-1}$  allows discrimination among different degrees of flocculation while the high shear rate experiments where all the flocculates can be assumed to have essentially broken down reflect the resistance caused by individual droplets or tightly bound doublets, triplets and multiplets.



FIG. 1. VISCOSITY VS AGING TIME AT THREE SHEAR RATES FOR SUCROSE EMULSIONS



FIG. 2. VISCOSITY VS AGING TIME AT THREE SHEAR RATES FOR SALT EMULSIONS

Oil-in-water emulsions that undergo flocculation show initially an increase in viscosity on aging due to the formation of aggregates which entrap continuous phase between the droplets (Sherman 1967). Viscosity subsequently decreases due to drainage of continuous phase associated with the interfacial film. When the film disappears completely coalescence occurs.

With sucrose emulsions (Fig. 1) the viscosities increased with increasing sucrose concentrations at each shear rate. This is best exemplified by comparing the formulation without any sucrose to those with various levels of sucrose. Viscosity increased as a function of aging time as well, up to a critical time. Replacing sucrose with water increased the continuous phase viscosity increasing diffusion time, significantly. While coalescence is significantly slower the film viscosity increases and results in enhanced aging stability. Furthermore, since sucrose competes with xanthan and propylene glycol alginate gums for available water the effective concentration of the gums at the interface is expected to increase and thus increase the viscosity and elasticity of the film than what it would otherwise have been.

At the shear rate of  $.35 \text{ s}^{-1}$  viscosity decay with time was most pronounced. Since aggregates are broken into individual droplets the effect of aging time on flocculated systems can be detected. On the other hand, the high shear viscosities reflect a system of individual oil droplets and few strongly aggregated groups of doublets or triplets.

Since the viscosities at the high shear rates changed little over the time period of study it appeared that the size distribution of the oil droplets have not changed significantly. The correlation between high shear viscosities and particle size data has been confirmed in at least two earlier studies (Carrillo and Kokini 1988; Kokini and Carrillo 1989). This suggested that only a slight amount of, if any, coalescence had occured for the sucrose emulsions over the two month study period.

The linear shape of the log  $\eta$  vs t curve for the sucrose emulsions at the .35 s<sup>-1</sup> can be simulated with a first order kinetics of the type;

$$\ln \eta = \ln \eta_0 - \mathrm{kt} \tag{7}$$

that could be useful to understand breakdown of aggregates as well as predict changes in viscosity with time. Table 1 shows the value of the rate constants, k, as well as the correlation of determination  $R^2$  over a 60 day viscosity period.

The salt emulsions also showed no increase in viscosity with aging time when apparent viscosities were measured at shear rates of  $10 \text{ s}^{-1}$  and  $100 \text{ s}^{-1}$ . However, a significant increase was observed when the measurements were conducted at a shear rate of .35 s<sup>-1</sup> suggesting flocculation of oil droplets. However, the aggregates formed had a short lifetime and were weaker than those which have formed in the case of sucrose emulsions since, they were not apparent at shear rates of  $10 \text{ s}^{-1}$  and  $100 \text{ s}^{-1}$ .

Furthermore, at aging times smaller than 15 days rheological properties of salt emulsions were essentially unchanged. However, after 15 days the emulsions became increasingly non-Newtonian as the level of added salt was decreased.

Using the same type of first-order kinetics for viscosity versus aging time for the salt emulsions, the rate constants in Table 2 were obtained. From these values it was clear that the decay constants were a function of salt concentration and became progressively smaller as the concentration of salt was increased. This clearly showed that increasing the concentration of salt stabilized the emulsions.

$\dot{y} = 0.35 \text{ sec}^{-1}$		
Sucrose conc. (%)	<u>K (sec<sup>-1</sup>)</u>	R <sup>2</sup>
6.0	0.0193	0.99
9.0	0.0200	0.99
12.5	0.0160	0.99
15.0	0.0146	0.99
20.0	0.0099	0.99

 TABLE 1.

 RATE CONSTANTS (K) AND COEFFICIENT OF DETERMINATION (R<sup>2</sup>)

 OF SUCROSE EMULSIONS

TABLE 2. RATE CONSTANTS (K) AND COEFFICIENT OF DETERMINATION (R<sup>2</sup>) OF SALT EMULSIONS

$\dot{y} = 0.35 \text{ sec } -1$		
Salt conc. (%)	K (sec <sup>-1</sup> )	R <sup>2</sup>
0.5	0.0203	0.99
1.0	0.0156	0.99
1.5	0.0159	0.99
2.0	0.0128	0.98
3.0	0.0092	0.99

### **Analysis of Creep Data**

Typical creep curves obtained in this study are shown in Fig. 3 and 4 for 3% salt and 6% sucrose levels as a function of aging time. There is essentially no recovery when the applied stress is removed, demonstrating that the emulsions behave as viscoelastic liquids and that any structure has broken down within the applied stress. Also from this curve, one can see that there is no significant instantaneous compliance.



FIG. 3. CREEP CURVE FOR THE 3% SALT EMULSION AFTER 8 DAYS STORAGE

All of the creep curves in this study were shown to be simulated by a single elasticity  $E_1$  and two viscosities  $\eta_1$  and  $\eta_N$ . The parameters  $\eta_1$ ,  $E_1$  and  $\eta_N$  obtained as a function of concentration and aging time are shown in Tables 3 and 4 for sucrose and salt emulsions, respectively. The maximum value of creep parameters  $E_1$ ,  $\eta_1$  and  $\eta_N$  for these concentrations are circled on these tables for clarity.

Analysis of Sucrose Emulsions. In all cases  $E_1$ ,  $\eta_1$  and  $\eta_N$  increased with concentration at all aging times. This was consistent with steady shear viscosity measurements. As more sucrose was added water was being removed in order to keep the phase ratio constant. This resulted in an increase in the effective gum as well as the sucrose concentration in the continuous phase which affected viscoelastic properties of the continuous phase. The increasing values in both  $\eta_1$  and  $E_1$  reflected this increasing concentration of xanthan and propylene glycol alginate at the interface.

Another factor that must be considered is the confirmation and rearrangement of the molecules at the oil-water interface (Vernon-Carter and Sherman 1980, 1981, 1981). Initially, the gum macromolecules adsorb randomly on the oil droplets with their hydrophobic groups. This leads to the existence of long tails extending into solution. These tails promote interdroplets and interaggregate interactions. Particularly, xanthan's helical conformation appears to allow a high degree of interaction between polymer chains which results in the formation of a network of molecular aggregates (Pettit 1982). This resulted in large  $E_1$  values due to network structure. Also  $\eta_N$  was largely due to the existence of relatively large aggregates that resulted in large friction forces during flow.



FIG. 4. AVERAGED CREEP CURVES FOR 6% SUCROSE EMULSIONS FROM 0 – 60 DAYS STORAGE

With increasing aging time however, the extended molecules will tend to rearrange on the oil droplets and the tails will become loops and trains. This should reduce the amount of interlinking between droplets. This phenomenon would explain the increase in  $\eta_1$  with time for a few days while  $E_1$  and  $\eta_N$  decreased during this same time period.

<b>n</b> .

		6%			%6			12.	5%		15%			20%	
Time Days	EI	۲ ۲	Ľ	EL	<b>۲</b>	× ۲	Еl	۲	Ľ	Еl	۲ '	۲ ۲	Еl	۲ '	z K
	$\left( \right)$				101	1050		<i>LLL</i>	647	6.60	979	2245	40	1922	6762
0	6.01	482	RR/		666	ACAT							)		$\mathbf{D}$
2	9.3	(572)	743	10.0	632	1012	12.8	781	1414	17.1	955	2113	38	1398	4929
2	9.4	517	554	9.3	640	842	11.8	804	1109	15.8	966	1857	37	0960	5855
œ	7.8	510	491	7.5	538	673	11.0	645	1043	13.0	793	1647	2.9	1422	4928
15	5.4	329	381	7.2	434	552	9.6	625	982	13.6	810	1638	28	1606	4891
30	3.6	210	168	4.5	281	241	7.4	434	455	9.2	512	826	20	1031	2660
45	3.0	171	84	4.2	280	96	5.2	344	261	6.8	408	303	15	688	1408
60	2.3	143	47	2.7	131	57	2.9	214	113	4.2	264	165	11	482	731

Note: The largest values for each parameter are circled.

	EMULSIONS
ABLE 4.	OF SALT-ADDED
	<b>P PARAMETERS</b>
	CREE

%	z L	B6 5	698	696	606	385	248	180
Э.С	۲	607	542	653	556	423	326	342
	Еl	7.2	6.8	۲. (	7.5	6.3	4.2	3.7
%	z z	612	434	456	388	244	157	119
2.0	2	470	588	(00)	384	372	311	91E
	E	6.0	9 · 2	(e . B	5.2	4.9	4.0	2.6
%	<b>Z</b>	612	413	379	333	194	126	78
1.5	7	488	(499)	463	386	351	169	218
	гı	9.3	5.7	5.0	5.1	3.0	2.2	3,1
~	Ľ	(47B)	400	384	300	204	102	14
1.03	1 1	254	418	470	371	277	513	198
	ш	6.3	5.1	5.0	4.7	3.8	2.8	1.9
%	r r	518	467	340	235	125	76	40
0.5	<b>7</b> 1	425	(433)	412	331	221	1.64	133
	E1	9	6.2	5.0	5.0	2.6	2.1	2.3
	Time Days	0	4	ω	15	30	45	60

Note: The largest parameters for each aging emulsion are circled.

#### **O/W EMULSIONS**

As aging time increased all of the creep parameters decreased steadily due to drainage of the film between droplets, resulting from gravitational effects. A first order approximation for the change in film thickness can be obtained using (Tadros and Vincent 1983);

$$\frac{1}{b} = \frac{1}{b_0^2} + \left[\frac{4\delta^4 t}{\eta \varrho g a^5}\right]^{\frac{1}{2}}$$
(8)

where  $b_0$  is the film thickness at zero time,  $\eta$  is the viscosity of the liquid forming the film,  $\varrho$  is the liquid density, a is the radius of the droplet and  $\delta$  is the interfacial tension. The equation suggests that the smaller the droplet the quicker the value of b to decrease with time. In this work, it was not possible to obtain data in relation to drop size distribution. However, in a parallel study in this laboratory (Carrillo and Kokini 1988), it was shown that drop size distribution tends to increase larger values with increasing continuous phase viscosity. This would explain the increasing change in magnitude of  $E_1$  and  $\eta_N$  with increasing sucrose concentration. Conceivably then, the larger droplet size distribution results in emulsions which undergo slower film drainage compared to those with relatively smaller droplet size distributions.

So, as the drainage occurred, there was not only a viscosity loss due to loss of entrapped continuous phase (decreasing  $\eta_N$ ) but loss of stabilizing molecules from the adsorbed layer of the interface (decreasing both  $E_1$  and  $\eta_1$ ) as well.

Analysis of Salt Emulsions. The viscoelastic parameters  $E_1$ ,  $\eta_1$  and  $\eta_N$  for the salt emulsions in Table 4 appeared to have two different types of behaviour depending on concentration. The parameters for the first three concentrations; .5%, 1% and .15% appeared to have little or no relationship to concentration, most notably for the first 15 days. Above this concentration, however, creep parameters increased with increasing concentration. With the first three emulsions,  $E_1$  decreased from the largest initial value continuously through 45 days. The values of  $\eta_1$  for the first three concentrations except at the 1.0% salt concentration showed a maximum value at four days. The change in  $\eta_1$  at a 1.0% salt concentration can be considered to be a random experimental variation. The 2.0% and 3.0% salt emulsions, on the other hand, tend to show an increase in creep parameters with an increase in concentration above 1.5%. The  $\eta_1$  values for the 2.0% salt emulsions increased until 8 days rather than 4 days as in the case of .5%, 1.0%, 1.5% salt.

When polyelectrolytes are present in the interface it is possible to have electrosteric stabilization which is a combination of both electrostatic and steric stabilization. The electrosteric component may originate from a net charge on the droplet surface due to the polymer attached to the surface. In a study of polystyrene lattices with polyelectrolyte stabilizer polyacrylate (Napper 1983), the effect of salt on the flocculation of the particles was shown. This is consistent with this work which showed increasing stabilization with increasing salt concentration. At the low concentration of salt, .5%, 1% and 1.5%, the creep parameters behaved as they did for the sucrose-added emulsions. That was, the value of  $E_1$  and  $\eta_N$  were highest initially while  $\eta_1$  increased with time for a short period before decreasing again. It was possible that these emulsions have undergone the same type of change as was described for the sucrose emulsions except on a small scale due to the less effective amount of xanthan and propylene glycol alginate present because of an enchanced surface concentration. After 15 days, the drainage of the film controls and the separation in parameters reflected continuous phase viscosity increase with concentration.

It appeared that the 2% and 3% salt emulsions fall in the ionic concentration range for electrosteric stabilization to occur. That is, considering the changes in  $E_1$  with time, it appears that the development of a network structure due to interdroplet interaction was delayed or even prevented. This could be attributed to the electostatic repulsion operating to prevent close approach of droplets. With time however, more and more interdroplet interaction has occurred due to diffusional effects and rearrangement of the polyelectrolyte. Rearrangement could have affected the charge distribution leading to an apparent decrease in some droplets for interaction.  $E_1$  adn  $\eta_1$  increased subsequently to their maximum value. At aging times greater than 8 days, the decrease in creep parameters again occurred as well described for the sucrose emulsions. The fact that the Newtonian compliance decreased with time indicated that the aggregates entrapped a large amount of continuous phase thus increasing the effective phase ratio. This would be the case since during electrostatic repulsion the individual droplets will be furthest apart.

In conclusion it can be said that both sucrose and salt have a stabilizing effect on oil/water emulsions prestabilized with xanthan gum and propylene glycol alginate. The sucrose emulsions appeared to develop a network structure due to the high effective xanthan gum concentration at the interface and the high continuous phase viscosity caused by sucrose. The salt emulsions appeared to have enhanced stability due to electrostatic effects. Both the sucrose and salt emulsions showed an increase in stability with an increase in concentration.

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#### **O/W EMULSIONS**

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# **RHEOLOGICAL AND GELATION PROPERTIES OF REDUCED SALT MEAT EMULSIONS CONTAINING POLYPHOSPHATES**

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# ABSTRACT

The gelation patterns and rheological properties of poultry meat emulsions prepared with average and reduced NaCl levels (2.5% and 1.5%, respectively) and with three polyphosphates (0.5% tripolyphosphate = TPP, hexametaphosphate = HMP, or sodium acid pyrophosphate = SAPP) added to the reduced salt emulsion were studied.

Using 2.5% NaCl resulted in the highest yield stress value as compared to all the other treatments; SAPP addition significantly reduced yield stress of the raw emulsion. The relationship between the shear rate and shear stress of the raw emulsions was nonlinear and appears to resemble the Bingham pseudoplastic behavior.

Plots of the modulus of rigidity (G) versus cooking temperature indicated differences in gelation patterns and maximum G for the different treatments. The highest G was observed in the 2.5% NaCl emulsion followed in decreasing order by the emulsions containing 1.5% NaCl + 0.5% SAPP, 1.5% NaCl + 0.5%HMP, 1.5% NaCl, and the 1.5% NaCl + 0.5% TPP treatment.

# **INTRODUCTION**

Since high sodium intake in the Western diet has been shown to lead to hypertension and subsequent cardiovascular disease, various recommendations have been made to limit dietary salt intake (Sebranek *et al.* 1983). However, sodium chloride (NaCl, salt) is an essential ingredient in processed meat products contributing to product flavor, providing binding (through protein extraction) and controlling microbial growth (Ingram and Kitchell 1967). Despite these advantages, for health reasons, it appears necessary to reduce the sodium chloride content of processed meats. Reducing or substituting NaCl requires precise understanding of the effects caused by each of the new factors introduced (Maurer 1983).

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Several ways to reduce the sodium content of processed meat products without adversely affecting their quality and shelf-life have been proposed. They include a slight sodium chloride reduction, partial replacement of NaCl with other chloride salts, replacement with non chloride salts and altering processing methods with any of the above (Terrell 1983).

Partial substitution of NaCl with non chloride salts, such as phosphates, has been reported to assist in overcoming some of the adverse effects of NaCl reduction (Knipe et al. 1985; Sofos 1986). However, differences in the ability of the different phosphates to improve various functional properties have been reported. Tripolyphosphate (TPP), for example, was observed to enhance water holding capacity to a greater extent than hexametaphosphate (HMP) and pyrophosphate in low salt (1%) poultry meat emulsions (Shults and Wierbicki 1973). Phosphates have also been reported to improve the overall acceptability of reduced salt meat products. Seman et al. (1980) reported that 0.13% tripotassium phosphate in combination with 1.25% NaCl resulted in bologna as acceptable as bologna containing 2.5% NaCl; however, with no phosphate supplement the 1.25% product suffered extensive cooking losses and was judged unacceptable. Barbut et al. (1988) also reported that phosphates significantly improved the texture and acceptability of low salt (1.5%) turkey frankfurters; sodium acid pyrophosphate (SAPP) was found to provide better results than produced by HMP and TPP.

Phosphate actions have been suggested by some to be due to a nonspecific effect reflecting the increase in ionic strength of the aqueous phase of the muscle, where phosphate anions align with cation groups on the protein molecules resulting in increased repulsion between the muscle fibers allowing more space for water. In addition, specific effects have also been reported. Certain phosphates, such as pyrophosphate, can participate in the dissociation of actomyosin to actin and myosin (Sofos 1986). However, so far no clear relationship between phosphate activity and their effect on functional properties in meat products has been established.

Knowledge of the rheological behavior of comminuted meat systems is necessary not only for the design of machines, processes and operations but also for the control of end-point characteristics and consumer acceptability (Rizvi 1981). Using both a visiometer and a plastograph, Hamm and Reisner (1967, 1968) reported that the rheological properties of beef muscle homogenates and a model sausage formulation were affected by added water, pH and the addition of polyphosphates. Garbatov and Garbatov (1974) presented various equations and correlations for the flow of Russian sausage meat batters under various conditions of stress. Correlation equations were presented for predicting pressures required to force sausage meat batters through pipes or nozzles.

Knipe et al. (1985) investigated the effects of sodium hydroxide, separately or in combination with various polyphosphates, on beef and pork emulsion pH,

emulsion stability, texture and apparent viscosity. They used the Poiseuille equation for calculating viscosity assuming Newtonian behaviour of the emulsion. However, previous studies (Toledo *et al.* 1977) reported meat emulsions to behave as Bingham-Pseudoplastic fluids. Lahtinen (1984) studied the flow characteristics of model food emulsions (not containing meat) by partly substituting potassium and magnesium chloride for NaCl. The Herschel-Bulkley model was found suitable to describe these flow properties. Barbut and Mittal (1988) also reported that the Bingham Pseudoplastic behavior best fitted the behavior of reduced sodium poultry meat emulsions containing either potassium or magnesium chloride.

Recently, continuous rigidity scanning was reported to be a more sensitive method than batch techniques for detecting sol-gel transformation transitions in protein systems. Rheological monitoring has the potential of becoming an extremely valuable and widely used instrumental method for evaluating and understanding the physical properties of heat-set gel formation and the effect of salts on gel formation (Hamann 1987).

Thus, the objectives of this study were to determine the effect of three phosphates, currently used by the meat industry, on the gelation and rheological properties of sensitive meat emulsions.

# MATERIALS AND METHODS

### **Ingredients and Meat Emulsions**

Five different poultry meat emulsions were prepared in duplicate for two separate trials. The treatments included 2.5% NaCl, which is a commonly used level in such a product (Terrell 1983), and reduced salt emulsions containing 1.5% NaCl or 1.5% NaCl with 0.5% TPP, HMP, or SAPP (Stauffer Chemical Inc., Washington, PA). Mechanically deboned turkey meat (MDTM) was obtained from a local processing plant. The MDTM was kept frozen up to 3 weeks at -20 °C prior to use. Proximate analysis of the raw meat as determined in triplicate was 65.1% moisture, 18.2% fat, 14.3% protein and 1.0% ash (AOAC 1980).

A nonvacuum bowl cutter (Hobart, model 84142, Troy, OH) was used to chop the batters (1.5 kg each). Batters were chopped for 3.5 min at high speed: final chopping temperature did not exceed 7 °C in any of the cases. The emulsions were vacuum tumbled using a table top tumbler (Lyco, Columbus, WI) for 30 s at 0.15 atm to remove small air bubbles. The pH of the raw emulsions was determined in duplicate (Chemcadet J-598, Cole Parmer, Chicago, IL).

## Rheology

The rheological parameters were determined using a Haake-type rotary viscometer (model RV3, Haake, Berlin, West Germany), and the SV-II measuring system. The gap between the cup and the rotor was very small (0.3 mm). The viscous drag of a rotating body, immersed in the emulsion, was converted to shear rate (1/s) by multiplying it by a factor of 0.89. Similarly, scale readings were converted to shear stress (Pa) by multiplying the values obtained by a factor of 34.94, provided by the manufacturer.

## **Modulus of Rigidity**

Continuous evaluation of the modulus of rigidity (G) during thermal processing of the meat emulsion was performed by using a thermal scanning rigidity monitor (TSRM) similar to the one described by Montejano et al. (1984). The TSRM consisted of a cylindrical jacketed chamber with a hollow cylinder held in the centre by upper and lower removable guides. The chamber was mounted on the base of a model TM Instron Universal Testing machine (Instron Engineering Corp., Canton, MA). The central cylinder was fitted to a 500 kg compression/tension load cell mounted on the crosshead of the machine. The TSRM was connected to a heating water bath (Haake model E-3). The rate of heating was manually controlled at 0.5 °C/min from 20 to 75 °C using a variable auto transformer Varian V20 (General Radio Co., Cambridge, MA) connected to a heating coil immersed in the water bath. Water circulated through the outer chamber and centre cylinder to insure uniform temperature conditions in the meat emulsion during cooking. The raw meat emulsion was loaded around the central cylinder between the upper and lower guides. Care was taken to insure that the emulsion was well packed and no visible air bubbles were present. Prior to the beginning of the test the guides were removed. No flow due to gravity occurred because of the large enough yield strength of the emulsion.

At 2 min intervals a cyclic force (from the upward-downward cyclic motion of the crosshead at .5 mm/min) was applied to the samples producing a small variable cyclic deformation in the samples. The samples were free to expand during cooking in order to avoid any influence of swelling on the deformation reading. Internal temperature was monitored by T-type thin thermocouples inserted into the specimen.

The peak to peak force (F) was calculated from the data recorded on the chart paper. The shear modulus or modulus of rigidity was calculated as the ratio of maximum shear stress ( $\tau$ ) to maximum shear rate ( $\dot{\gamma}$ ). Mathematically it was calculated by:

$$G = F \ln (R_2/R_1) / (2 \pi LD)$$

where G is shear rigidity modulus, Pa; F is force amplitude, N;  $R_2$  is inside radius of outer cylinder, 2.2975 cm;  $R_1$  in outside radius of inner cylinder, 1.503 cm; D is displacement amplitude, 0.0005 m; and L is the average length of the annular volume occupied by the meat emulsion. The (L) was determined by dividing the area of the cooked meat emulsion in contact with the inner cylinder by  $2\pi R_1$ .

Statistical analyses were performed using the Statistical Analysis System (SAS 1982). The General Linear Model and Nonlinear procedures for regression analysis, ANOVA procedure for analysis of variance and Duncan's multiple range test for ranking were used.

# **RESULTS AND DISCUSSION**

### **Raw Emulsion Viscosity**

The relationship between shear rate and shear stress for the different treatments is illustrated in Fig. 1. The relationship is nonlinear, and appears as Bingham pseudoplastic behavior. The shearing rate tended to increase faster than the shearing stress, with a yield value. Metzner (1961) suggested that particles in an emulsion are randomly oriented initially and become increasingly more aligned as shear is applied. The contibution of particle interactions to the apparent emulsion viscosity was reported to be reduced when shearing stress increased. All the emulsions required the application of a certain shearing force before any noticeable flow took place. On a molecular level, Bingham materials are envisioned as consisting of a three dimensional network when at rest. Forces applied to this network could be resisted up to a certain point, and then, the network breaks down, and the flow becomes essentially pseudoplastic. No marked pH differences among the treatments were observed. Both NaCl treatments had the same pH (6.35); HMP did not affect the pH; TPP slightly increased the pH to 6.45 and SAPP addition decreased the pH to 6.25. These results are in agreement with previously reported data (Knipe et al. 1985; Barbut et al. 1988). These relatively small pH differences ( $\pm 0.1$  pH units from the NaCl treatment) are not believed to play the major role in the viscosity differences observed, rather the type and concentration of the salt ions involved seems to be the most influential. However, additional research is required to precisely determine the pH effect at these ranges.

The general power law (Herschel-Bulkley) model with yield stress, was used to fit the data:

 $\tau = T_0 + b \dot{\gamma}^n$ 



**EMULSION RHEOLOGY** 

In this model  $\tau$  is shear stress, Pa;  $\dot{\gamma}$  is shear rate, 1/s; T<sub>0</sub> is yield stress, Pa; b is consistency coefficient, Pa.s<sup>n</sup> and n is flow behavior index. Table 1 summarizes these rheological parameters for the different treatments. This equation was found to be an adequate model for describing the flow behavior of meat emulsions in the range of the experimental conditions used.

#### TABLE 1. EFFECT OF SODIUM CHLORIDE AND PHOSPHATES<sup>(1)</sup>, ON THE HERSCHEL-BULKLEY EQUATION CONSTANTS<sup>(2)</sup>

<u>Parameters</u>	Estimate	Asymptotic <u>error</u>	95% asymptotic confidence <u>interval</u>	MSSE	<u>r2</u>	df for <u>error</u>
2.5% NaCl						
T s b n	586.80a 45.36b 0.752a	32.7 14.3 0.07	520 - 654 16 - 75 0.6 - 0.9	2674	.998	27
1.5% NaCl						
T <sub>o</sub> b n	380.38b 234.77a 0.442b	98.9 74.4 0.06	179 - 583 83 - 387 0.3 - 0.6	4098	.997	29
1.5% NaCl -	+ 0.5% TPP					
Te b n	424.48b 64.44b 0.663a	31.7 16.5 0.06	360 - 490 31 - 98 0.55 - 0.78	1689	.99	27
1.5% NaCl -	⊦ 0.5% HMP					
To b n	504.82b 53.02b 0.743a	34.2 15.2 0.066	435 - 575 22 - 84 0.61 - 0.88	2822	.998	27
1.5% NaCl -	+ 0.5% SAPP					
T b n	281.77c 52.80b 0.695a	19.9 9.7 0.04	241 - 323 33 - 73 0.61 - 0.78	777	.969	27

Results are the average of both trials. MSSE = mean sum of square of errors,

df = degree of freedom, and  $r^2$  = coefficient of determination.

<sup>(1)</sup>Phosphates: TPP = Tripolyphosphate; HMP = Hexametaphosphate;

SAPP = Sodium acid pyrophosphate

 $^{(2)}\tau = T_0 + b \dot{\gamma}^n$ ,  $T_0$  is yield stress, Pa: b is consistency coefficient, Pa.s<sup>n</sup>:

and n is the flow behavior index.

<sup>a,b,c</sup> - means of a parameter with the same letter are not significantly different at 95% level.

The confidence interval at the 95% level for  $T_0$  were 291 to 580 Pa, for b they were -11 to 191.0 Pa.s<sup>n</sup>, and for n they were 0.50 to 0.82 (Table 1). These values indicate that the  $T_0$  for the emulsion containing 1.5% NaCl + SAPP was significantly lower than for the other treatments. Thus, SAPP decreased the Bingham behavior of the emulsion. Similarly, the  $T_0$  value for the control (2.5% NaCl) was significantly higher than the other treatments. Thus, low NaCl (1.5%) reduced the  $T_0$  value. The "b" value of the meat emulsion with 1.5% NaCl was significantly higher than the other emulsions containing either 2.5% NaCl or 1.5% NaCl plus the various other phosphates. The "n" value was between 0 and 1, indicating pseudoplasticity. Similarly, the "n" value of meat emulsion containing 1.5% NaCl was significantly lower than the other treatments. According to Garbatov and Garbatov (1974) more stable formulations tended to have higher values for the "b", lower values for "n", and larger values for the yield stress.

Toledo *et al.* (1977) determined the constants for sausage meat batters of varying composition containing 2.25% NaCl using a tube viscometer. They reported values of  $T_0$  ranging from 0 to 28 Pa, values of "b" between 14 and 858 Pa.s<sup>n</sup>, and "n" values between 0.104 and 0.722. Bianchi *et al.* (1987) reported  $T_0$ , "b" and "n" values of beef, pork, and chicken muscle homogenates, which were 3.5 - 11.7 Pa, 6.9 - 14.9 Pa.s<sup>n</sup>, and 0.31 - 0.37, respectively. Addition of NaCl (2%) to the muscle homogenates changed  $T_0$ , "b" and "n" to 17.6 - 26.4 Pa, 27.2 - 31.6 Pa.s<sup>n</sup>, and 0.23 - 0.25, respectively. All the latter parameters are significantly lower than the values reported in our study. Comparing our results to the work done by Toledo *et al.* (1977) indicates that our yield values are much larger, but the values of "b" and "n" are within their ranges. These differences may be due to the use of the rotational viscometer in our study.

The rheological parameters were also determined using the Casson equation:

$$\sqrt{\tau} = \mathrm{K}_0 + \mathrm{K}_1 \sqrt{\gamma}$$

 $K_0$  is Casson flow limit and  $K_1$  is Casson viscosity. These constants are given in Table 2. The  $K_0$  value for the meat emulsion containing 1.5% NaCl + 0.5% SAPP was significantly lower than the other treatments. The  $K_1$  value for the emulsion with 2.5% NaCl was significantly the lowest. Feher *et al.* (1981), also using a Haake-type rotary viscometer and the SV measuring system, reported these constants for meat emulsions:  $K_0 = 13.1$  to  $14.1 \sqrt{Pa}$ , and  $K_1 = 0.88$  to  $1.13 \sqrt{Pa.s.}$  Their emulsions were prepared with 1.6% NaCl, and the shear rate was from 1.5 to 44 l/s. In our study shear rates from 1.8 to 57 l/s were used. Thus, the difference in rheological parameters may be due to the NaCl and phosphate content as well as the type of meat used. Feher *et al.* (1981) concluded that the higher value of  $K_0$  was associated with an extremely uniform structure with ordered distribution.
Parameter	<u>Estimate</u>	PR >[T]	SEE	MSSE	<u>r²</u>
2.5% NaCl					
K., Pa	22.42a	**	0.28	0.59	0.978
K1, VPa.s	2.1886	**	0.06		
1.5% NaCl					
K₀√Fa	23.22a	**	0.35	1.03	0.973
K1 VFa.s	2.596a	**	0.08		
1,5% NaCl +	0.5% TPP				
K <b>₀√</b> Pa	19.66a	**	0.23	0.409	0.986
K1 VFa.s	2.29a	**	0.05		
1.5% NaCl +	0.5% HMP				
K. VPa	20.62a	**	0.27	0.558	0.984
K1, Pa.s	2.50a	**	0.06		
1.5% NaCl +	0.5% SAPP				
K. VFa	15.67b	**	0.18	0.243	0.993
Ki VPa.s	2.44a	**	0.04		

#### TABLE 2. EFFECT OF SODIUM CHLORIDE AND PHOSPHATES<sup>(1)</sup>, ON CONSTANTS OF THE CASSON EQUATION<sup>(2)</sup>

Results are the average of both trials. SEE = Sum of squares of error;

MSSE -Mean sum of squares of error.

(1) Phosphates: TPP = Tripolyphosphate; HMP = Hexametaphosphate;

SAPP = Sodium acid pyrophosphate

<sup>(2)</sup> Model is  $\sqrt{\tau} = K_0 + K_1 \sqrt{\gamma}$  where  $k_0$  is Casson flow limit and  $K_1$  is Casson viscosity.

\*\* - PR > [T] is 0.0001.

<sup>a,b</sup> - Means of a parameter with the same letters are not significantly different at 95% level.

Burge and Acton (1984) used a capillary extrusion viscometer to determine the rheological parameters of meat batters. The batters exhibited pseudoplastic behavior with the apparent viscosity increasing as the fat level decreased. The batter's fat level did not significantly affect the coefficient of shear rate, but yield

stress significantly decreased with the increase in fat levels from 11 to 18%. They reported  $T_0$  values from 148 (26% fat) to 587 (11% fat) Pa, b values from 245 to 442 Pa.s<sup>n</sup>, and n values from 0.37 to 0.42. Our values of  $T_0$  were within this range, but "b" values were significantly lower and "n" values were significantly higher at the 90% level. Differences may be due to the measuring method and type of meat.

Knipe *et al.* (1985) reported that beef and pork emulsion viscosity was significantly (P < 0.001) affected by the addition of polyphosphates. Viscosities were ranked as: the control with 0.75% salt (593cp) > 0.75% salt + 0.5% HMP (552 cp) > 0.75% salt + SAPP (545 cp) > 0.75% salt + TPP (517cp). TPP further (P < 0.05) reduced emulsion viscosity below that of HMP and SAPP. In a single phosphate study, Hargett *et al.* (1980) also reported SAPP to effectively reduce emulsion viscosity. As indicated by these authors such a viscosity reduction can be very beneficial in manufacturing emulsion type sausages since the risk of excessive heat build up during chopping, which subsequently can cause an emulsion breakdown, can be minimized.

The finely minced meat, water and salt mixture forms a complex colloidal system consisting of three phases. The first is an aqueous solution of the salt and water soluble proteins, the second phase is comprised of the muscle and connective tissue particles, while the third phase is the minced fat tissue. In this system, the dispersed particles are bound to one another by coagulation forces resulting from the excess of their surface energy (Feher *et al.* 1981). Hamm and Reisner (1967, 1968) concluded that the batter's consistency was highly dependent on the degree of swelling of the particles in the system, and there was a strong relationship between the water holding capacity (WHC) and the flow properties of the system.

If the bonds within the aggregates are extremely strong, the system may display a yield value. When flocs, for example are aggregated in a dispersion, the reaction of the aggregates to shear can result in shear thinning flow. At low shear rates, the aggregates may be deformed but remain essentially intact. As the shear rate is increased, the aggregates may be broken down into individual flocs, decreasing friction and therefore viscosity.

When the particles in a dilute suspension are electrically charged, an electrical double layer of ions surrounds each particle. In concentrated emulsions, the double layers may interact or overlap, and the mutual repulsion may cause an increase in viscosity. This is known as the second electroviscous effect and was reported to be inversely proportional to the ionic strength (Darby 1984). This electroviscous effect is also known to increase as particle size decreases. Because of interactions between the extent of the double layer and the entrapment of the dispersed phase in floculated particles, a viscosity minimum, at a specific electrolyte concentration, may be observed. This effect is probably due

to a complex interaction between electrostatic repulsion, entrapment of the continuous phase as a function of the extent of the double layer, and crowding effects of high concentrations.

Apparent viscosity was calculated using:

$$U_{app} = T_0 / \dot{\gamma} + b \dot{\gamma}^{n-1}.$$

Figure 2 illustrates the relationship between  $U_{app}$  and shear stress. Apparent viscosity decreased with the increase in shear rate and shear stress and a significant change was observed due to the use of SAPP. Since the relatively small pH differences (6.35  $\pm$  0.1) among the treatments are not believed to be the main cause for the viscosity differences observed the type of phosphate ions present seemed to play a major role in affecting the apparent viscosity. However, additional research is needed in order to determine the exact effect of specific phosphate ions on the rheological properties, especially in light of the widely believed concept that most polyphosphates are rapidly broken down into short chain compounds upon addition to the meat (Sofos 1986).

Knowledge of the flow properties of minced meats and sausage emulsions is especially essential to the technology of mincing, transportation through pipes and stuffers. In addition, understanding of the rheological behavior of the comminuted meat system is necessary for the control of end point characteristics and consumer acceptability of the products.

#### Gelation - Modulus of Rigidity

Plots of modulus of rigidity (G) versus internal temperature of the meat emulsions containing the various phosphates are shown in Fig. 3. Each point on the curve is the average of both trials. The emulsion with 2.5% NaCl (control) had the highest maximum rigidity value (12.2 kPa) followed by the emulsion with 1.5% NaCl + 0.5% SAPP (11.9 kPa). The emulsion with 1.5% NaCl had a maximum G value of 9.9 kPa, the emulsion containing 1.5% NaCl + 0.5% HMP had a maximum G value of 10.5 kPa, while the lowest G was observed in emulsion with 1.5% NaCl + 0.5% TPP (9.1 kPa).

During heating the 1.5% NaCl emulsion showed a small but linear increase in rigidity up to 55 °C indicating that a stiff protein matrix was continuously developing. In the temperature range of 55 to 63 °C, there was a rapid increase in G exhibiting a maximum rigidity value of 9.9 kPa at 63 °C. This was followed by a rapid linear decrease from 69 °C, reaching a final value of 5.1 kPa at 78 °C. The transition at 55 °C has been attributed to myosin and the one at 63 °C to collagen and to the sarcoplasmic proteins (Wright *et al.* 1977).



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The other four emulsions (2.5% NaCl, and 1.5% NaCl with either 0.5% TPP, HMP or SAPP) showed very similar G-temperature relationships. In all four cases, there was a small linear increase in rigidity up to 53-55 °C followed by rapid G increase up to 68–73 °C where it reached peak values. This indicates the formation of stable, stiff, and elastic matrix structures typical of heat induced protein gels. With the further increase in temperature, there was no increase in G value up to 80 °C. Thus, these four curves provided two transition temperatures: one at 53–55 °C and the other at 68–73 °C. The rapid increase in rigidity starting at 54–56 °C, observed in the TPP treatment, can be attributed to shifting the myosin denaturation to a higher temperature. The general pattern observed here for the denaturation of the control (2.5% NaCl) is similar to the pattern reported for a turkey paste containing 2.5% NaCl (Montejano *et al.* 1984), showing transition temperatures to occur at about the same points. The same trend was observed when 2.5% NaCl was replaced with 50% isoionic strength of KCl or MgCl<sub>2</sub> in poultry meat emulsions (Barbut and Mittal 1988).

Wu *et al.* (1985) studied transitions occurring during the gelation of meat emulsion using a constant heating rate of 1 °C/min also by TSRM. Three transition temperatures were observed at 38, 46 and 60 °C. Rigidity increased beyond 46 °C, was related to the formulation of a stable network and a strong gel structure. According to Patana-anake and Foegeding (1985), shear modulus sharply decreased from 20–35 °C due to the melting of pork fat; and G remained relatively constant over the 40–50 °C range. The major rigidity transition occurred around 54–57 °C and continued to increase for the remainder of the heating period. Schweid and Toledo (1981) also reported transition points in meat batter to occur at 33–36 °C and at 57–67 °C. They suggested that these temperatures represent the points where insolubilization and solubilization of collagen occur, respectively. Foegeding and Ramsey (1987) determined the rigidity changes during heating of meat batters containing various carrageenans and xanthan gum. There was a slight variation of G values from 34 to 58 °C but major variations in rigidity developed from 58 to 70 °C due to the gums.

This study showed similar results for poultry meat emulsions. However, the G values and transition ranges were different probably due to the different type of meat used. The effect of various phosphate ions was also observed on the peak value of rigidity, transition ranges and rate of rigidity change.

Table 3 summarizes the statistics of the G values for the different treatments. G is highly correlated with temperature for all the treatments. Generally, G increases with the increase in temperature. The average values of G in increasing order for the various emulsions are 2.5% NaCl (6.5 kPa); 1.5% NaCl + HMP (5.0 kPa); 1.5% NaCl (4.98 kPa); 1.5% NaCl + 0.5% SAPP (4.7 kPa); and 1.5% NaCl + 0.5% TPP (3.9 kPa). A similar trend was observed when peak values of G were considered.

<u>Treatment</u>	Mean (1)	<u>Std.</u> Dev.	<u>Min.</u>	Max.	Correlation of G <u>with Temp.</u>	<u>N</u> (3)
2.5% NaCl	6523.3	4061.3	1860	12244	0.924 (0.0001)(3)	42
1.5% NaCl	4984.7	3412.2	1963	9928	0.618 (0.0001)	43
1.5% NaCl + 0.5% TPP	3868.5	3569.7	447	9136	0.896 (0.0001)	44
1.5% NaCl + 0.5% HMP	5007.8	3947.7	940	10521	0.885 (0.0001)	45
1.5% NaCl + 0.5% SAPP	4711.1	4180.9	2082	11969	0.904 (0.0001)	39
Overall					0.825 (0.0001)	213

TABLE 3. STATISTICS OF THE MODULUS OF RIGIDITY (G), Pa

(1) Results from both trials.

<sup>(2)</sup> Number of observations.

<sup>(3)</sup> Probability of the hypothesis that there is no correlation

The correlation of G with temperature for all the treatments was 0.825, and the correlation of G with treatment was 0.08. The following regression model can be applied:

G	= -5295 +	193.17 (tem)	p.) + 57.48 (treatment)
PR> [T]	0.0001	0.0001	0.028
Standard error of estimate	565	9.0	53

 $[r^2 = 0.682; df error = 210]$ 

Rigidity modulus is an indication of the rigidity and stiffness of the material and not its degree of elasticity. It has been suggested that the toughening reaction of muscle fibers to heat could be due to tightening of the network of protein structure during denaturation (Hamm and Deatherage 1960). On cooking, coagulation of myofibrillar proteins is believed to result in toughening. The coagulation and increase in rigidity occurred between 30 °C and 40 °C and were completed at 55 °C (Hamm and Deatherage 1960). Thus, at low temperatures, where coagulation and other structural changes occurred, temperature plays a vital role in regard to the extent of textural changes. Harris (1976) reported that by the time meat temperature reached 60 °C, the majority of the sarcoplasmic and myofibrillar proteins were denatured. The denaturation was said to be accompanied by a very large decrease in WHC and an increase in sample rigidity. Cooking to temperatures above 60 °C increasead the rigidity of the myofibrillar structure and caused meat fibers to contract along their longitudinal axis. The contraction started at 55–60 °C, with the largest rate of contraction occurring at 70–75 °C. Cooking resulted in softening the connective tissues, such as collagen, and toughening the other meat proteins. However, process conditions during cooking will largely determine whether the toughening effect of heat will be offset by such a tenderizing effect.

The processes taking place during the cooking of a meat batter are complex. However, changes taking place during the transition of a gel meat system to a solid system and the rate of change can be measured. The kinetics of gelation and its effect on the physical properties of the finished product can then be studied. In this experiment, the three phosphate salts evaluated were found to affect gelation and rheological properties of the poultry meat emulsions differently. Such changes should be taken into account when attempts are made to partially replace sodium chloride in meat emulsions.

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# **COMPUTER CODES AND THEIR APPLICATION**

# CORRELATION COEFFICIENT AND CONFIDENCE INTERVAL ESTIMATION BETWEEN SENSORY AND OBJECTIVE MEASUREMENTS UNDER DESTRUCTIVE TESTING USING COMPUTER SIMULATION

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# ABSTRACT

Estimation of correlation between sensory and instrumental data for food products is used in research and industrial applications. Often the measurement techniques destroy the sample making it impossible to collect more data on the same item. This results in a correlation based not on individual observations but rather on the average of the data, leading to an erroneous and artificially high correlation. A method using maximum likelihood techniques and conditional simulation is proposed for estimating the correlation in this case.

# **INTRODUCTION**

The situation may arise when it is desired to estimate the correlation coefficient between two measurements that cannot both be observed for a given item. The measurement of X on a sample item may require that the item be destroyed or completely exhausted in the measurement process. However, it is known that the X and Y measurements of the item are related. The measurement of Y may also require total destruction of the item. The extent of correlation (r) between the values of X and Y may be of interest, but because measurements of both X and Y cannot be taken on the same sample, the usual formula for estimating the correlation coefficient cannot be applied.

This type of situation may occur in a number of engineering and scientific applications. For example, in food science, professional "tasters" are often used

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to judge the taste, texture, viscosity, hardness and other qualities of a food product. Instrumental procedures are also available for measuring the same qualities. Often manufacturers would prefer an automated measurement procedure if they could be confident that there is a high correlation between the two types of measurements. In many situations, the food product is destroyed by either testing procedure.

CLA	CLASSIFIED INTO 3 GROUPS (CLARK AND RAO 1977)					
X <sub>i,j</sub> : Har	dness Scores	(i = 1 to 3 a)	nd j = 1 to 8)			
	1 Green	2 Ripe	3 Overripe			
1	7.000	3.000	1.667			
2	7.667	5.000	2.333			
3	8.000	4.500	3.000			
4	8.333	4.000	1.333			
5	8.333	5.500	2.333			
6	8.000	4.500	1.333			
7	8.667	5.500	1.333			
8	8.333	6.000	2.667			
Mean	8.042	4.750	1.999			
Y <sub>i,j</sub> : Bul	k Modulus Val	ues				
	1 Green	2 Ripe	3 Overripe			
1	4.82835	3.88834	2.15463			
2	5.09801	3.75159	4.28753			
3	5.43209	4.43958	3.36548			
4	4.10435	4.17394	2.74741			
5	5.39790	3.71381	2.15199			
Mean	4.97214	3.99345	2.94141			

			T	ABLE 1						
SENSORY	HARDNESS	SCORES	AND	BULK	MODUL	US V	VALUES	FOR	PEACHES	S
	CLASSIFIE	ED INTO	3 GRO	OUPS (	CLARK A	AND	RAO 19	77)		

Correlation Coefficient from data means (3 pairs) = 0.997Average Correlation Coefficient from Simulation = 0.84690% Confidence Interval = (0.715, 0.958)

As an example of this problem Clark and Rao (1977) attempted to determine the relationship between sensory and instrumental measures on peaches. They used 3 groups of peaches classifying them as Green, Ripe and Overripe (Table 1). In their tests they used 8 panelists to score the hardness of the peaches on a hedonic scale of 1 (very soft) to 10 (very hard). Measurements were also made on the bulk modulus (which is an indication of incompressibility) of the peaches. However they could not use the same peach from each group for both the measurements. Another example of this type of problem is shown in a study by Diehl and Hamann (1979). They examined the relationships between sensory profile parameters and fundamental mechanical parameters for raw potatoes, melons and apples. Each of the three foods tested was divided into groups based on variety, ripeness or storage condition. For each food the coefficient of correlation between instrumental and sensory parameters was determined using the group mean values of the instrumental scores and the consensus of the sensory panel scores. In a study performed by Stone and Young (1985) physical and sensory attributes were used to measure the effect of cultivar, blanching techniques and cooking methods on the quality of frozen green beans. The correlations determined in this work were based on mean values, since sensory and instrumental measurements could not be taken from the same item in each group. In general with all such experiments, a rough grouping of the items can sometimes be performed on the basis of an approximation of the values of one of the two measurements of interest, say X, into groups thought to have (for example) large values of X, medium values of X, or small values of X. A straightforward (and overly optimistic) estimate of the correlation coefficient can then be obtained by the following procedure. The items in each group can be divided into two subgroups; measurements of X are taken on one subgroup and measurements of Y are taken on the other subgroup. Thus, for each group, an average value of X and an average value of Y can be estimated. These group points of averages can be plotted, a least squares line can be fitted to these points, and the pairwise correlation coefficient can be estimated from the slope of the line (Clark and Rao 1977). Several problems arise from this method of estimation. A major concern is that fitting lines to points of averages instead of the actual data points leads to overly optimistic estimates of the goodness of fit of the line (the so-called ecological correlation problem, Freedman et al. 1978). The meaningfulness of confidence limits for parameter estimates is also therefore questionable. Another element that should be considered is that if the sample sizes are different from one group to another, the accuracy of the point estimates are not the same for the different groups. Furthermore, the extent of separation of the groups should be important in obtaining useful results. If it is very difficult to assign an item to a group, then there is likely to be very little difference between the outcome measures from the different groups, making even ecological correlation estimates small.

A similar problem that has been investigated in the literature is destructive testing when the first measurement might destroy the item being tested, thus making it impossible to take the final half of the paired measurement for a certain fraction of the items sampled (Evans *et al.*). However, in that situation it is possible to obtain both measurements per item for at least some of the items sampled. For the discussion in this paper, we assume that either measurement will result in the complete destruction of the item.

## ESTIMATION OF THE CORRELATION COEFFICIENT

The method used in this study to estimate the correlation coefficient is based upon generating likely pairs of points for two measurements, X and Y. The items which are to be tested are divided into levels based upon some characteristic (e.g. brand, ripeness, age, quality level). Each of these levels (denoted by the subscript i) is further divided into two subgroups, and the measurements of  $X_{i,j}$  and  $Y_{i,j}$  (with j denoting the particular observation) are made from the two subgroups. The mean and standard deviation of each level of X and Y is determined. A microcomputer is used to generate 24 random numbers between 0 and 1, which are then summed. The sum of these random numbers will always be normally distributed (mean = 12 and variance = 2) as a result of the central limit theorem. The sum of these random numbers, together with the mean and standard deviation of the particular level is used to calculate a pair of simulated observations by the following equations:

 $SX_i = ((RN - 12.0)/1.4142) * SDX_i + MX_i$ 

$$SY_i = ((RN - 12.0)/1.4142) * SDY_i + MY_i$$

where

SXi	is the simulated observation of X for any level i
SYi	is the simulated observation of Y for any level i
RN	is the sum of the random numbers
SDX <sub>i</sub>	is the standard deviation of the measurements of X for any level i
MX	is the mean of the measurements of X for any level of i
SDY	is the standard deviation of the measurements of Y for any level i
MYi	is the mean of the measurements of Y for any level i

The above algorithm will in effect generate a random pair of points for each group with the mean and standard deviation based on observed values for that group. This procedure is repeated for every level, with the random number (RN) being recalculated each time the simulation is performed on a different level. When simulated pairs of observations have been generated for each level a correlation coefficient (r) is calculated using the Pearson product-moment correlation (Bruning and Kintz 1977) given below:

$$\mathbf{r} = \frac{\mathbf{N} \Sigma \mathbf{X} \mathbf{Y} - (\Sigma \mathbf{X}) (\Sigma \mathbf{Y})}{([\mathbf{N} \Sigma \mathbf{X}^2 - (\Sigma \mathbf{X})^2] [\mathbf{N} \Sigma \mathbf{Y}^2 - (\Sigma \mathbf{Y})^2])^{\frac{1}{2}}}$$

where

Ν	= number of pairs of scores
ΣΧΥ	= sum of the products of the paired scores
ΣΧ	= sum of scores on one variable
ΣΥ	= sum of scores on the other variable
$\Sigma X^2$	= sum of the squared scores on the X variable
$\Sigma Y^2$	= sum of the squared scores on the Y variable

The simulation of pairs of points and calculation of a correlation coefficient are repeated 5000 times, and an average correlation coefficient is calculated from the 5000 individual correlation coefficients. Upper and lower confidence levels are determined by taking the 5th and 95th percentile of the 5000 correlation coefficients. The above procedure was written in PASCAL and compiled to machine language code on a personal computer (Fig. 1). The program was used to determine the correlation coefficients for data where simultaneous measurements on both the X and Y variables were not possible and when it was possible.

# **DISCUSSION OF RESULTS**

The data for peaches are shown in Table 1. The only estimate of the correlation between hardness and bulk modulus using the means for each group was 0.997. Using the simulation procedure described above the correlation coefficient was determined to be 0.846 with 90% confidence interval of 0.715 and 0.958. In other words the correlation between hardness and bulk modulus is likely to be 0.846 and we are 90% confident that it will not be lower than 0.715 and higher than 0.958. This value is remarkably different from the estimate obtained using the means for each group and as expected is lower in value. Now the question arises if the simulation method is indeed an accurate procedure. In answer to that question data was used from experiments by Nortje (1986) on texture of extruded corn curls. In his experiments Nortje used a stereological measure viz. Pore wall area per unit volume and a texture measure viz. maximum force. He used the same corn curl for both tests by first cutting the sample into two pieces and measuring each variable (Table 2). Therefore in his experiments each pair of observations was matched. Hence using the direct procedure the correlation coefficient was estimated to be 0.895. Using the mean value for each group (A, B and C) the correlation coefficient was determined to be 0.950. This is obviously an over optimistic estimate of the actual value. Using the simulation procedure the correlation coefficient was estimated to be 0.893. This value is much closer to the actual value compared to the CC determined from the means. It should be



FIG. 1. FLOWCHART FOR COMPUTER PROGRAM TO ESTIMATE CORRELATION COEFFICIENT BETWEEN UNMATCHED VARIABLES

Maximum	Pore wall	
Force	area/ volume	
9.04	4.10	
8.89	4.70	
7.90	4.00	
12.9	5.89	
13.4	5.37	
11.3	6.04	
19.8	6.98	
22.8	6.89	
24.3	6.87	
and the second		

TABLE 2.
ESTIMATION OF CORRELATION BETWEEN MAXIMUM FORCE
AND PORE WALL AREA/VOLUME FOR CORN CURLS

Correlation Coefficient using data points = 0.895Correlation Coefficient using data means = 0.950

Correlation Coefficient using simulation = 0.893

90% Confidence Interval = (0.857, 0.941)

noted that the groups in this example are 3 and should result in a higher percent error. If the number of groups were increased to over 7 or 8, the estimate for correlation coefficient should be more appropriate using the data means.

In order to illustrate the procedure for larger number of groups, data from tests on rye bread was used. The relationship of interest in this case was between lactic acid and titratable acid. The tests were conducted on 4 different brands of bread and each brand was tested 4 times. Once again the 2 variables, viz. lactic acid and titratable acid, were measured on the same sample and therefore yielded matched pairs of data. The correlation coefficient determined using individual paired observations was 0.980, while the correlation coefficient calculated using the means of the four brands was found to be 0.990. The simulation procedure was tested four times on the rye bread data and yielded correlation coefficients of 0.979, 0.981, 0.983 and 0.981. It is fairly obvious that the simulation procedure proposed in this work is a very good estimate of the correlation coefficient. The repeatability of the procedure would appear to be sound as demonstrated by the four simulations. The percent error between the different trials is less than 4/10 of 1%.

In summary, an algorithm is proposed in this work that can be used to determine the correlation coefficient (and confidence interval) between variables when simultaneous measurements on both are not possible. As an improvement to this procedure, the simulation of pairs of points can be done using a bivariate normal after getting an initial estimate of the correlation coefficient. The computer program proposed in this work should suffice for most applications and can be used effectively on a personal computer. The complete algorithm with the original source code in Pascal is freely available from the authors.

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