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MASS TRANSFER IN PLANT MATERIALS IN CONTACT WITH AQUEOUS SOLUTIONS OF ETHANOL AND SODIUM CHLORIDE: EQUILIBRIUM DATA

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ABSTRACT

A process for osmotic dehydration of plant materials using aqueous solutions of ethanol and sodium chloride is described. Equilibrium data for blanched and unblanched carrots in contact with binary aqueous solutions of ethanol and sodium chloride are presented. Based on the equality of activities of the solutes in carrots and the external solution a successful representation of equilibrium has been obtained in terms of the measurable practical variables such as the total solids in the material at full turgor, solute concentration in the bath and the temperature of contacting.

INTRODUCTION

Freezing of plant materials such as fruits and vegetables is an energy intensive process. The large amount of energy spent in this process includes the energy spent in freezing, packaging, transportation and storage of the large amount of water present in the fresh materials (Rao 1977). One way to deal with this problem is to partially concentrate or dehydrate the product before freezing so that the overall energy requirement of the process is reduced (Huxsol 1982).

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The concept of partial dehydration before freezing of plant materials was originally suggested by Howard and Campbell (1946), and subsequently studied by Talburt and Legault (1950), Talburt *et al.* (1950), Lazar *et al.* (1961), and Kitson (1970). These studies, however, considered air drying as the means of partial dehydration. Air drying is also an energy intensive process and when conducted at elevated temperatures, it may impair the quality of the product by introducing unfavorable changes in color, texture and flavor. Part of the nutrients may also be lost. For a dehydrofrozen product, therefore, to be quality competitive with the conventionally frozen product, it is necessary that the prefreezing, partial dehydration be conducted at a low temperature. Low temperature air drying using vacuum techniques, though a possibility, is also energy intensive, and cost prohibitive. The technique which we propose is a low temperature osmotic dehydration using aqueous solution of sodium chloride and ethanol. Named as aqueous freezant, AF (15–15) i.e., 15% NaCl and 15% ethanol in water, this solution was originally used by Robertson *et al.* (1976) and Cipolletti *et al.* (1977) for direct contact freezing of vegetables.

Osmotic dehydration of fruits and vegetables using aqueous solutions of sugar, salt or combinations thereof has been described by Ponting *et al.* (1966), Hartal (1967), Farkas and Lazar (1969), Ponting (1973), Hawkes and Flink (1978), Flink (1980), Bolin *et al.* (1983), Conway *et al.* (1983), Lenart and Flink (1984a,b), Lerici *et al.* (1985), Kim and Toledo (1987), Gianogiacomo *et al.* (1987), Torreggiani *et al.* (1987) and LeMaguer and Biswal (1988). In short, osmotic drying consists of placing the materials to be dried in a solution of high osmotic pressure, such as sugar or salts, and allowing water to transfer from the product to the solution via osmosis. From an organoleptic point of view sugar solutions have been used for the dehydration of fruits and salts for vegetables. A comprehensive review of the process has been given by Le Maguer (1988).

A proposed scheme for the dehydrofreezing of fruits and vegetables using low temperature osmotic dehydration as a prefreezing operation (“dehydrocooling”) is shown in Fig. 1. As can be seen in the process diagram, plant materials (vegetables in the piece form) are partially dehydrated in a dehydrocooling contactor and then frozen by immersion freezing. The osmotic solution diluted during the process is reconstituted before it is recirculated. It is worth mentioning that the method employed in reconstituting the osmotic solution should be less energy intensive than the freezing process.

The design and development of a contactor for low temperature osmotic dehydration of plant materials using AF(15–15) requires data on the mass and heat transfer behavior of the system. A contacting of this nature, being a novel concept, mass transfer information meeting the operating conditions is not available. A study on the mass transfer kinetics of the system using diced carrots as the working material was conducted in our laboratory. The results of this study have been planned as a series of three papers in this Journal. This paper is the

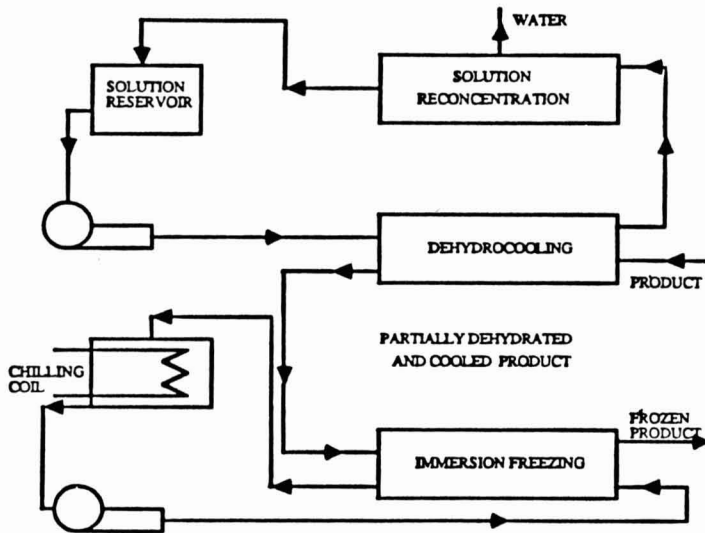


FIG. 1 DEHYDROCOOLING AS AN INTERMEDIATE STEP IN FREEZING

first one in the series, where the mass transfer equilibrium data for blanched and unblanched carrots in contact with aqueous solutions of ethanol and sodium chloride are presented. The second paper will describe the model for mass transfer kinetics of the system, and the third one will report the mass transfer rate parameters obtained through the study.

At equilibrium with the aqueous solution of ethanol and sodium chloride the plant material can be considered made of four components: salt, ethanol, water and total solids non-salt. The total solids non-salt includes both soluble and insoluble solids of the material. As a first approximation this quaternary system has been treated separately as two ternary systems i.e., salt-water-total solids non-salt and ethanol-water-total solids non-salt. These two ternary systems are obtainable by soaking plant materials separately in binary aqueous solutions of ethanol and sodium chloride. Equilibrium data on these two ternary systems for blanched and unblanched plant materials are reported here. These results will be combined on a weighted average basis to describe the quaternary system of interest.

EXPERIMENTAL AND ANALYTICAL METHODS

Experimental Methods

Carrots, obtained from a local produce market were hand peeled, trimmed at both ends and cut into 1 cm dices using a Hobart cutting machine. Carrot cubes

were graded, washed and standardized by soaking them in tap water for one hour. For the experiments using blanched products, carrot cubes were blanched in live steam for three minutes, cooled by ice water, and blotted to remove the excess water adhering to the surface.

Aqueous solutions of NaCl and ethanol (binary mixtures only) with concentrations of 0%, 5%, 10% and 15% (wt basis) were prepared. Two parallel sets of experiments were planned: one with unblanched and the other with blanched carrots. Each set was conducted with NaCl water and ethanol water solutions at two different temperatures: Blanched carrots in NaCl-water and ethanol-water solutions at 9 and 31°C, and unblanched carrots in NaCl-water solution at 8 and 29°C and in ethanol-water solution at 10 and 30°C.

The contacting of carrot cubes with the solutions was conducted in beakers with magnetic stirrers, maintaining a solids to liquid ration of 1:10. To obtain the equilibrium values within the desired range of concentration of the exterior solution, four beakers with solute concentrations of 0%, 5%, 10% and 15% were used for each equilibrium run. Carrots were made to equilibrate with the exterior solution by allowing contacting for 16 h in case of NaCl water runs and 36 h in case of ethanol water runs. At the completion of the soaking period the solution was drained out, and the carrots were blotted and separated for the measurement of density, total solids, solute component, and the weight fraction of water. Weight fraction of the solute in the solution and the density of the solution were also measured.

Analytical Methods

The following is a summary of the methods employed in the analysis of samples.

Total Solids. Total solids of carrots was determined by vacuum drying the samples at 70°C overnight (AOAC 1980).

Density of Carrots and Solution. The density of carrots was measured using wide mouth pycnometers. Three replicates of approximately 5 g carrots were taken from each beaker. Hexane was the reagent used in measuring the density. The density of the solutions was measured using 25 mL pycnometers.

Ethanol Determination. Measurement of ethanol in carrots and the soaking solution was done by gas chromatography. (Varian Model 3700 gas chromatograph, Cole Palmer chart recorder, column: 30% carbowax 400 on chromosorb G 60/80, 1/4 in. by 6 ft stainless steel).

Four replicates of 12 g of carrots were taken from each beaker. The cubes were blended dry, then about 30 mL of distilled water was added and blending continued until a slurry with fine particles was obtained. The solution was filtered in a vacuum filtration system. An aliquot of the solution was weighed and a known amount of acetone was added.

Each solution was injected into the column 3 times alternately with the ethanol-acetone-water standard. A digitizer was used to measure peaks.

A similar procedure was used for the soaking solution. Weighed aliquots were taken from the filtered solution and a known weight of acetone was added. The solutions were injected alternately with the standards.

Determination of NaCl. The sodium chloride content of the carrots were measured using a chloride electrode (Anon. 1977). Fisher Accumet Model 230 pH/ion meter, Accumet Selective Ion Analyser Model 750, Mettler PC 400 balance, and Orion chloride electrode (94-17) were used.

Four replicates of 12 g of carrots were taken from each beaker. The cubes were heated in distilled water until they were soft. They were then mashed with a pestle. $\text{Ca}(\text{OH})_2$ was added until pH 11. The solution was then filtered, and acidified to pH 5. A weighed aliquot of the acidified solution was taken and 2 mL/100 g solution of ionic strength adjuster was added. The solution was then reweighed. Each solution was measured for mV response against the standard.

For solutions, four weighed aliquots were taken from each beaker. A similar procedure as above was followed to obtain the mV response against the standards.

ANALYSIS OF EQUILIBRIUM DATA

For the purpose of estimating the mass transfer driving forces between surface and inside of the material, one would require data on the surface properties. Assuming that the process is not externally mass transfer limited the surface concentrations are in equilibrium with the bath concentrations. By performing equilibrium experiments where the whole tissue is in equilibrium with the solution one would therefore be in a position to predict the surface concentration. As discussed below, the knowledge of the weight fraction of the total solids (non-salt) at equilibrium is sufficient for calculating the density of the material and obtaining concentration of any species at equilibrium.

Since both ethanol and salt are penetrating solutes i.e., they can pass the semipermeable membranes of the live cells, at equilibrium with binary solutions on the outside the material can be considered made of three components: salt or ethanol, component 1, water, component 2, total solids (not including component 1), component 3. Assuming that at equilibrium the activity of the salt or ethanol in the bath and the aqueous phase of the carrots are identical, the internal composition on a binary basis is given by (Del Valle and Nickerson 1967; Zugarramurdi and Lupin 1980):

$$w_i^* = w_{iB} (1 - w_3^*) \quad (1)$$

where w_3^* is the weight fraction of total solids (not including component 1) at equilibrium, w_{iB} is the weight fraction i in the bath, and w_i^* is the weight fraction of i in the material at equilibrium ($i = 1$, salt or ethanol and $i = 2$, water).

It should be mentioned that Eq. (1) has been derived based on the assumption that at equilibrium the activity of salt or ethanol in both the phases are equal. A limitation of this assumption is the effect of soluble solids present in the original material on the activity of salt or ethanol in the liquid phase of carrots. The total solids non-salt (component 3) described earlier includes both insoluble and soluble solids. Ideally, the soluble solids portion is the one which would go into solution in the liquid phase of the material and affect the activity of salt or ethanol present in that phase. When leached out the soluble solids could also affect the activity of salt or ethanol in the external solution. For the purpose of present work, it has been assumed that the effect of leaching on the activity of solutes in either of the phases is negligible.

A volume additive consideration given by Eq. (2) below establishes the fact that density of the material at equilibrium, can be estimated from the knowledge of w_3^* and the partial specific volumes \bar{v}_1 , \bar{v}_2 and \bar{v}_3 .

$$\rho_1^* \bar{v}_1 + \rho_2^* \bar{v}_2 + \rho_3^* \bar{v}_3 = 1 \quad (2a)$$

or,
$$w_1^* \bar{v}_1 + w_2^* \bar{v}_2 + w_3^* \bar{v}_3 = 1/\rho^* \quad (2b)$$

where ρ_i^* is the concentration of component i in the material ($i = 1, 2, \text{ or } 3$) at equilibrium, and w_i^* as explained in Eq. (1). It should be mentioned that for the solid (ternary system) Eq. (3a) holds good and

$$\sum_{i=1}^3 w_i^* = 1 \quad (3a)$$

for the external solution (binary) Eq. (3b) holds good.

$$\sum_{i=1}^2 w_{iB} = 1 \quad (3b)$$

As mentioned in the previous section, our experimental w_3^* is the w_3 measured after 36 h of contacting as in the case of ethanol-water solution and 16 h as in the case of salt-water solution. The experimental values of w_3^* as obtained for blanched and unblanched carrots in equilibrium with four different concentrations (w_{1B}) and two different temperatures ($t^\circ\text{C}$) are given in Table 1 and 2. Also given in Table 1 are w_3^F the weight fraction of total solids in the original material at full turgor and w_3^0 the weight fractions of total solids in the material at equilibrium with pure water. The attempt to measure the density of carrots in equilibrium with the aqueous solutions (ρ^*) resulted in inconsistent and nonreproducible data for those carrots which were soaked in the ethanol-water solution. The ρ^* of carrots soaked in pure water and in NaCl-water were consistent and reproducible; these data are given in Table 1 and 2.

TABLE I.
EQUILIBRIUM DATA FOR UNBLANCHED CARROTS

System	Temperature °C	w _{1B}	w ₃ [*]	ρ ₃ [*] kg/m ³	(w ₃ [*] /w ₃ ⁰)
NaCl-water	8	Blank	0.1002 (w ₃ ^F)	1030.4	--
		0.00	0.0867 (w ₃ ⁰)	1056.9	1.0
		0.0441	0.1090	1075.1	1.26
		0.0882	0.1081	1109.2	1.25
		0.1296	0.1051	1136.0	1.21
	29	Blank	0.1010 (w ₃ ^F)	1047.6	--
		0.0	0.0766 (w ₃ ⁰)	1033.9	1.0
		0.0517	0.1048	1067.7	1.37
		0.0957	0.1111	1094.6	1.45
		0.1360	0.1067	1123.7	1.39
Ethanol-water	10	Blank	0.0963 (w ₃ ^F)	1031.1	--
		0.00	0.0834 (w ₃ ⁰)	1049.1	1.00
		0.0426	0.0852	N/A	1.02
		0.0871	0.0811	N/A	0.97
		0.1261	0.0522	N/A	0.63
	30	Blank	0.1136 (w ₃ ^F)	1029.9	--
		0.0	0.0621 (w ₃ ⁰)	1048.7	1.00
		0.0455	0.0674	N/A	1.09
		0.0970	0.0568	N/A	0.91
		0.1275	0.0460	N/A	0.74

RESULTS

The description of w₃^{*} has been proposed through a polynomial relation in w_{1B}, with the coefficients being functions of temperature as given in Eq. (4).

$$\frac{w_3^*}{w_3^F} = a_0 + a_1 w_{1B} + a_2 w_{1B}^2 + \dots \quad (4)$$

TABLE 2.
EQUILIBRIUM DATA FOR BLANCHED CARROTS

System	Temperature °C	w_{1B}	w_3^*	ρ_3^* kg/m ³	(w_3^*/w_3^0)	
NaCl-water	9	Blank (UB)	0.0970 (w_3^F)	1032.8	--	
		Blank (B)	0.0933	1046.1	--	
		0.0	0.0403 (w_3^0)	1022.5	1.0	
		0.0443	0.0392	N/A	0.97	
		0.0912	0.0337	N/A	0.84	
		0.1368	0.0302	N/A	0.75	
	31	Blank (UB)	0.1037 (w_3^F)	1030.5	--	
		Blank (B)	0.0957	1036.3	--	
		0.0	0.0410 (w_3^0)	1019.8	1.0	
		0.0458	0.0383	N/A	0.93	
		0.0918	0.0336	N/A	0.82	
		0.1376	0.0304	N/A	0.74	
	Ethanol-water	9	Blank (UB)	0.0999 (w_3^F)	N/A	--
			Blank (B)	0.0941	N/A	--
0.0			0.0422 (w_3^0)	N/A	1.0	
0.0429			0.0460	N/A	1.09	
0.0760			0.0452	N/A	1.07	
0.1437			0.0470	N/A	1.11	
31		Blank (UB)	0.1049 (w_3^F)	1030.1	--	
		Blank (B)	0.0836	1060.1	--	
		0.0	0.0454 (w_3^0)	1026.7	1.0	
		0.0483	0.0446	N/A	0.98	
		0.0964	0.0458	N/A	1.01	
		0.1116	0.0466	N/A	1.03	

UB: Unblanched, B: Blanched

where w_3^F is the weight fraction of total solids of the original material at full turgor, and a_0 , a_1 and a_2 are functions of temperature. Since our experimental measurements were limited to only two temperatures, a linear temperature effect as given in Eq. (5) is assumed.

$$a_0 = b_0 + b_1 t \tag{5a}$$

$$a_1 = c_0 + c_1 t \tag{5b}$$

and so on for a_2 and other higher order terms in Eq. (4).

Neglecting the terms beyond quadratic and limiting the temperature effect only to a_0 and a_1 , Eq. (4) can be rewritten as given below:

$$\frac{w_3^*}{w_3^F} = b_0 + b_1 t + (c_0 + c_1 t) w_{1B} + a_2 w_{1B}^2 \tag{6}$$

As a special case when $w_{1B} = 0$ (pure water):

$$w_3^* = a_0 w_3^F = w_3^0 \tag{7}$$

or,
$$w_3^0 = (b_0 + b_1 t) w_3^F \tag{8}$$

Equation (8) enabled us to obtain b_0 and b_1 from the data on w_3 of the original material at full turgor (w_3^F) and w_3 at equilibrium with pure water (w_3^0). The coefficients b_0 and b_1 were estimated through a linear regression in SAS (1985).

Analysis of experimental data indicated that the effect of temperature as given by Eq. (8) exists only in case of unblanched materials. Therefore, for blanched materials w_3^0 is independent of temperature and is given by:

$$w_3^0 = b_0 w_3^F \tag{9}$$

The behavior explained by Eq. (9) is attributed to the effect of blanching on cell membrane of the plant tissue. The effect of blanching is to destroy the cell membranes and allow soluble solids to leach out. When followed by soaking, given sufficient time, this will lead to total loss of soluble solids. It was found that for blanched carrots $b_0 = 0.42$; in other words w_3^0 is 42% of w_3^F . For unblanched materials partial denaturation of the membrane may still take place due to the effect of temperature. This has been represented in Eq. (8).

After obtaining b_0 and b_1 from the experimental data on pure water contacting as explained before, Eq. (6) was rearranged to a multilinear form in t and w_{1B} ,

and the parameters c_0 , c_1 and a_2 were estimated for all cases (blanched or unblanched materials in salt-water or ethanol-water solution) through a multi-linear regression in SAS (1985). The parameters are listed in Table 3.

In summary, if we need to estimate the surface properties of carrots in contact with either a salt-water or ethanol-water solution we must know the weight fraction of total solids of the original material at full turgor, w_3^F , through experimental measurements (vacuum oven technique). For the particular situation at hand, we then obtain appropriate coefficients from Table 3 and calculate w_3^* using Eq. (6). Using the calculated value of w_3^* we then calculate w_1^* and w_2^* through Eq. (1) which are used in Eq. (2b) to obtain ρ^* , the density of the material at the surface. The ρ^* is used along with w_1^* and w_2^* to give the mass concentration of any component i as given by Eq. (10).

$$\rho_i^* = \rho^* w_i^* \quad (10)$$

DISCUSSION

A graphical description of w_3^* as a function of w_{1B} at different temperatures is shown in Fig. 2. In this figure, the ratio w_3^*/w_3^0 (Eq. (11), as obtained by

TABLE 3.
COEFFICIENTS FOR w_3^* AS A FUNCTION OF w_{1B} , w_3^F AND t AS GIVEN BY EQ. 6

System	Coefficient	Unblanched carrots	Blanched carrots
Ethanol-water	b_0	0.944 ($r^2=0.80$)	0.42
	b_1	-0.009	0.0
	c_0	2.20 ($r^2=0.92$)	0.0
	c_1	0.03	0.0
	a_2	-35.15	0.0
Salt-water	b_0	0.944 ($r^2=0.80$)	0.42
	b_1	-0.009	0.0
	c_0	5.89 ($r^2=0.97$)	0.0
	c_1	0.095	0.0
	a_2	-44.09	-4.48 ($r^2=0.80$)

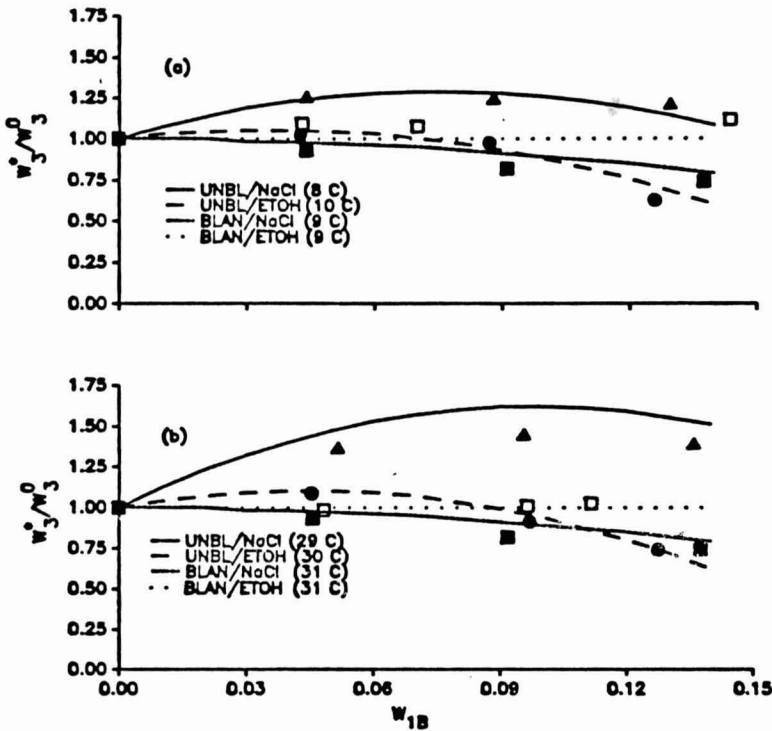


FIG. 2 CONCENTRATION DEPENDENCE OF (w_3^*/w_3^0) (a) Low temperature (b) High temperature. Symbols used for experimental points: \blacktriangle Unblanched/NaCl-water, \bullet Unblanched/ethanol-water, \blacksquare Blanched/NaCl-water and \square Blanched/ethanol-water.

substituting Eq. (8) in Eq. (4)) rather than w_3^* alone or the ratio w_3^*/w_3^F has been plotted for all the four cases at hand (unblanched or blanched carrots in salt-water or ethanol-water solution).

$$\frac{w_3^*}{w_3^0} = 1 + \frac{a_1}{a_0} w_{1B} + \frac{a_2}{a_0} w_{1B}^2 \tag{11}$$

As it can be seen in Eq. (11), w_3^*/w_3^0 is equal to 1 when $w_{1B} = 0$ this gives a common starting point for all the curves in Fig. 2. Also, at any given temperature, the ratio w_3^*/w_3^0 is a function of w_{1B} only. Therefore, plotting w_3^*/w_3^0 against w_{1B} is a sound way of comparing the equilibrium behavior of the four different cases at hand.

Figure 2a shows the w_3^*/w_3^0 curves for the lower temperature group of runs (8 to 10°C) and Fig. 2b for the higher temperature (29 to 31°C). It is seen that the

experimental and fitted w_3^*/w_3^0 for all four cases at different temperatures match very well with the exception that w_3^*/w_3^0 for unblanched carrots in NaCl-water solution at 29°C is slightly overpredicted. This is consistent with the reported r^2 value in Table 3 i.e., $r^2 = 0.80$ toward the temperature effect. The nature of the curves as a function of w_{1B} , however, needs to be discussed.

From the definition of w_3^* and w_3^0 the ration w_3^*/w_3^0 can also be written as:

$$\frac{w_3^*}{w_3^0} = \frac{m_3^*}{m_3^0} \times \frac{m_T^0}{m_T^*} \quad (12a)$$

and if there is no leaching of soluble solids:

$$m_3^* = m_3^0 = m_3^F \quad (12b)$$

which leads to

$$\frac{w_3^*}{w_3^0} = \frac{m_T^0}{m_T^*} \quad (12c)$$

In case of unblanched carrots in salt-water solution, the ratio w_3^*/w_3^0 as shown in Fig. 2, is always greater than 1. But we know that if there is no leaching

$$\left. \frac{m_3^0}{m_3^*} \right|_{\max} = 1 \quad (13)$$

Therefore, the only way w_3^*/w_3^0 can attain a value greater than 1 is if m_T^* is less than m_T^0 , i.e., if there is a net loss in the total mass of the material. This net change in the total mass can occur only if the flux of water is greater than that of the salt. In this contacting system it is also seen in Fig. 2 that at about $w_{1B} = 0.09$, the ratio w_3^*/w_3^0 starts to decrease with the increase in bath concentration. This behavior might be due to the combined effect of leaching, if any, and the relative rates of water and salt exchange.

As shown in Fig. 2 for unblanched carrots in ethanol-water solution, the nature of the plot of w_3^0/w_3^* vs w_{1B} is exactly the same as before at low concentrations. At higher concentrations, the ratio w_3^*/w_3^0 decreases progressively with the increase in w_{1B} and goes below 1.0. The rate of penetration of ethanol is larger than for salt and close to water rate. Therefore, the ratio m_T^0/m_T^* tends to stay close to 1.0. However, as reported by Glinka and Reinhold (1972), ethanol, at higher concentration for a longer period of contacting, irreversibly destroys the

carrot cell membrane (mostly by dissolving the lipids), and consequently leaching of soluble solids will occur leading to the observed w_3^*/w_3^0 less than 1.0.

As shown in Fig. 2, the ratio w_3^*/w_3^0 for blanched carrots is much less affected by the solute concentration in the bath. For blanched carrots soaked in ethanol-water solutions the ratio is observed to be constant at 1.0. For salt-water solutions, however, this ratio is seen to be less than 1.0 for the entire range of concentrations, and to be decreasing progressively with the increase in bath concentration. Since blanching destroy most of the cell structure it is expected that, given sufficient time for contacting the extent of leaching will be the same whether the material is soaked in pure water or in an aqueous solution i.e., $m_3^* = m_3^0$ in Eq. (12). Therefore, the ratio w_3^*/w_3^0 would be affected only by the relative exchange of water and the solute (ethanol or salt) giving rise to a change in the ratio of m_T^0/m_T^* as shown in Eq. (12). For ethanol water system the fluxes of water and ethanol, to a great extent, compensate each other on a mass basis, leading to m_T^0/m_T^* to be practically constant at 1.0. In case of salt-water system, however, as observed by Le Maguer and Biswal (1983), the flux of salt is bigger than that of water giving rise to m_T^* to be greater than m_T^0 and consequently leading to w_3^*/w_3^0 to be less than 1.

As a verification of the proposed correlation a comparison of the predicted and experimental densities of carrots in equilibrium with the aqueous solutions has been made. Since the experimental equilibrium density data on ethanol-water runs are incomplete (see Table 1 and 2) data of only the NaCl-water runs have been used in the comparison. Assuming that the total solids non-salt component does not affect the partial specific volumes of other components present in carrots, the partial specific volumes of NaCl and water, \bar{v}_1 and \bar{v}_2 respectively, have been estimated from the density data of NaCl-water solutions within the concentration and temperature ranges of interest (Anon. 1929). Using the procedure of Smith and Van Ness (1975) the correlations for \bar{v}_1 and \bar{v}_2 as obtained through BMDP Statistical Software (1982) are the following:

$$\bar{v}_1 = 0.283 \times 10^{-3} + 8.0 \times 10^{-7} t + 0.4794 \times 10^{-3} w_{1E}^* - 0.2397 \times 10^{-3} w_{1E}^{*2} \quad (14a)$$

$$\bar{v}_2 = 0.997 \times 10^{-3} + 2.0 \times 10^{-7} t - 0.2397 \times 10^{-3} w_{1E}^{*2} \quad (14b)$$

where \bar{v}_1 and \bar{v}_2 are partial specific volumes of NaCl and water respectively, m^3/kg , w_{1E}^* is the binary equivalent of ternary NaCl weight fraction in carrots, and t is temperature in °C. With reference to Eq. (1) one can easily visualize that the numerical value of w_{1E}^* is the same as w_{1B} . The applicability of Eq. (14) is for t in the range of 0 to 30°C and w_{1E}^* in the range of 0.05 to 0.25. A constant value for \bar{v}_3 the partial specific volume of the total solids non-salt, has been assumed. Assuming that the density of total solids non-salt is 1588 kg/m³, same

as the density of sucrose (Perry and Chilton 1973), \bar{v}_3 the inverse of the density is equal to $0.6297 \times 10^{-3} \text{ m}^3/\text{kg}$. Following the outline given at the end of the preceding section i.e., obtaining w_3^* from Eq. (6), w_1^* from Eq. (1), w_2^* by difference, \bar{v}_1 and \bar{v}_2 from Eq. (14) and using $\bar{v}_3 = 0.6297 \times 10^{-3} \text{ m}^3/\text{kg}$, the density of carrots as calculated for known experimental conditions are given in Table 4. As can be seen in Table 4, the predicted values match with the experimental values very well.

CONCLUSION

Based on the equality of activities of the solutes in bath and the carrots a successful representation of equilibrium has been obtained in terms of measurable practical variables, w_{1B} the solute concentration in the bath, w_3^F the total solids in the material at full turgor and t °C the temperature of contacting. Results of this study will be used in modeling the mass transfer kinetics of the "dehydro-cooling" process. The description of mathematical model and estimation of mass transfer rate parameters are the subjects of two forthcoming papers.

NOTATION

a_1, a_2	Coefficients in Eq. 4
b_0, b_1, c_0, c_1	Coefficients in Eq. 5
m	Mass of the material (or of any component)
t	Temperature, °C
\bar{v}	Partial specific volume of component i (m^3/kg)
w_i	Weight fraction of component i
ρ	Density of carrots (kg/m^3)
ρ_i	Concentration of component i (kg/m^3)
Subscripts:	
1,2,3	Components 1, 2 and 3
B	Bath
E	Binary equivalent of the ternary composition
EXP	Experimental
i	Component 1, 2 and 3
PRED	Predicted
T	Total
Superscripts:	
F	Material at full turgor
o	Soaked in pure water for infinite length of time
*	Soaked in the solution for infinite length of time

TABLE 4.
COMPARISON OF PREDICTED AND EXPERIMENTAL DENSITIES OF UNBLANCHED
CARROTS IN EQUILIBRIUM WITH NaCl-WATER SOLUTIONS

	Case 1		Case 2					
	t = 8°C and $w_3^F = 0.1002$		t = 29°C and $w_3^F = 0.1010$					
w_{1B} (Experimental Condition)	0.0	0.0441	0.0882	0.1296	0.0	0.0517	0.0957	0.1360
w_3^* (Eq. 6)	0.0874	0.1082	0.1118	0.0995	0.0690	0.1022	0.1118	0.1054
ρ_{PRED}^* (kg/m ³ , Eq. 2b)	1034.0	1073.2	1108.7	1131.8	1022.6	1071.0	1105.5	1131.9
ρ_{EXP}^* (kg/m ³)	1056.9	1075.1	1109.2	1136.0	1033.9	1067.7	1094.6	1123.7

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A SIMPLIFIED ANALYTICAL MODEL FOR FREEZING TIME CALCULATION IN BRICK-SHAPED FOODS

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ABSTRACT

A simplified analytical model for the freezing time prediction of brick-shaped foodstuffs was developed. It was assumed in the model that the solution to the unsteady, one-dimensional heat conduction equation with constant thermophysical properties was valid during cooling and freezing for each of the three directions of the brick-shaped food. Cooling and freezing times were calculated by superposition of the solutions of the unsteady, one-dimensional heat conduction equation with constant thermophysical properties for each direction. The latent heat effects were incorporated into an effective thermal diffusivity term. The predictions of the model were compared to the available experimental data on freezing of two- and three-dimensional bricks and to the experimental data obtained in this research for the freezing of ground beef and mashed potato bricks. Mean errors varying between -6.3% and 2.3%, and standard deviations from the mean being between 6.6% and 14.0% were obtained for the data sets considered.

INTRODUCTION

The prediction of freezing times of brick-shaped foodstuffs is of great importance in the food industry since most packages are of that shape. The numerical models used in the prediction of freezing times have a sound theoretical basis but require excessive computer time (Cleland and Earle 1979; Hayakawa *et al.* 1983b).

The simplest analytical model for the prediction of freezing times of brick-shaped foodstuffs is the Plank (1941) equation. However, the Plank equation

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does not take into account the initial superheat of the foodstuff and the final temperature at the end of the freezing operation. Cleland and Earle (1979) fitted empirical equations to the shape factors in the Plank (1941) equation to increase the level of accuracy of the predictions. de Michelis and Calvelo (1983) calculated the freezing time from a modified form of the Plank (1941) equation and corrected this freezing time by adding the cooling and tempering times which were calculated from the solution of the unsteady state heat conduction equation. Cleland and Earle (1984a) have proposed a parameter which they called the equivalent heat transfer dimension, EHTD, to be used in the calculation of the freezing times of brick-shaped foodstuffs. EHTD is considered to be the effective dimension of the brick-shaped food. EHTD approach compares the freezing time of a brick with that of a slab whose thickness is the smallest dimension of the brick. The freezing times were calculated from a modified form of the Plank (1941) equation. According to this approach, the freezing times of a cube and a sphere having the same characteristic dimension are identical. However, the cube takes longer to freeze than the sphere. Pham (1985, 1986), defined a mean heat conducting path which was the mean of the heat conducting paths from the surface of the body to the thermal center. Freezing times were calculated from a modified form of the Plank (1941) equation.

Hayakawa *et al.* (1983a) have developed an equation for calculating the freezing time of two-dimensional rectangular blocks. The equation contained eight dimensionless groups. The dimensionless groups were obtained by dimensional analysis. De Reinick and Schwartzberg (1986) have derived a short-cut equation for predicting the temperature versus time behavior of rectangular foods at low Biot number range based on the effective heat capacity characteristics of biological materials.

Ilıcalı and Sağlam (1987) have developed an analytical model for the prediction of freezing times of foods undergoing unidirectional heat conduction. The model assumed the solution to the unsteady, one-dimensional heat conduction equation was valid during the cooling and the freezing periods. Latent heat effects were taken into account by defining an effective thermal diffusivity for the freezing period. In this work this model was extended to cover the prediction of freezing times of two or three dimensional brick-shaped foodstuffs. The predictions of the present model were compared with the experimental data obtained from Cleland and Earle (1979), de Michelis and Calvelo (1983), Hayakawa *et al.* (1983a), and with experimental data obtained in this research.

MATERIALS AND METHODS

For an infinite slab with constant physical properties of thickness $2\Delta x$, with a uniform initial temperature T_i , in an ambient temperature of T_a as shown in

Description of the Model

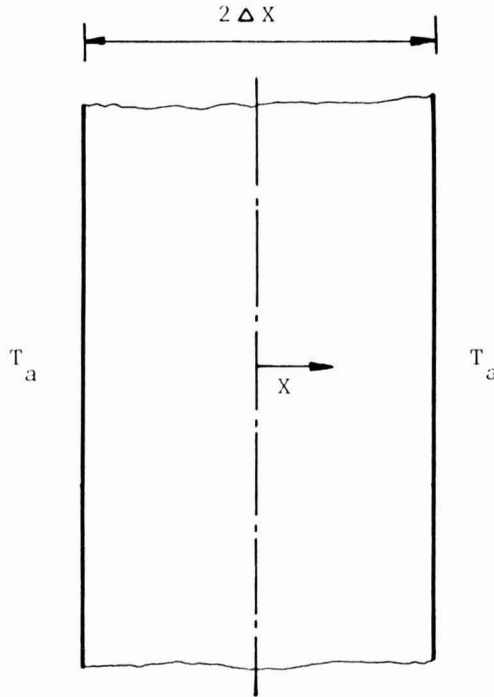


FIG. 1. ONE-DIMENSIONAL HEAT CONDUCTION PROBLEM

Fig.(1), the solution to the unsteady, one-dimensional heat conduction equation is

$$\frac{T - T_a}{T_i - T_a} = \sum_{n=1}^{n=\infty} A_n \cos \left(\lambda_n \frac{x}{\Delta x} \right) e^{-\lambda_n^2 Fo} \quad (1)$$

where λ_n (in radians) is the n th root of

$$\cot \lambda_n = \lambda_n / Bi \quad (2)$$

A_n is a constant which is a function of λ_n , Fo is the Fourier number, x is the distance from the centerline of the slab where temperature T is measured (Kutateladze and Borishanskii 1966). The first three values of A_n and λ_n for the Biot number range from 0.2 to ∞ are shown in Table 1.

As the Fourier number increases the first term of the series solution given in Eq.(1) becomes the dominating term. Table 2 shows the percent error in the dimensionless temperature in Eq.(1) resulting from the utilization of the first

TABLE 1.
THE FIRST THREE VALUES OF A_n AND λ_n IN EQ. (1) AS A FUNCTION OF BIOT
NUMBER (KUTATELADZE AND BORISHANSKII 1966)

Bi	A_1	A_2	A_3	λ_1	λ_2	λ_3
0.2	1.0312	-0.0381	0.0100	0.4328	3.2039	6.3148
3.0	1.2102	-0.2881	0.1154	1.1925	3.8088	6.7040
10.0	1.2612	-0.3934	0.2104	1.4289	4.3058	7.2281
∞	1.2732	-0.4244	0.2546	1.5708	4.7124	7.8540

term of the series solution. As can be observed from Table 2, for Fourier numbers approximately greater than 0.1, the first term of the infinite series given in Eq.(1) is the dominating term at any Biot number.

For Fourier numbers greater than 0.1 Eq.(1) can be written as

$$\frac{T - T_a}{T_i - T_a} = A_1 \cos\left(\lambda_1 \frac{x}{\Delta x}\right) e^{-\lambda_1^2 Fo} \quad (3)$$

The average temperature for an infinite slab at a given Fourier number is

$$T = \int_0^{\Delta x} T dx / \Delta x \quad (4)$$

The average temperature \bar{T} in the slab for Fourier numbers greater than 0.1 can be obtained by combining Eq. (3) and (4).

TABLE 2.
PERCENT ERROR IN THE DIMENSIONLESS CENTER TEMPERATURES RESULTING
FROM THE FIRST TERM APPROXIMATION FOR A SLAB. ALL DATA ARE EXPRESSED
AS PERCENT DIFFERENCES FROM THE SERIES SOLUTION

Fo	Bi = 0.2	Bi = 3	Bi = 10	Bi = ∞
	%E	%E	%E	%E
0	3.1	21.0	26.1	27.3
0.025	2.5	16.2	18.9	19.0
0.050	2.1	14.1	14.2	12.9
0.100	1.3	6.7	6.3	4.8
0.150	0.8	3.5	2.7	1.7

$$\frac{T - T_a}{T_i - T_a} = A_i \frac{\sin \lambda_i}{\lambda_i} e^{-\lambda_i^2 Fo} \tag{5}$$

The error associated with the utilization of Eq.(5) in the calculation of the dimensionless average temperature of an infinite slab is given in Table 3.

As can be observed from Table 3, for Fourier numbers greater than 0.1, the average temperature in a slab may be evaluated regardless of the Biot number with negligible error.

For a brick with constant physical properties of dimensions $2\Delta x$, $2\Delta y$ and $2\Delta z$, with a uniform initial temperature T_i , in an ambient temperature of T_a , the overall solution to the simultaneous unsteady heat conduction in all three directions can be found by superposition of the solutions in each of the three directions (Newman 1931):

$$\left(\frac{T - T_a}{T_i - T_a}\right)_{\text{brick}} = \left(\frac{T - T_a}{T_i - T_a}\right)_{2\Delta x} \left(\frac{T - T_a}{T_i - T_a}\right)_{2\Delta y} \left(\frac{T - T_a}{T_i - T_a}\right)_{2\Delta z} \tag{6}$$

Similarly the average temperature for the brick at a given Fourier number is

$$\left(\frac{T - T_a}{T_i - T_a}\right)_{\text{brick}} = \left(\frac{T - T_a}{T_i - T_a}\right)_{2\Delta x} \left(\frac{T - T_a}{T_i - T_a}\right)_{2\Delta y} \left(\frac{T - T_a}{T_i - T_a}\right)_{2\Delta z} \tag{7}$$

In the model the freezing operation was assumed to take place in two periods: An initial cooling period from a uniform initial temperature of T_i to an average temperature of \bar{T} and a freezing period in which the centerline temperature reached a prescribed value starting from an average temperature of \bar{T} . If such a model is assumed for the freezing operation and the solutions to the unsteady state heat conduction equation for materials having constant physical properties

TABLE 3.
PERCENT ERROR IN THE DIMENSIONLESS AVERAGE TEMPERATURES RESULTING FROM THE FIRST TERM APPROXIMATION FOR A SLAB

Fo	Bi = 0.2	Bi = 3	Bi = 10	Bi = ∞
	%E	%E	%E	%E
0	-0.1	-5.7	-12.6	-19.0
0.025	-0.1	-3.6	-6.7	-7.1
0.050	-0.0	-2.5	-4.2	-4.2
0.100	-0.0	-1.5	-1.6	-1.5
0.150	-0.0	-0.7	-0.8	-0.6

are to be used in the prediction of freezing times, two major points to be considered are the specification of the cooling period termination temperature and the determination of the effective thermal diffusivity for the freezing period.

In a previous paper (Ilıalı and Sağlam 1987) for foodstuffs having infinite slab, infinite cylinder or spherical shape, the initial freezing point of the foodstuff to be frozen was considered to be a reasonable choice for the cooling period termination temperature. Similarly in the present work it was assumed that the cooling period terminated when the average temperature in the brick reached the initial freezing point of the foodstuff.

The cooling times were calculated from Eq.(7). Only the first term of the series solution given in Eq.(1) was used in the calculations. Unfrozen properties were employed in the determination of the cooling period thermal diffusivity.

For an infinite slab the effective thermal diffusivity for the freezing period was defined by Ilıalı and Sağlam (1987) as

$$\alpha_{\text{eff}} = \frac{k_f}{\rho_f C_{\text{Peff}}} \quad (8)$$

where k_f and ρ_f are the thermal conductivity and density of the fully frozen food, respectively. The effective heat capacity C_{Peff} was defined as

$$C_{\text{Peff}} = \Delta H_{\text{eff}} / (\bar{T} - T) \quad (9)$$

where ΔH_{eff} was taken to be 75% of the experimental enthalpy change between the initial freezing point and the final center temperature at the end of freezing (Ilıalı and Sağlam 1987).

When the thermal diffusivities obtained from Eq. (8) and (9) were used in the prediction of freezing times of bricks, the predicted freezing times were generally higher than the experimental freezing times for the data sets considered. It is very difficult to explain theoretically the positive errors observed due to the complexity of the freezing of bricks compared to slabs. Large positive errors as high as 30% were obtained for some of the cases. Investigation of the calculation procedure revealed that the cause of the large positive errors observed was mostly due to the representation of the series solution given in Eq. (1) by the first term. Since the brick has three dimensions, the representation of the series solution by a single term can be valid for one dimension, while being invalid in the other directions. It can be calculated that for a ground beef brick having the dimensions of 2cm \times 4cm \times 15cm, assuming an effective diffusivity of 0.79×10^{-7} m²/s (Ilıalı and Sağlam 1987), having a freezing time of 1800 s which is a typical value for the test conditions in this research, the Fourier numbers in the x, y and z directions will be 1.42, 0.36 and 0.025, respectively. Using the first term of the series solution will be valid for the x and y directions. However, it will be invalid for the z direction. As the Fourier number approaches zero, the right

hand side of Eq.(3) will converge to A_1 . For a freezing Biot number of three, A_1 is equal to 1.2102 (Kutateladze and Borishanskii 1966). The invalidity of the assumption of the representation of the series solution by the first term will lead to positive prediction errors in the calculation of freezing times. Since the use of the slab effective thermal diffusivities reported in Ilıcalı and Sađlam (1987) generally resulted in positive prediction errors, the positive error in the predicted freezing times will be larger if Fourier number is less than approximately 0.1 for one or two directions of the brick. To compensate for the positive prediction errors encountered, greater effective thermal diffusivities must be used for the freezing period of bricks compared to the effective thermal diffusivities used by Ilıcalı and Sađlam (1987) for infinite slabs. Comparison with experimental data revealed that the predictions of the present model were improved to a large extent when the effective thermal diffusivities in the freezing period for each direction of the brick were taken to be 20% higher than the effective thermal diffusivities for the freezing period of infinite slabs reported in Ilıcalı and Sađlam (1987). Therefore, the slab effective thermal diffusivities were multiplied by 1.2 and used in the prediction of freezing times of bricks.

The freezing times were calculated from Eq. (6) where T_i was replaced by \bar{T} , the initial freezing point of the foodstuff and T was the center temperature of the foodstuff when the freezing operation was assumed to be completed. Only the first term of the series solution was employed in the predictions. The total freezing time was obtained from the summation of the cooling time and the freezing time.

Experimental Verification

Ground beef and mashed potato having moisture contents between 63 and 65, 79 and 81 weight percent respectively were chosen as the test material. Ground beef bricks frozen contained medium fat. Copper molds which were open on the two shortest sides and the top were used to form the bricks (Fig. 2). The thickness of the copper sheet from which the molds were made had a thickness of approximately 1 mm. Therefore, its resistance to heat transfer could be neglected without any appreciable error. The dimensions of the brick were 0.02m \times 0.04m \times 0.15m. Small holes were drilled to the mold for the insertion of the thermocouples.

Ground beef and mashed potato bricks having a uniform initial temperature were frozen in an Aga-Frigoscandia Laboratory type freezer. The variation in the temperature of the ambient air was ∓ 1 °C. Temperature profiles at the geometric center was measured by copper-constantan thermocouples connected to an Omega 2175 A Type digital thermometer and Dataplex automatic scanner. The experiments were terminated when the temperature at the geometric center reached -18 °C. After the termination of the experiments, the bricks

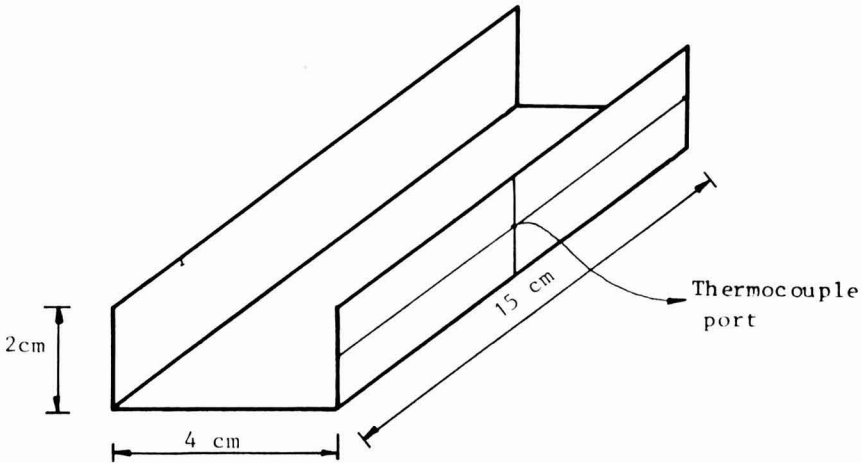


FIG. 2. COPPER SAMPLE-HOLDER USED IN THE EXPERIMENT

were thawed and the location of the thermocouple at the geometric center was examined. If the location of the thermocouple deviated more than 5 % of the smallest dimension of the brick from the geometric center, the run was discarded.

The bricks were frozen in three different orientations: Different orientations were obtained by making the direction of air flow parallel to different dimensions of the brick.

The heat transfer coefficients were obtained from the cooling curves of an Aluminum block having identical dimensions with the brick and located at the identical position in the freezer (Hung and Thompson 1983; Suzuki and Singh 1985).

The heat transfer coefficient was calculated from the slope of Eq. (10):

$$\ln \frac{T - T_a}{T_i - T_a} = - \frac{hA}{mC_p} t \quad (10)$$

where h is the heat transfer coefficient, A , m , and C_p are the surface area, mass and the specific heat of the Aluminum block, respectively. Highest heat transfer coefficients were obtained when the smallest dimension of the brick was parallel to the direction of air flow. Nine ground beef and ten mashed potato bricks were frozen. The initial temperature of the bricks varied from 18.8 °C to 27.2 °C. The ambient air temperatures were between -23.6 and -34.7 °C. The heat transfer coefficients varied from 63.3 W/m² to 74.9 W/m²K depending on the orientation of the brick with respect to the direction of air flow. For the ground beef bricks the cooling and freezing Biot number ranged from 1.43 to 12.77 and from 0.44 to 3.88, respectively. Similarly, for mashed potato bricks the cooling and freezing Biot number ranged from 1.20 to 10.66, and from 0.33 to 2.97, respectively.

RESULTS AND DISCUSSION

The predictions of the present model for bricks were compared to three independent data sets and to the experimental data obtained in this work for the freezing of ground beef and mashed potato bricks: Freezing of 72 Tylose bricks to a final center temperature of -10°C (Cleland and Earle 1979); the data of de Michelis and Calvelo (1983) for the freezing of 17 lean beef bricks to a center temperature of -10°C and 2 two dimensional bricks to a final center temperature of -18°C ; the data of Hayakawa *et al.* (1983a) for the freezing of 6 lean beef two dimensional bricks to final center temperatures of -15°C , -20°C and -27.8°C ; and to the freezing of 9 ground beef and 10 mashed potato bricks to two different final center temperatures; -10°C and -18°C . Table 4 lists the measured test conditions and the experimental freezing times of ground beef and

TABLE 4.
EXPERIMENTAL TEST CONDITIONS AND FREEZING TIMES TO TWO DIFFERENT
CENTER TEMPERATURES ($\Delta x = 0.01\text{ m}$, $\Delta y = 0.02\text{ m}$, $\Delta z = 0.075\text{ m}$)

Material	h ($\text{W/m}^2\text{K}$)	T_i ($^{\circ}\text{C}$)	T_a ($^{\circ}\text{C}$)	t_{exp}^a (h)	t_{exp}^b (h)
Ground beef	63.3	24.2	-23.6	0.53	0.62
	63.3	23.8	-29.0	0.48	0.52
	63.3	24.6	-34.7	0.41	0.45
	70.1	23.0	-24.3	0.47	0.52
	70.1	23.2	-27.7	0.44	0.48
	70.1	25.0	-34.5	0.34	0.37
	74.9	22.0	-23.6	0.43	0.52
	74.9	26.6	-27.0	0.41	0.48
	74.9	24.2	-33.1	0.34	0.38
	63.3	26.0	-24.5	0.60	0.67
	63.3	23.4	-28.4	0.52	0.56
	63.3	26.6	-33.4	0.47	0.50
	70.1	26.6	-24.7	0.50	0.56
	70.1	23.6	-28.5	0.40	0.44
Mashed potato	70.1	27.2	-34.7	0.40	0.43
	74.9	27.2	-24.1	0.51	0.57
	74.9	23.2	-28.3	0.38	0.45
	74.9	18.8	-28.6	0.37	0.42
	74.9	27.2	-32.6	0.36	0.40

^a indicates the time for the geometric center to reach -10°C

^b indicates the time for the geometric center to reach -18°C

mashed potato bricks to two different final center temperatures of $-10\text{ }^{\circ}\text{C}$ and $-18\text{ }^{\circ}\text{C}$.

The thermophysical data used in the predictions were taken from Cleland and Earle (1984b), Hung and Thompson (1983), Succar and Hayakawa (1983) and Schwartzberg (1977). The thermal data used in the predictions for the calculation of freezing times is given in Table 5. The effective thermal diffusivities for each direction of the brick was assumed to be constant. The effective thermal diffusivities used for the freezing period may be evaluated by dividing the frozen thermal conductivities to the effective volumetric specific heats for the freezing period given in Table 5.

In the model cooling times were calculated from Eq. (7). If the initial temperature of the foodstuff is low and the Biot numbers are high, the cooling times will be short and the representation of the solution given in Eq. (1) by the first term may not be valid. As shown in Table 3, if the cooling Biot number is low, the invalidity of the representation of the series given in Eq. (1) by the first term will have no significant effect on the predicted cooling times. At high Biot numbers percent error in the predicted cooling times becomes appreciable if the cooling Fourier number is much smaller than 0.1. For a Tylose cube having a cooling Biot number of 10, in an ambient temperature of $-20\text{ }^{\circ}\text{C}$, the cooling Fourier numbers for initial temperatures of $20\text{ }^{\circ}\text{C}$ and $10\text{ }^{\circ}\text{C}$ may be calculated as 0.052 and 0.03 by using Eq. (7). Utilization of the first term resulted in errors of -30% and -39% respectively in the predicted cooling Fourier numbers.

TABLE 5.
THERMAL DATA USED FOR THE CALCULATION OF FREEZING TIMES
BY THE PRESENT MODEL

Property	Tylose	Potato	Lean beef	Ground beef
k_l (W/mK)	0.55	0.53	0.51	0.44
k_f (W/mK)	1.65	1.90	1.55	1.45
C_l ($\text{J/m}^3\text{K}\times 10^{-6}$)	3.71	3.66	3.65	3.38
$\rho_r C_{p_{eff}}^a$ ($\text{J/m}^3\text{K}\times 10^{-6}$)	16.57	16.84	15.75	15.29
$\rho_f C_{p_{eff}}^b$ ($\text{J/m}^3\text{K}\times 10^{-6}$)	9.11	9.96	9.42	9.02

^a effective volumetric specific heat between the initial freezing point and $-10\text{ }^{\circ}\text{C}$ for each direction of the brick

^b effective volumetric specific heat between the initial freezing point and $-18\text{ }^{\circ}\text{C}$ for each direction of the brick

However, cooling times will be short compared to the freezing times. For the experimental conditions studied in this research, the cooling times were for most of the cases less than twenty percent of the total freezing time. Therefore, the effect of the error in the predicted freezing time will be small, although percent error in the predicted cooling time may be large depending on the cooling Fourier number and Biot number. In some cases Eq. (7) will only be satisfied by negative values of cooling times. The condition which will lead to negative cooling times is given in Eq. (11):

$$\left(\frac{\bar{T} - T_a}{T_i - T_a}\right) \tau \left(\frac{\bar{T} - T_a}{T_i - T_a}\right)_{2\Delta_x} \left(\frac{\bar{T} - T_a}{T_i - T_a}\right)_{2\Delta_y} \left(\frac{\bar{T} - T_a}{T_i - T_a}\right)_{2\Delta_z} \quad (11)$$

For such cases cooling times were taken to be zero. Since the actual times will be greater than zero, this will lead to negative errors in the predictions. However, the magnitude of the error will not be significant since the cooling times for such cases will be very small compared to the freezing times.

The freezing times were predicted by Eq. (6) using the effective diffusivities calculated from Table 5. The truncation of the series solution after the first term will have the reverse effect on the calculation of freezing times in comparison to cooling times. When one of the dimensions of the brick is much longer than the other two dimensions, or two of the dimensions of the brick is much longer than the third dimension the dimensionless temperature for these directions will essentially be equal to one. However, the model will predict dimensionless temperatures greater than one. The magnitude of the error will depend on the freezing Biot and Fourier numbers as shown in Table 2. The predicted freezing times for bricks using the effective diffusivities of infinite slabs (Ilcalı and Sağlam 1987) were generally higher than the experimental freezing times. This behavior and the invalidity of the representation of the series solution given in Eq. (1) by the first term for one or two directions of the brick encountered in some of the cases were taken into account by using effective thermal diffusivities which were twenty percent higher than the slab case for each direction of the brick. The total freezing time was found by summing the cooling and freezing times.

The mean errors and standard deviations obtained by using the present model are shown in Table 6. The predictions of two analytical models which were obtained by modifying the Plank (1941) equation (Cleland and Earle 1984a; Pham 1986) are also shown in Table 6. As can be observed from Table 6, the predictions of the present model were quite satisfactory. The mean error for most of the cases was less than $\pm 5\%$. The method of Cleland and Earle (1984a) and especially the method of Pham (1986) gave predictions having relatively large mean errors with the experimental data of Hayakawa *et al.* (1983a). The data of Hayakawa *et al.* (1983a) was obtained at a low ambient temperature

TABLE 6.
SUMMARY OF COMPARISONS BETWEEN EXPERIMENTAL AND PREDICTED
FREEZING TIMES BY THE PRESENT MODEL, THE METHOD OF CLELAND AND
EARLE (1984a) AND PHAM (1986). ALL DATA ARE EXPRESSED AS PERCENTAGE
DIFFERENCES FROM EXPERIMENTAL DATA

Data Source	Present model		Method of Cleland and Earle		Method of Pham	
	%E	s.d.	%E	s.d.	%E	s.d.
Cleland and Earle						
72 Tylose bricks	1.0	7.8	0.9	5.7	-0.1	4.6
de Michelis and Calvelo						
19 lean beef bricks	-0.1	14.0	8.2	10.0	4.1	6.6
Hayakawa et al.						
18 two dimensional bricks	0.3	7.7	12.2	7.3	28.8	8.6
Present data						
9 ground beef bricks ^a	1.3	6.6	-9.6	7.4	-7.6	5.7
10 potato bricks ^a	2.3	7.0	-9.8	7.2	-3.9	8.4
9 ground beef bricks ^b	-6.3	7.4	-5.1	6.0	-6.3	4.8
10 potato bricks ^b	-3.3	7.7	-9.6	5.6	-1.8	6.7

^a indicates a final center temperature of -10°C

^b indicates a final center temperature of -18°C

(-40°C), low heat transfer coefficients ($h \cong 10 \text{ W/m}^2\text{K}$) and low initial temperatures (4°C). Since these two models were essentially derived from the data of Cleland and Earle (1979) by curve fitting techniques, they represent this brick data with excellent accuracy. However, when the test conditions are varied, the original accuracy decreases significantly.

In the present model the anisotropic nature of thermal conductivity was not considered. Although this assumption is valid for Tylose, ground beef and mashed potato, it is not so for lean beef. For lean beef the thermal conductivities in the fully thawed state are 0.51 W/mK in the direction parallel to fibers and 0.45 W/mK in the direction perpendicular to fibers. These values for the fully frozen state are 1.51 W/mK and 1.17 W/mK , respectively (Schwartzberg 1977). The anisotropic nature of the thermal conductivity can also be taken account in the present model. For the two lean beef infinite square rods of de Michelis and Calvelo (1983), the percent error in the predictions were improved by 3% when two different thermal conductivities were used depending on the direction of heat flow.

The cooling Biot number range for which the model was tested was from 0.58 to 33. The Biot number range for the freezing period was approximately one

third of the above values. The ambient air temperatures were between -19.7 and -40.7 °C. The initial temperatures of the bricks were between 1.9 °C and 32.1 °C. The ratio of the second shortest dimension to the shortest dimension was between 1 and 4. The ratio of the longest dimension to the shortest dimension was between 1 and infinity.

CONCLUSIONS

An analytical model for the prediction of freezing times of brick-shaped food-stuffs was developed. The solution to the unsteady, one-dimensional heat conduction equation with constant physical properties was assumed to hold for each of the three directions of the brick. The cooling and freezing times were calculated from Newman (1931) rule. Defining an effective thermal diffusivity for the freezing period resulted in predictions which agreed closely with experimental data. Mean errors varying between -6.3 % and 2.3 % and standard deviations from the mean being between 6.6 % and 14.0 % were obtained for the data sets considered.

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NOMENCLATURE

A	Heat transfer area, m ²
A ₁	Constant in Table 1 and Eq. (3), dimensionless
A ₂	Constant in Table 1, dimensionless
A ₃	Constant in Table 1, dimensionless
Bi	Biot number, $h\Delta x/k$, $h\Delta y/k$, $h\Delta z/k$, dimensionless
C ₁	Volumetric specific heat capacity of the unfrozen material, J/m ³ K
C _p	Specific heat capacity, J/kgK
C _p ^{eff}	Effective heat capacity, defined by Eq. (9), J/kgK
%E	Percent error, dimensionless
Fo	Fourier number, $\alpha t/\Delta x^2$, $\alpha t/\Delta y^2$, $\alpha t/\Delta z^2$, dimensionless
h	Heat transfer coefficient, W/m ² K
ΔH_{eff}	Effective enthalpy difference for the freezing period, J/kg
k	Thermal conductivity, W/mK
m	Mass of the aluminum block, kg
s.d.	Standard deviation, dimensionless
t	Time, seconds in Eq. (10), hours in Table 4
T	Temperature, °C

T_a	Ambient temperature, °C
T_i	Initial temperature, °C
\bar{T}	Average temperature at the onset of freezing, °C
x	Distance from the center of slab, m
Δx	Half-thickness of slab or half-thickness of the shortest dimension of the brick, m
Δy	Half-thickness of the second shortest dimension of the brick, m
Δz	Half-thickness of the longest dimension of the brick, m

Greek Letters

α	Thermal diffusivity, $k/\rho C_p$, m^2/s
α_{eff}	Effective thermal diffusivity, $k_f/\rho_f C_{peff}$, m^2/s
λ_1	First root of Eq. (2), rad
λ_2	Second root of Eq. (2), rad
λ_3	Third root of Eq. (2), rad
λ_n	nth root of Eq. (2), rad
ρ	Density, kg/m^3

Subscripts

a	Ambient
eff	Effective
exp	Experimental
f	Frozen phase
i	Initial
l	Unfrozen phase

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ON-LINE VISCOMETRY FOR PUREED BABY FOOD: CORRELATION OF BOSTWICK CONSISTOMETER READINGS AND APPARENT VISCOSITY DATA

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ABSTRACT

The relationship between Bostwick Consistometer (BC) readings and apparent viscosity was evaluated as an initial step in developing an on-line viscometer for pureed baby food. Rheological properties of three baby food products (apple-sauce, carrots, and vegetable beef dinner) were investigated using a BC and a concentric cylinder viscometer in a temperature range from 73 to 92 C. Shear-thinning, power law fluid behavior was found for all products. The overall correlation between results of the two methods of measurement, considered in terms of the Bostwick readings and apparent viscosity, was very poor. Developing an on-line viscometer for pureed baby food products based on the relationship between Bostwick readings and apparent viscosity is not recommended.

INTRODUCTION

Monitoring rheological quality during processing is necessary during the production of pureed baby food. This concern is thought of in terms of product consistency and evaluated using a Bostwick Consistometer (BC). The BC is a low cost, durable and simple instrument that is widely accepted in industrial practice. A major problem with the device is that it cannot be used in generating rheological information for continuous process monitoring or on-line control of food processing systems. More fundamental information, such as apparent viscosity, must be generated for these applications. BC readings, however, cannot be simply discarded because they are so firmly entrenched in current practice: BC readings are used to access final product quality and to check consistency during various stages of baby food processing.

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The logical first step in considering on-line rheological measurements for baby food is to determine the relationship between BC readings and fundamental rheological parameters over the range of temperatures and conditions found in processing. The reason being that on-line viscometers measure apparent viscosity (or a similar parameter), never Bostwick numbers. If there is a good correlation between the measurements, on-line viscosity measurements could be directly related to current quality control criteria involving BC readings.

A number of important considerations were instrumental in formulating this study. First, if an on-line viscometer (or process monitoring device) for pureed baby food is to be economically feasible, there must be good correlation between BC readings and apparent viscosity with different types of products—fruit, vegetable and meat products. The correlation must be good for specific products *and* across product lines! This is true because baby food processing lines are used to manufacture a wide variety of products and regularly changed over from one product to another. The second, and very encouraging, factor was the result of an earlier study (Rao and Bourne, 1977) indicating a correlation between the BC readings of many pureed foods and apparent viscosity. The information in that study was not directly applicable to our problem because their data was not collected at the processing temperatures where an on-line viscometer would operate. In addition, Rao and Bourne (1977) noted that the BC does not work with some foods, such as hydrocolloid solutions, that have a tendency to stick to the gate of the consistometer.

Given the above considerations, the following objectives were formulated for this study:

- (1) Determine the correlation between BC readings and apparent viscosity, across product lines, for different types of pureed baby food products under the processing conditions relevant to on-line viscometry.
- (2) Evaluate the possibility of using the correlation between BC readings and apparent viscosity for developing an on-line viscometer or a quality monitoring device for pureed baby food products.

REVIEW OF LITERATURE

The BC consists of a 25 cm long, stainless steel, rectangular trough closed at one end with a floor that is graduated in 5 mm increments. One end has a spring-loaded valve that forms a compartment with a capacity of 100 cm³. At the time of the measurement the sampling container is filled to the top and the valve is released. Fluid starts to flow under its own weight, and movement down the trough reflects fluid properties. The instrument has a fixed inclination to increase the flowrate and to overcome a small yield stress, if present. Measurements are taken after a specified time and are reported in units of cm/s or cm/

min, thus the apparent physical quantity would be velocity. However, since the time is fixed for a particular test, readings are actually distances.

Bostwick readings are only meaningful if they are totally described and specified. There is poor agreement in the literature upon the fixed testing time: Bookwalter *et al.* (1968) read the Bostwick value after exactly 1 min, Davis *et al.* (1954) and Luh *et al.* (1954) took readings after 30 s, Rao and Bourne (1977) obtained their data after 10 s. A typical industrial testing time for pureed baby food products is 5 s. Testing time is closely related to the consistency of the product and of the speed one wants to perform the measurements. Many variables affect the readings: product loading, testing time, nature of the product, location of the reading (center or wall), trough inclination, geometry and surface roughness of the trough, trough temperature and overall condition (old, new, dropped, etc.).

Davis *et al.* (1954) reported BC readings as an average of the values obtained at the center and walls. Marsh *et al.* (1980) used a special consistometer with a trough, 50 cm long, made of plexiglass acrylic plastic. The effect of temperature on the Bostwick reading has been investigated by Davis *et al.* (1954). They reported a linear relationship in a range from 21 to 87 C for tomato puree.

MATERIALS AND METHODS

Three strained baby food products—applesauce, carrots and vegetable beef dinner (Gerber Products Company, Fremont, Michigan)—were examined at four temperatures reflecting actual processing conditions: approximately 73, 80, 86 and 92 C. All materials were received in jars as finished products. It was not practical to take samples directly from the processing line because production tends to be seasonal and the subjects of this study are produced during different times of the year.

Since the desire was to evaluate the product under processing conditions, it was assumed that the product was fully broken down (mechanically) in the process line. This assumption is very reasonable considering the presence of finishing screens (diameter = 0.84 mm) in the processing system. To achieve complete mechanical degradation of the experimental materials, each product was stirred in a 4.5 oz (127 g) baby food jar for 12 min until the time-dependency was removed. This was accomplished with a mixer, turning at approximately 400 rpm using a three-blade, marine-type mixing propeller. The mixing occurred while the container remained in a temperature controlled waterbath. A lid, with a hole for the impeller shaft, was left on the jar to minimize product evaporation.

After stirring, all products were checked for complete breakdown using a concentric cylinder viscometer (Haake RV-12). A thixotropy test was performed by recording the torque over time, while the product was sheared at a constant

speed (50 rpm). The temperature was controlled using a Haake FC-3 waterbath. The fully broken down and heated sample was loaded into the compartment of the BC and the gate was opened. Bostwick readings were recorded after 5 s. Four replicates were taken for each product at each temperature.

A Haake RV-12 concentric cylinder viscometer equipped with a MVI sensor system and a M150 head was used to collect steady shear rheological data. Sample preparation techniques and temperatures (73, 80, 86 and 92 C) were identical to those considered in BC testing. A small protective lid covered the samples to prevent evaporation. Test speed ranged from 2-150 rpm which generate shear rates (approximately 10 to 400 s^{-1}) typical of commercial processing operations. Twenty five data points were taken in this range for each test. Soluble solids content (or sucrose concentration) was measured with an Abbe-3L Refractometer (Bausch and Lomb). Values for applesauce and carrots were 9.0% and 6.8%, respectively. The total solids content data were determined using a drying oven operated at 103 C for 24 h. Values for applesauce, carrots and vegetable beef dinner were 20.7%, 18.1% and 25.3%, respectively.

RESULTS AND DISCUSSION

Results for the Bostwick readings are presented in Fig. 1. Variation observed among the four replicates may be partially attributed to the force needed to release the gate, which produced a different shock wave with each test. This

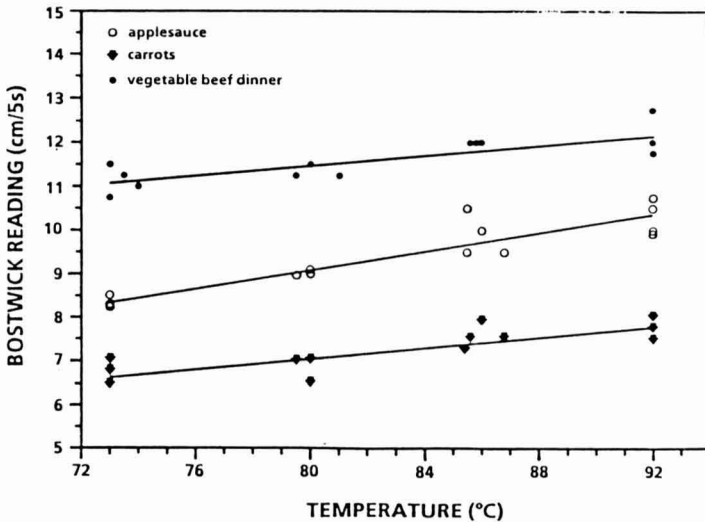


FIG. 1. EFFECT OF TEMPERATURE ON BOSTWICK CONSISTOMETER READINGS

shock wave, which has not received adequate attention in published literature, was observed to be an important factor in data collection. Different results can be obtained if the shock wave is reduced by holding the instrument (pushing the instrument to the table with the hand) during testing. One of the problems with the holding technique is that different operators (authors observed this in a food plant setting) provide different degrees of instrument stability while holding the instrument. In an effort to achieve reproducible results in the current study, the gate was always released without holding the instrument.

The flow pattern in the trough was curved with the highest velocity at the center where measurements were taken. Carrots was the only product which showed a separation of the liquid and the semi-solid portion and the data given in Fig. 1 is for the semi-solid portion only.

Bostwick readings increased with increasing temperature. As mentioned earlier, Davis *et al.* (1954) found a linear relationship between the Bostwick measurements and the temperature in a range from 20-90 C for tomato puree. Assuming similar linearity, the following equations were obtained for the products in the current study:

applesauce	BR = .11 T + 0.60	r ² = .87
vegetable beef dinner.	BR = .06 T + 6.98	r ² = .64
carrots.	BR = .06 T + 2.18	r ² = .77

where: BR = BC reading at 5 s (cm/5s)
 T = temperature of the product (C)

The regression lines for these equations are plotted in Fig. 1. The effect of temperature, in the 70–92C range, on BC readings is clearly shown by the equations. Carrots and vegetable beef dinner were only half as sensitive to temperature as the applesauce.

The method developed by Krieger (1968) was used to determine the shear rate and shear stress from the concentric cylinder viscometer data. A nonlinear regression analysis was carried out on the combined data from the four replicates (100 data points at each temperature) using the technique employed by Barbosa and Peleg (1983). The power law model and the Herschel-Bulkley (Herschel and Bulkley 1926) model were both fitted. The Herschel-Bulkley equation only gave a marginal improvement in curve fitting producing intercept yield stresses generally less than .05 Pa. Hence, the yield stress was taken as zero and the power law model was accepted as an accurate reflection of the rheogram. Slip was not evident but may have been present because the experimental samples were suspensions. This phenomenon would also be a problem in an on-line viscometer and no attempt was made in the present work to calculate slip coefficients.

The power law parameters (Table 1) indicate that the flow behavior index is not highly temperature dependent. The consistency coefficient, however, is strongly influenced by temperature variations. This is particularly evident for the vegetable beef dinner. In addition, the low correlation coefficients (Table 1) indicate a wide spread in data points which is also seen in Fig. 2. The degree of variation was different for each product being most serious for the vegetable beef dinner.

The effect of temperature on the apparent viscosity (shear stress divided by shear rate) was summarized using the Arrhenius equation:

$$\text{Apparent viscosity} = A \exp [E_a/(R T)]$$

where: E_a = activation energy (J/mol)

R = universal gas constant (8.314 J/ mol K)

A = constant (Pa s)

T = absolute product temperature (K)

A nonlinear regression was carried out using apparent viscosities evaluated at 200 s^{-1} . This shear rate was selected because it accurately reflects processing conditions and is a middle range value between the shear rates (50 to 400 s^{-1}) considered in the Rao and Bourne (1977) study. Figure 2 represents the regression lines through the apparent viscosities of the four replicates at each temperature for the different products. The parameters obtained by this method are sum-

TABLE I.
POWER LAW PARAMETERS FOR DIFFERENT PRODUCTS DETERMINED FROM
CONCENTRIC CYLINDER VISCOMETER DATA

Product	Temperature (°C)	K^* (Pa s ⁿ)	n^{**} (-)	Correlation Coefficient
Applesauce	73	2.59	0.44	0.98
	80	3.24	0.35	0.94
	86	2.30	0.32	0.99
	92	2.19	0.30	0.97
Carrots	73	4.55	0.29	0.90
	80	5.80	0.23	0.85
	86	5.25	0.20	0.96
	92	2.98	0.22	0.98
Vegetable beef dinner	73	3.42	0.45	0.84
	80	2.47	0.48	0.94
	86	2.15	0.51	0.99
	92	0.42	0.49	0.70

*Consistency coefficient

**Flow behavior index

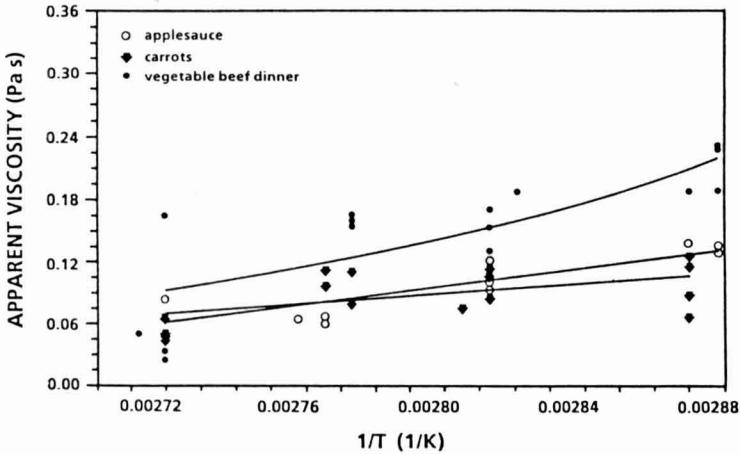


FIG. 2. EFFECT OF TEMPERATURE ON APPARENT VISCOSITY EVALUATED AT 200 S^{-1}

marized in Table 2. Parameters reflect the general behavior of the data but specific correlation to the Arrhenius equation is not good.

Results (Fig. 2) show that the average flow behavior index is proportional to the activation energy. The higher the value of n (less shear-thinning behavior), the higher the activation energy and the greater the effect of temperature on the apparent viscosity. This relationship is in agreement with earlier observed data on fruit purees and juices (Holdsworth 1971). Carrots had almost no temperature dependency under the experimental conditions considered. Applesauce and vegetable beef dinner showed a small temperature effect. The same trend was observed with the BC, although applesauce was a little more sensitive at those low shear rates (Fig. 1).

It is difficult to bring together the results of the two methods of measurement—BC and concentric cylinder—because there are some inherent differences. The

TABLE 2.
ACTIVATION ENERGY AND CONSTANT FOR DIFFERENT PRODUCTS

Product	A (Pa s)	E_a (kJ/mol)	Correlation Coefficient
Applesauce	$9.2 \cdot 10^{-8}$	40.76	0.92
Carrots	$7.4 \cdot 10^{-5}$	20.87	0.50
Vegetable beef dinner	$3.2 \cdot 10^{-8}$	45.16	0.78

Bostwick is a low shear rate device which reports a distance traveled over a period of time. In contrast, the concentric cylinder viscometer data are investigated in terms of two or more parameters eventually leading to an apparent viscosity. The relationship between BC readings and apparent viscosity must be considered to evaluate the possibility of developing an on-line viscometer or a quality monitoring device for pureed baby food products.

Averaging the previously reported data, the influence of temperature on the relationship between BC and apparent viscosity may be illustrated. With three points to represent four different temperatures, Fig. 3 indicates the pattern of the relationship for each product. Data points between the different products for a constant temperature are connected. Apparent viscosity increases with increasing BC readings at 73 and 80 °C but irregular behavior is observed at the higher temperatures (Fig. 3). The tendency of apparent viscosity to be inversely proportional to the BC readings is consistent for each product; however, given the poor correlations found between flow behavior data and temperature (previously discussed), this tendency is not reliable.

Overall, a general pattern of behavior between BC readings and apparent viscosity does not emerge (Fig. 3). Hence, the hope of finding a general correlation between BC readings and apparent viscosity over a range of pureed baby food products was not realized. Unfortunately, under the conditions of our work, the excellent relationship found, by Rao and Bourne (1977), between BC readings and apparent viscosity was not observed. It should be noted that conducting the

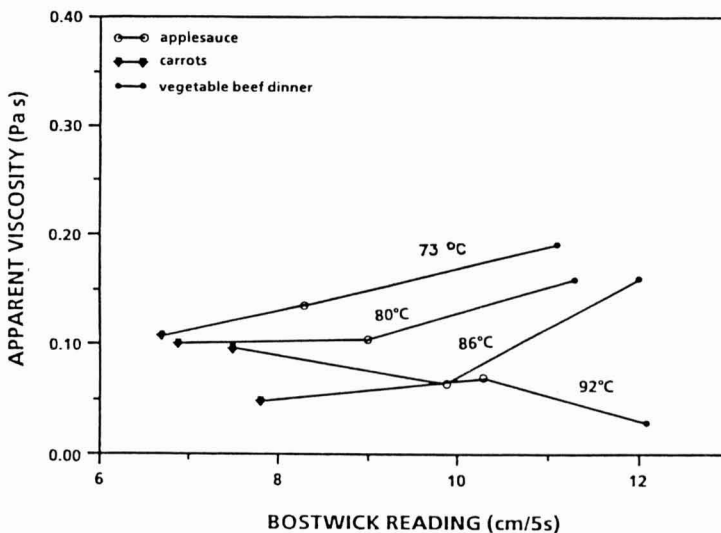


FIG. 3. BOSTWICK CONSISTOMETER READINGS VERSUS APPARENT VISCOSITY, EVALUATED AT 200 S^{-1}

analysis with the apparent viscosity calculated at a different shear rate would have little effect on the overall shape of the curve; it would only shift the curves (Fig. 3) to the right or left depending on whether or not the shear rate was increased or decreased.

The lack of good correlation between BC readings and apparent viscosity means that it is unacceptable to develop an on-line viscometer, or rheological process monitor that measures apparent viscosity as an index of Bostwick consistency. The relationship is poor for a specific product at different temperatures and across products lines (fruit, vegetable and meat) at the same temperature. The idea of getting some sort of continuous measurement of Bostwick consistency should be abandoned. On-line viscometers for pureed baby food will not be attractive until rheological quality is specified in terms of apparent viscosity or a similar basic rheological parameter. This change is inevitable if the long range goal is to develop process control systems based on material flow behavior.

SUMMARY AND CONCLUSION

Rheological properties of three baby food products have been determined in two ways: with a BC and a concentric cylinder viscometer. Poor overall correlation was found between BC readings and apparent viscosity for each product (applesauce, carrots, vegetable beef dinner) and for all products considered together. Development of an on-line viscometer (or process monitoring device) for pureed baby food based on the relationship between apparent viscosity and BC readings is not recommended.

ACKNOWLEDGMENTS

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INCORPORATION OF MILK PROTEINS HARVESTED BY DIRECT ACIDIFICATION INTO PROCESS CHEESE PRODUCTS

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ABSTRACT

Casein curd, a 5:1 combination of casein curd and lactalbumin, traditional coprecipitate curd, and Hiddink's coprecipitate curd were each used to replace varying amounts of natural cheese protein in a process cheese product.

Traditional coprecipitate was used successfully to replace up to 40%, and Hiddink's coprecipitate up to 50%, of natural cheese protein in an experimental product. At higher levels of replacement, the product mixtures would not become homogeneous. Casein, and the combination of casein and lactalbumin, were used to replace up to 80% of natural cheese protein in an experimental product.

Experimental products were compared rheologically to a group of similar use commercial products. Stress-strain, stress relaxation, and creep behavior indicated that the experimental products were generally more rigid than the commercial products. The meltability of all the experimental products was similar to that of the commercial products.

INTRODUCTION

Process cheese products have been important commercial foods since the early years of this century (Meyer 1973). A general production method, outlined in Fig. 1, allows the manufacture of a wide variety of products by adjustment of processing parameters, product composition, and basic ingredients.

In recent years approximately a billion pounds of process cheese products have been manufactured in the United States annually (Kosikowski 1977). Most

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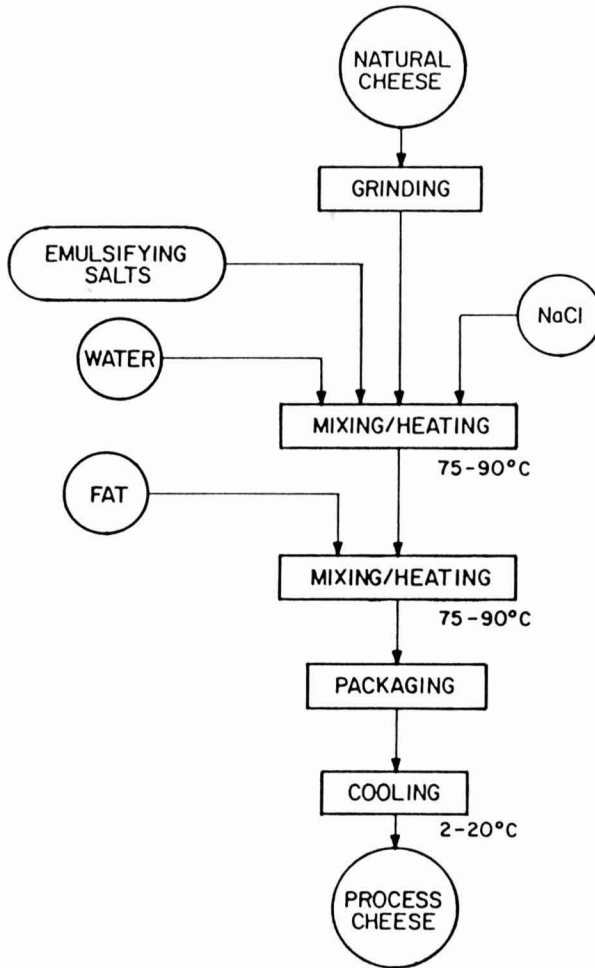


FIG. 1. GENERAL PROCESS CHEESE PRODUCTION FLOWCHART

of these products use natural cheese as the basic raw material. Unfortunately, natural cheese production is relatively inefficient in harvesting proteins from milk, removing only 80% of total milk proteins, and is a complex process generally involving both fermentation and the use of the enzyme coagulant rennet. A method of harvesting milk proteins for use in process cheese products that increases protein yield, while avoiding fermentation and renneting could thus translate into a direct increase in product yield, while reducing operating and capital costs.

Processes using ultrafiltration to produce a milk concentrate for subsequent incorporation into process cheese products as a partial replacement for natural cheese have been shown to be feasible (Sood and Kosikowski 1979; Ernstrom *et al.* 1980). While these methods harvest virtually all the milk proteins, they are somewhat complex and the ultrafiltration equipment involved is relatively expensive.

Milk proteins may be divided operationally into three groups (Brunner 1976; Walstra and Jenness 1984). The caseins are defined as those proteins which precipitate from 20°C milk at pH 4.6. They represent approximately 80% of the milk proteins. The whey proteins are defined as those proteins which, remaining in solution after the precipitation of the caseins, may be precipitated by applying a fairly severe heat treatment (95°C, up to 30 min) and then acidifying to pH 4.6. They represent approximately 15% of the milk proteins. The proteoseptones are defined as those proteins that remain in solution after the caseins and whey proteins are removed from the milk as outlined above.

The caseins and whey proteins, in keeping with the use of nonfermentative, nonrenneted processes, may be harvested from milk quite easily in a variety of forms by the use of heating, manipulation of pH, or a combination thereof. Basically, the caseins and whey proteins may be harvested separately, or in an associated form. The four ways in which milk proteins were harvested for use in a specific process cheese product in this research are discussed below.

Casein Curd

The use of acid precipitated casein curd as a replacement for natural cheese protein in process cheese has been shown to be feasible (Rosenau *et al.* 1982). Casein curd is formed by direct acidification of skim milk, heated to firm and aid in whey expulsion from the curd, and then collected. The curd may then be used in any proportion (total protein replacement possible) with traditional cheese in the manufacture of process cheese products. Specific products manufactured in this manner, even with total replacement of cheese protein by casein curd, have been shown to be similar to traditional process cheese (Rosenau *et al.* 1982; Rosenau *et al.* 1978).

Unfortunately, this method of milk protein use has the same disadvantage as that encountered in traditional cheese manufacture, i.e., only approximately 80% of the total milk protein is harvested.

Casein Curd and Lactalbumin

This method of protein replacement involves the use of casein curd as discussed above, as well as the heat precipitated whey proteins, generally known as lactalbumin. Once the casein is removed from milk, the resulting whey is heat

treated to precipitate the whey proteins. These proteins are then collected, usually by centrifugation (Robinson *et al.* 1976; Greig 1979).

The use of casein curd and lactalbumin allows approximately 95% of the total milk proteins to be used in process cheese products.

Traditional Coprecipitate

Traditional coprecipitate is a heterogeneous total milk protein curd formed by heat-treating milk (95°C, 15 min) prior to curd formation by direct acidification to approximately pH 4.8–5.3 (Southward and Aird 1978). The protein curd thus precipitated involves an association of the caseins and whey proteins, and so represents approximately 95% of the milk proteins.

Hiddinck's Coprecipitate

Hiddinck's coprecipitate is also a total milk protein curd, but one claimed to have a greater aqueous solubility than a traditional coprecipitate (Hiddinck 1985). The pH of the milk is raised to between 7.0 and 7.5 prior to heat treatment (95°C, 15 min), after which the protein curd is formed by direct acidification to approximately pH 4.4–4.7.

The objectives of this work were:

- (1) To produce a group of "standardized" process cheese products using each of the protein sources discussed above to replace natural cheese protein to various extents.
- (2) To compare specific rheological properties of the process cheese products manufactured to those of a group of commercially available process cheese products.

MATERIALS AND METHODS

Equipment

- (1) 25 gallon vertical stainless steel tank with heating/cooling jacket and mechanical agitator (Cherry Burrell Univat).
- (2) Hydraulic platen press (Wabash Metal Products, Model 30-24-SM).
- (3) Atmospheric pressure, gravity convection oven (Blue M Model OV-8A).
- (4) Conical collander with 2 mm holes.
- (5) Auger type grinder with 3.2 mm ($\frac{1}{8}$ in) die plate (Toledo Chopper Model 5125).
- (6) Process cheese cooker with heating/cooling jacket and variable speed agitator (Charles Ross & Son Co., Model LDM-2).
- (7) Instron Universal Testing Machines (Model TM equipped with a 50 lb load cell and Model 1000 equipped with a 50 kg load cell).

- (8) Creep testing apparatus (Purkayastha *et al.* 1985), interfaced with an Apple II+ computer via an Analog to Digital converter (AI13 Data Acquisition System, Interactive Structures Inc.) to allow digital collection of data. The computer was also equipped with a real time clock (Timemaster II H.O., Applied Engineering).

Reagents and Ingredients

- (1) Skim milk (raw, obtained from a local dairy).
- (2) Partially hydrogenated vegetable (soybean) oil (Durkee, Code 321).
- (3) Cheddar cheese (Stop & Shop brand—Mild).
- (4) Lactalbumin (New Zealand Milk Products—Alatal 820).
- (5) Hydrochloric acid, concentrated (approximately 37%), ACS grade (Fisher Scientific Co.).
- (6) Disodium phosphate (heptahydrate), ACS grade (Fisher Scientific Co.).
- (7) Trisodium citrate (dihydrate), ACS grade (Fisher Scientific Co.).
- (8) NaOH, ACS grade (Fisher Scientific Co.).
- (9) Various commercial process cheese products.

Protein Harvesting

Casein curd, traditional coprecipitate curd, and Hiddink's coprecipitate curd were produced from skim milk as depicted in Fig. 2.

All protein curds were manufactured in the CB Univat, drained in the collander, and pressed (approximately 1 min @ 750 kPa) using the hydraulic press (cheese-cloth media) described above. After pressing, the protein curds were placed in plastic bags (two layers) and stored at approximately 1–4°C for up to 2 weeks.

Curd Moisture Content

Curd moisture content, necessary to calculate product formulations, was determined by drying to constant weight (approximately 30 h @ 100°C) in the atmospheric pressure oven. (Similar to Kosikowski 1977.) Curd protein content was assumed to be 95% of the dry matter in all cases.

Product Manufacture

Experimental process cheese products were manufactured as depicted in Fig. 3 and 4, using the grinder and process cheese cooker.

In order to effectively compare experimental process cheese products, the desired product composition and the processing conditions were standardized. Compositional standards were calculated using average composition data for cheddar cheese (Kosikowski 1977; Watt & Merrill 1963). Compositional and processing parameters chosen for the experimental products are shown in Table

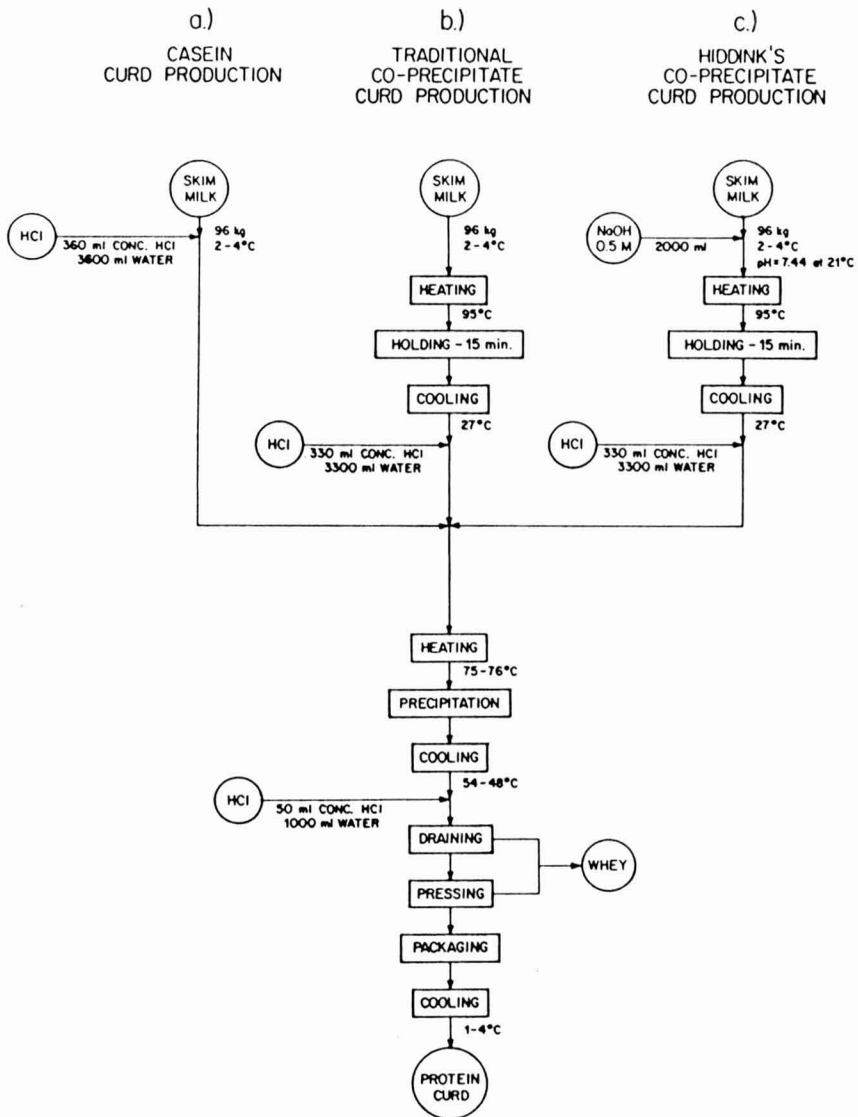


FIG. 2. PROTEIN CURD PRODUCTION FLOWCHARTS

1. As indicated, a standard processing time of 15 min was used. If, after this time, the product remained nonhomogeneous, an additional 3 min of processing time was allowed. If after this time the product was homogeneous, processing was ended. If the product remained nonhomogeneous, processing was continued

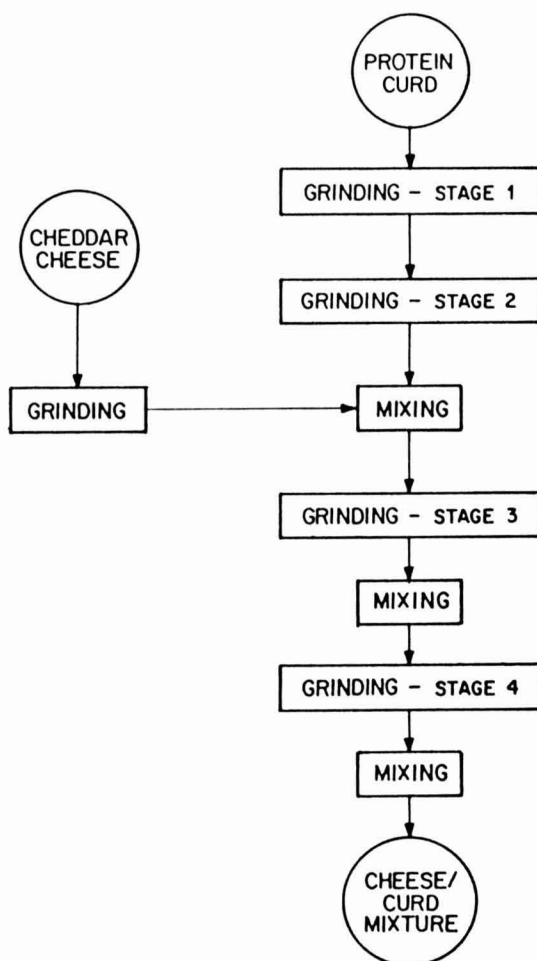


FIG. 3. GRINDING PROCEDURE FOR PRODUCT MANUFACTURE

for 3 min longer. Processing was terminated after this time; if the product remained nonhomogeneous, the batch was considered a failure.

After processing was ended, the successful (homogeneous) products were spooned into metal loaf pans, immediately covered by three layers of plastic wrap, and placed into storage as shown in Table 1.

Rheological Evaluation of Products

Samples for all rheological testing were left (in block form) for 20 to 24 h at an ambient temperature of 22°C ($\pm 1^{\circ}\text{C}$). Cylindrical samples approximately

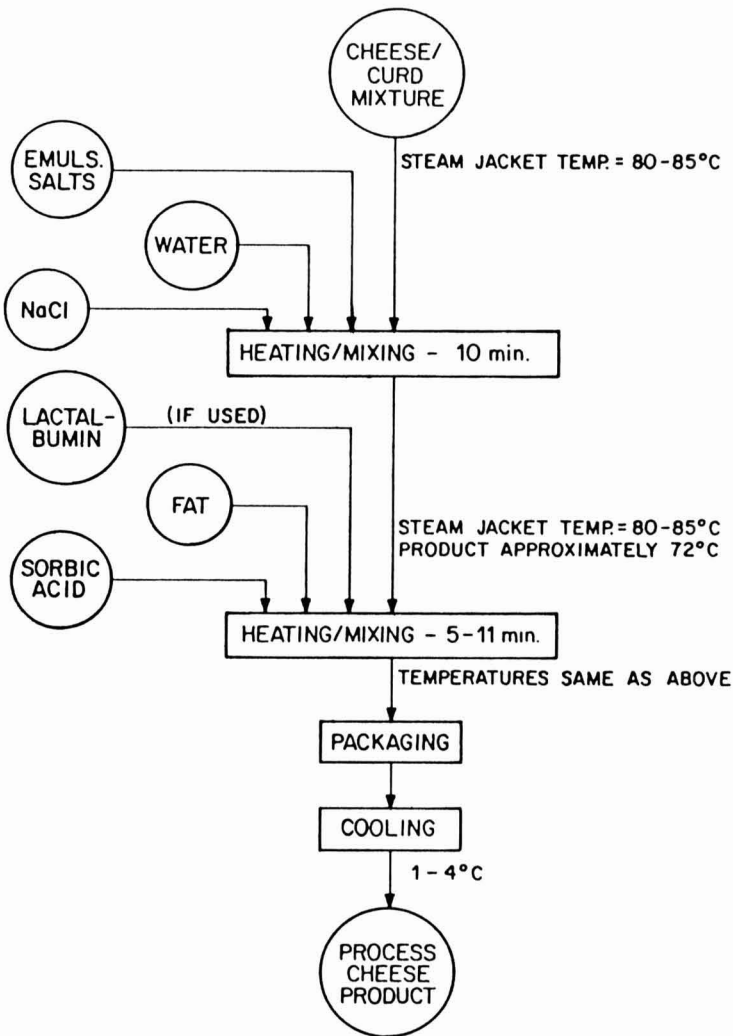


FIG. 4. PROCESSING PROCEDURE FOR PRODUCT MANUFACTURE

2.15 cm in diameter and 1.5 cm in height were then cut using a cork borer and a parallel blade cutter immediately before stress-strain, stress relaxation, or creeping testing. For melting, cylindrical samples approximately 4 cm in diameter and 0.5 cm in height were cut using a cylindrical borer and a wire cheese cutter immediately before testing.

(1) True stress-true strain behavior was determined using the Instron UTM

TABLE 1.
PROCESSING PARAMETERS OF EXPERIMENTAL PROCESS CHEESE PRODUCTS

PROXIMATE COMPOSITION

23% Protein
30% Fat
40% Moisture
1.29% NaCl
0.2% Sorbic Acid

EMULSIFYING SALTS

1.25% Sodium Citrate (dry basis)
1.25% Disodium Phosphate (dry basis)

PROCESSING TEMPERATURE

Jacket temperature maintained at 80–85°C

PROCESSING TIME

15–21 min

AGITATION

Cooker speed 1 for 1 min
Cooker speed 3 for 7 min
Cooker speed 7 for duration of processing
(Cooker speed scale from 1 to 7)

BATCH SIZE

5 lb

COOLING

At a temperature of 1–4°C

STORAGE

At 1–4°C for 4–5 days days prior to rheological testing

(Rosenau *et al.* 1978). The force versus time data generated by the Instron was converted to true stress and true (Hencky's) strain in the following manner:

True strain was calculated using the equation:

$$\text{True strain} = \ln (HO/H(t)) \quad (1)$$

Where HO = original sample height, and H(t) = sample height at time = t.

True stress was calculated by corrected for changing cross sectional area (assuming constant sample volume, and circular cross section) using the equation:

$$\text{True stress} = F(t)/A(t) \quad (2)$$

Where F(t) = force at time = t, and A(t) = cross sectional area at time = t.

Failure was defined as the point of the first sharp decline in the force versus time curves generated by the Instron. In the cases where a sharp failure point

was not evident, the force versus time curves generated by the Instron were digitized on a graphics tablet (Houston Instruments Hi-Pad) and then converted to true stress versus true strain curves using an Apple II computer. This allowed the graphical depiction of true sample behavior over the entire testing period.

A deformation rate of 10 cm/min, was used for all samples.

(2) Stress relaxation, performed using the Instron UTM, allowed the residual stress present after a specific relaxation time (8 min) to be determined for a specific strain (Rosenau *et al.* 1978). The ratio of the residual stress and the initial stress generated was then used for product comparison.

A specific true (Hencky's) strain of approximately 0.25 and a deformation rate of 10 cm/min were used.

(3) Compressive creep testing was performed using the general procedure given in Purkayastha *et al.* 1985. Data was collected digitally for a period of 13 to 16½ min using the computer and interfacing equipment described above. One data point per second was collected. This data, recording change in sample height for a specific loading over time, was converted to true compliance using the equation:

$$\text{Compliance} = J = \text{True Stress/True Strain} \quad (3)$$

Compliance curves thus generated were then fit via linear regression to the equation (MacCarthy and Rosenau 1985)

$$J = Kt^n \quad (4)$$

by conversion to the form $\ln(J) = \ln(K) + n \ln(t)$, allowing the constants K and n to be used to compare products.

An initial stress of 10.4 kPa was used for all samples.

(4) Melting properties were evaluated using an adaptation of the Schreiber test, in which the diameter increase of a circular cheese slice of specific dimensions is measured after heating under defined conditions (Kosikowski 1977; Park *et al.* 1984). Samples prepared as given above were placed in the center of a glass petri dish, which was then covered and placed in a preheated (232°C) gravity convection oven for 5 min. After removal from the oven, the sample was allowed to cool briefly at room temperature and then scored using a template with numbered concentric circles. The innermost circle, number 0, had a radius of 20 mm, while each of the higher numbered circles (numbered 1 through 10) increased in radius by 2.5 mm over the previous one. Scores were assigned at six equally spaced radii, and then averaged.

Experimental Procedure

(1) Using the objective rheological tests outlined above, the following types and brands of sliceable, block type commercial process cheese products were evaluated to define an envelope of commercial acceptability:

Process cheese

- Land-o-Lakes Process American Cheese
- New Yorker Process American Cheese
- Stop & Shop Process American Cheese
- Waldbaum's Process American Cheese

Imitation cheese

- Universal Foods Imitation American Cheese (Uni-Chef)

Process cheese product

- Naturally Slender Process Cheese Product

(2) A standard product using only natural cheese as a protein and fat source was manufactured using the parameters defined in Table 1 to provide a basis of comparison for both commercial and other experimental products.

(3) To establish the maximum level of traditional cheese protein that could be replaced by each of the coprecipitate protein sources, a series of products was made, each with an increasing amount of cheese protein replaced. Replacement increments of 20% were used initially. When a replacement level was reached where the batch failed to form a homogeneous product under standard processing conditions, an intermediate batch only 10% higher in replacement level than the last successful (homogeneous) batch was attempted. The maximum level of replacement was then defined as that of the last successful batch produced.

When using casein or a combination of casein and lactalbumin as the replacement protein source, since either may totally replace cheese protein from a mixing aspect, products were made at 20% replacement increments up to 80% replacement for further evaluation.

(4) The rheological tests used to define commercial acceptability were used to evaluate each experimental product. Results were used to determine whether or not a specific product was within the commercially acceptable range for each objective test.

RESULTS AND DISCUSSION

Product Manufacture

Product manufacturing results are given in Table 2. As shown, seventeen batches were attempted, with fourteen being successful. Several products, as indicated, had minor lumping problems caused by the incomplete dissolution of the replacement protein sources. The undissolved lumps were approximately 1 to 2 mm in diameter, and were not considered numerous enough to significantly affect product characteristics.

All products manufactured using Casein, or Casein and Lactalbumin, as the replacement protein source were successful and had processing times of approximately 15 min.

TABLE 2.
RESULTS OF EXPERIMENTAL PRODUCT PRODUCTION

Replacement Protein Source	Replacement %	Processing Time (min)	Homogeneous	Notes
Casein	20	15	Yes	1
Casein	40	15	Yes	1
Casein	60	15	Yes	
Casein	80	15	Yes	
Casein+Lactal.	20	15	Yes	1
Casein+Lactal.	40	15	Yes	1
Casein+Lactal.	60	15	Yes	
Casein+Lactal.	80	15	Yes	
Trad. Co-Ppt.	20	15	Yes	1
Trad. Co-Ppt.	40	21	Yes	1
Trad. Co-Ppt.	50	21	No	
Trad. Co-Ppt.	60	21	No	
Hidd. Co-Ppt.	20	15	Yes	
Hidd. Co-Ppt.	40	18	Yes	
Hidd. Co-Ppt.	50	21	Yes	
Hidd. Co-Ppt.	60	21	No	2
None (All Cheese)	NA	15	Yes	

Notes: 1 = Minor lumping

2 = Very nearly homogeneous

Casein + Lactal. = Casein and Lactalbumin in a 5:1 ratio

Trad. Co-Ppt. = Traditional coprecipitate

Hidd. Co-Ppt. = Hiddink's coprecipitate

Traditional coprecipitate was used to successfully replace up to 40% of natural cheese protein. An extended processing time was, however, necessary to achieve homogeneity at this level of replacement.

Hiddink's coprecipitate was used to successfully replace up to 50% of natural cheese protein. Extended processing times were again necessary at high replacement levels.

The higher replacement level possible using Hiddink's coprecipitate seems to indicate that there is some difference in the coprecipitates. It is not possible to conclude whether that difference is due to increased aqueous solubility or not. The main points that may be noted are that fairly high replacement levels are possible using coprecipitates, especially Hiddink's, and that the use of coprecipitates allows a simple, almost complete, method of harvesting milk proteins for use in process cheese.

Stress-Strain Behavior

Two types of stress-strain behavior were observed. In the first type of behavior a clear failure of the sample was evident. In these cases, true stress and true strain at the failure point were read and recorded. In the second type of behavior an approximate failure zone was discernible. When this was the case, true stress and true strain were read and recorded in the zone of failure.

Values recorded, as described above, for commercial and experimental products are given in Table 3. Comparison of commercial and experimental results indicates that none of the experimental product results fit completely within the range of results obtained for the commercial products. Generally, failure stresses for the experimental products were considerably higher than those for the commercial products. Failure strains for the experimental products, in contrast, were often within the range of results obtained for the commercial products. The experimental products were thus seen to be generally harder than the commercial products tested. This was true even for the standard experimental product, made with all natural cheese protein, indicating that processing parameters, and not protein replacement, are at least partially responsible.

Stress Relaxation Behavior

Values used to characterize stress relaxation behavior for both commercial and experimental products are given in Table 3. At a true (Hencky's) strain of approximately 0.25, it can be seen that F_8/F_0 values for products made using casein as the replacement protein source, or containing all cheese are considerably higher than those obtained for the commercial products. All except one of the products containing whey proteins, however, fit within the range of commercial results. This is reasonably explained by considering casein and cheese protein as ingredients that create structure in the product. The whey proteins, however, not only decrease the amount of casein and natural cheese protein in the product, but also, due to their relatively low solubility, act as largely inert materials, disrupting the structure of the product.

It is also interesting to note that the standard experimental product had the highest F_8/F_0 value recorded for all products. This indicates, as was the case for stress-strain testing, that processing parameters and not protein replacement are responsible for the relatively low levels of relaxation observed for many of the experimental products.

Compressive Creep Behavior

Values used to characterize creep behavior obtained for commercial and experimental products are given in Table 3. An example of how well the mathematical model fit the experimental data is shown in Fig. 5. (The example shown

TABLE 3.
RESULTS OF RHEOLOGICAL TESTING OF CHEESE PRODUCTS

Product	Stress-Strain ¹ σ_f (kg/cm ²)			Stress Relaxation ² F_0/F_8 , n , ϵ_{Hencky} = .25			Compressive Creep ³ $K \times 10^5$ (1/Pa)			Melting ⁴ Index			
	μ	σ	ϵ_f	μ	σ	n	μ	σ	ϵ	Melting	Index		
Commercial:													
Land-O-Lakes	0.51	0.02	0.79	0.01	0.099	0.005	8.19	0.62	0.38	0.03	10.9	5.2	0.2
Naturally Slender	.46	.01	.57	.03	.146	.006	2.92	.30	.22	.02	1.30	-	-
New Yorker	.35	.02	.32	.02	.098	.003	3.95	.45	.24	.01	2.02	-	-
Stop & Shop	.54	.01	.55	.01	.145	.001	3.57	.26	.21	.01	1.49	-	-
Uni-Chief	.31-.47	-	.33-.70	-	.065	.004	5.02	0.32	.44	.01	10.0	7.2	.6
Waldbaums	.47-.53	-	.79-.93	-	.069	.008	13.60	2.83	.46	.03	31.1	4.8	.2
Experimental:													
Casein 20%	0.89	0.08	0.67	0.06	0.176	0.002	2.32	0.06	0.27	0.01	1.46	6.2	0.2
Casein 40%	0.94	.12	.69	.03	.172	.010	1.99	.49	.29	.02	1.43	6.1	.2
Casein 60%	1.00	.07	.74	.07	.183	.005	1.77	.58	.25	.03	0.97	5.2	.3
Casein 80%	1.11	.10	.76	.05	.184	.002	1.15	.14	.25	.01	0.63	5.0	.3
Casein + Lact. 20%	0.64	.03	1.11	0	.128	.007	2.85	.25	.39	.01	4.05	5.4	.1
Casein + Lact. 40%	.80	.16	0.87	.08	.120	.007	2.66	.37	.33	.01	2.51	5.3	.4
Casein + Lact. 60%	.81	.03	0.84	.03	.141	.004	1.60	.38	.32	.03	1.41	4.7	.3
Casein + Lact. 80%	.82	.03	1.01	.03	.123	.001	1.49	.10	.34	.01	1.51	5.2	.2
Trad. Co-Ppt. 20%	.65	.07	0.86	.12	.129	.012	2.37	.10	.40	.02	3.60	5.0	.2
Trad. Co-Ppt. 40%	.87	.09	.65	.02	.108	.001	1.73	.29	.37	.01	2.14	3.5	.2
Hidd. Co-Ppt. 20%	.61	.06	.77	.06	.167	.010	2.94	.25	.33	.01	2.78	6.2	.4
Hidd. Co-Ppt. 40%	.70	.05	.76	.02	.134	.001	2.18	.23	.33	.01	2.06	4.1	.1
Hidd. Co-Ppt. 50%	.69	.02	.90	.06	.118	.003	1.89	.24	.38	.01	2.51	3.2	.3
Std. All Cheese	.79	.02	.73	.04	.195	.002	3.36	.13	.25	.01	1.84	6.8	.2

Notes: ¹ σ_f = true stress at failure; ϵ_f = Hencky's strain at failure; where a range of values are recorded, an approximate zone of failure was observed; cross-head speed = 10 cm/min.
² F_0 = initial force; F_8 = force after 8 minutes relaxation; ϵ_{Hencky} = Hencky's strain; cross-head speed = 10 cm/min.
³ J = compliance; K and n are constants of equation 4 to which compliance data was fit; initial stress = 10.4 kPa.
⁴where — is recorded no melting was observed; Schreiber type melting test 232°C for 5 minutes.

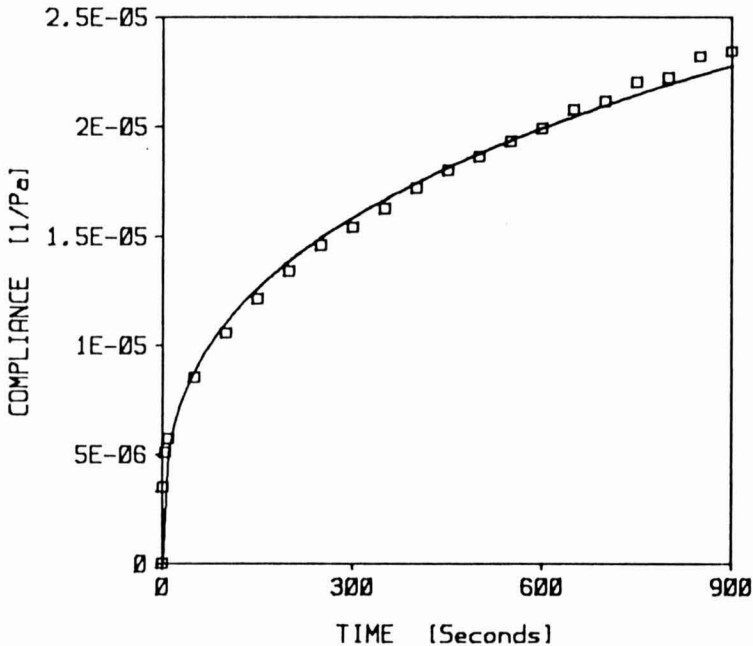


FIG. 5. CREEP CURVE FIT EXAMPLE, \square INCOMPLETE SET OF EXPERIMENTAL POINTS, _____ CURVE GENERATED FROM EQ. 4 FIT TO DATA

in somewhat representative of all the curves fit, which, due to the large number, are not presented.) It can be seen that the creep results obtained for both commercial and experimental products cover a wide range of behavior. Furthermore, the range of values of n obtained for experimental products is nearly as broad as that obtained for the commercial products, and that all the experimental n values fit within the commercial range. The K values obtained for experimental products, however, are in nearly all cases below the commercially defined range. This indicates that the instantaneous compliance of the experimental products was lower than that of the commercial products. To determine whether or not behavior of the experimental products fit within the range of behavior exhibited by the commercial products, compliance values at an arbitrary time (900 s) were calculated from the equations obtained from curve fitting (results shown in Table 3) and compared. Only two experimental products, Casein 60% and Casein 80%, were found to fall outside the range of compliance defined by the commercial products. Each of these products showed compliance below the commercial range, indicating a greater rigidity than exhibited by the commercial products.

While most of the experimental products were seen to exhibit compliance behavior within the range defined by the commercial products, it must be noted

that they all showed behavior similar to the more rigid commercial products. As was noted for stress-strain and stress relaxation behavior, this is probably due to the selected processing parameters and not to protein replacement.

Melting Behavior

Melting indices for both commercial and experimental products are shown in Table 3. As can be seen, commercial products showed a wide range of melting responses. The most interesting result was that several products showed virtually no melting as evaluated by this test. All the experimental products, in contrast, showed some melting, and all experimental responses therefore fit within the commercially defined range. It should also be noted that use of any of the replacement protein sources seems to decrease meltability relative to the standard experimental product, especially when the coprecipitates were used.

CONCLUSIONS

From the results discussed previously, the following specific conclusions may be drawn:

(1) All of the protein replacement sources were used successfully to some extent in the manufacture of process cheese products. Casein, or a 5:1 ratio of casein and lactalbumin were used to replace up to 80% of natural cheese protein. Traditional coprecipitate was used to replace up to 40%, and Hiddink's coprecipitate up to 50%, of natural cheese protein.

(2) The use of casein and lactalbumin, or one of the coprecipitates allows the incorporation of whey proteins into process cheese products. This could increase the yield of process cheese product per pound of milk by up to approximately 15% (with 80% protein replacement), as compared to the use of natural cheese.

(3) Because of the high level of protein replacement possible, the use of casein and lactalbumin would seem to hold the greatest commercial promise for incorporating whey proteins into process cheese products. However, the use of Hiddink's coprecipitate would also merit consideration for applications where lower levels of protein replacement are required.

(4) None of the experimental products, when tested rheologically, fit totally within the envelope of "commercial acceptability" defined by evaluation of selected commercial products. However, the fact that this was true even for the standard product, made with all natural cheese protein, indicates that the replacement protein sources may not be the cause of this result. Alteration of the processing parameters would possibly allow the manufacture of products, incorporating one of the replacement protein sources, that more closely mirror the rheological behavior of the selected commercial products.

DISCLAIMER

Selection of specific commercial process cheese products for comparison testing was arbitrary, and does not imply any judgement as to quality, or preference over similar products, by the authors or sponsoring agencies.

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A PRELIMINARY STUDY OF HEATING CHARACTERISTICS AND QUALITY ATTRIBUTES OF PRODUCT PACKAGED IN THE RETORT POUCH COMPARED WITH THE CONVENTIONAL CAN

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ABSTRACT

The advantages of using a retort pouch to replace the conventional metal can were investigated. Required processing times for equivalent volume packages subjected to either a still or agitated cook were compared for three products, and the resulting product quality was determined by evaluating Vitamin C content, color, and drained weight. Pureed pumpkin represented conduction heating products; peas in brine and pineapple in juice represented products using conduction/convection heating. It was found that pouches yield greatly reduced process times for conduction heating products. For particulates in a liquid medium, the agitated cooking of cans require less processing time than the thinner still-cooked pouches.

INTRODUCTION

With the costs of energy, wages and distribution on the rise retort pouches may well be one of the coming forms of packaging, replacing the conventional tin can for many foods in U.S. supermarkets. Most of the research work concerning the retort pouch occurred in the 1970's and early 1980's. Presently, the focus of the food processing industry has turned to the area of aseptic processing

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dealing primarily with plastic and paperboard containers. But if, in the future, pouch filling speeds approach those of the conventional can, then we will no doubt see the retort pouch once again in the spotlight of this country's food industry.

The retort pouch has many advantages over canned and frozen food packages, for both the consumer and the food manufacturer. These advantages include the pouch profile (Mermelstein 1978), storage and preparation efficiency (Mermelstein 1976), savings in shipment costs, container cost, and improved flavor (Badenhop and Milleville 1980), and a savings of energy (Steffe *et al.* 1980).

While the number of published studies pertaining to the retort pouch are limited, there were nonetheless, several very good papers concerning quality and processing comparisons of foods packaged in the retort pouch and one of the more conventional, rigid containers. Tung *et al.* (1977) found that flexible packaging technology for shelf stable foods appeared to permit production of very high quality vegetable products. Gomez *et al.* (1980) found that pouches may be rapidly heated and cooled without the hazards of thermal shock and product packed in aluminum laminate pouches or glass jars stored better than product in transparent pouches. Tung *et al.* (1975) found that after 6 months of storage, pouch packs of cream style corn were significantly superior in aesthetic appeal to the canned product and there were no significant differences between the pouches and cans in overall acceptability or storage stability. A study by Green (1979) indicated that the retention of nutrients in heat sterilized foods may be significantly improved during thermal processing and maintained in storage with the use of the retort pouch. Lyon and Klose (1981) found the retort pouch process offered an advantage over canning for fowl meat. Chen and George (1981) compared the sensory quality of green beans processed in retort pouches and metal cans and found the retorted pouched green beans were rated more desirable than the canned products in flavor, texture and overall acceptance.

The United States Army Natick Laboratory (Burke and Schulz 1972) showed that flexible pouches were capable of withstanding drop and vibration hazards at least, as well as metal cans; they also found that the fiberboard shipping containers suffered considerably more damage from cans than from flexible packages. Williams *et al.* (1981) determined that a retort pouch system yielded an overall minimum cost when compared to an existing canning line or a new canning line.

During the past few years a number of articles favoring the retort pouch have appeared in food science trade journals. These articles list several advantages that the pouch has over other packaging forms, primarily the metal can. There has been, however, limited published data reported in the literature to back-up some of these "claimed" advantages of the retort pouch. Therefore, the *purpose of this study* (Snyder 1985) was to verify, or refute, two commonly stated advantages attributed to the retort pouch: (1) reduced processing times and (2)

improved product quality compared to similar product packaged and processed in the conventional metal can. Currently accepted practice, e.g., processing temperatures and packaging techniques, was used to obtain a meaningful comparison between the two packaging types rather than holding constant all the variables that were not being compared. Three products were used in the study: pumpkin puree, peas packed in brine, and pineapple chunks packed in juice. The metal cans and retort pouches used in this study held the same volume of solid material; the juice/brine content varied.

MATERIALS AND METHODS

An important decision when carrying out a heat penetration test is where to locate the thermocouple. The temperature in a container of food is not uniform during a sterilization process. The point of measurement should be in the slowest heating zone of the product to insure that the process determined with the results of that data will be adequate for all points in the container (Pflug 1975).

When collecting heating data for the pureed pumpkin the thermocouple was located in the geometric center of the pouch or can. Also, several thermocouple probes were placed throughout the retort in order to verify that uniform heat distribution had been achieved. For the cans containing peas in brine or pineapple in juice, which were to receive an agitated cook, the thermocouple tip was placed in the container's geometric center along the axis of the can to minimize probe interference.

For canned peas packed in brine receiving a still (nonagitated) process heating tests were run to determine the location of the slowest heating point in the container. This is simply done by placing thermocouples at various locations along the can's axis until the cold zone was located.

FMC Central Engineering Laboratories, Santa Clara, California had done this type of testing with a product system identical to that used in this research (Adams and Hardt 1980). With a can size 303 × 406 and a fill weight of 11 oz. of peas covered with brine, the cold zone was found to be in the area of 1¼ in. from the container bottom.

Hypothetically, particulates packed in a retort pouch should approach a conduction-type heating process. This holds for several reasons: (1) reduction of brine (or juice) causes the food particulates to form a near solidly packed product which would more likely heat by conduction, (2) since the pouched product is to receive a still process, no forced convection will be experienced and (3) due to the geometry of the pouch and its ¾ in. height, there is little likelihood that free convection currents, normally found in a can (containing liquid or liquid and particulates) will occur in a pouch.

If the particles packed in liquid did indeed exhibit conduction-type heating, then the cold spot would be located in the geometric center of the container, as

it was for the homogeneous pumpkin puree. A test was run in order to determine the slowest heating point in the $6\frac{1}{2}$ in. \times $8\frac{1}{2}$ in. \times $\frac{3}{4}$ in. pouch filled with particulates (peas) and brine. The results were in close agreement with the above reasoning. Therefore for convenience, this was where the thermocouple probe was located throughout the pouch test runs.

A retail size 303 \times 406 electrolytic tinplate can with enameled body and ends was used in this study. A needle thermocouple (type CNS by O. F. Ecklund Inc., Cape Coral, Florida) was placed through a compatible Ecklund receptacle located in the bottom, center of the can.

For a given run, the first and last containers filled were equipped with thermocouples to represent the coolest and warmest containers. Two to three other filled containers were also equipped with thermocouples per run. These containers were picked at random during the filling. This procedure was followed for both the pouch and can runs, for all three products.

To gather the pouch heating data a needle type thermocouple penetrating into the pouch via a specially designed receptacle was used. The needle (Ecklund—type CNL) was placed into the pouch via a thermocouple receptacle (Ecklund—brass type C-5.1) designed for a plastic (flexible) pouch. An experiment was designed to determine if the brass receptacle/needle combination effected the heating data collected. The results indicated that a minimal temperature difference existed between the two methods, hence, the brass receptacle, based on the convenience it afforded, was used throughout this study.

A $6\frac{1}{2}$ in. by $8\frac{1}{2}$ in. preformed Flex Can R pouch with rounded corners was used throughout this study. As the pouches were processed in the horizontal position, the needle was located at the horizontal midpoint. Along the height of the pouch heating variations would most likely occur during convection-type heating. A plug device was used to vary the location of the needle tip along the vertical axis of the pouch by drilling holes every $\frac{1}{8}$ in.

For those canned products receiving a still cook, no headspace was provided. When providing an agitated process, a measured headspace was provided to insure mixing of the contents during the process. The industry standard of $\frac{1}{4}$ in. was used when headspace was required.

When pineapple was the experimental product, a pineapple chunk was placed on the thermocouple needle so that the needle tip would be approximately in the fruit center. This procedure was carried out in both pouches and cans containing the temperature sensing probes.

Two different methods for removal of residual gas from the pouches were utilized during this research, air removal via a vacuum pump and by water immersion. Both methods gave satisfactory results.

The products used for this research study were obtained in either a fresh (pumpkin or pineapple) or a frozen (pea) form.

The pureed pumpkin was heated in a steam-jacketed kettle to 180°–190°F

prior to filling the containers with 500g. It was essential, both from the standpoint of cutting down processing time and for prevention of occluded air in the product, to fill the product in the container at temperatures above 180°F (Lopez 1981).

Frozen (No. 3) peas were put into a wire mesh basket and immersed in a simmering water bath (190–200°F) for 2 min. Although the peas were blanched prior to freezing, this additional short blanch was performed in order to raise the product's initial temperature to approximately 140–150°F. It also helped to remove any gases remaining within the particulates.

Each container was filled with 312 g (11 oz.) of drained, blanched peas. The still cooked cans contained 180 g of brine and the cans to be processed by agitation held 160 g of brine. Only 65 g of brine were added to the pouched peas. The brine itself was a solution of salt (2%) and water at 180°F.

Fresh pineapple was cut by hand into chunks approximately ½ in. thick, ¾ in. wide, and ¾ in. long. Fresh pineapple is high in occluded gas, therefore a vacuum was drawn on the pineapple chunks prior to filling (Hagland 1981). Each container contained 350 g of drained pineapple. All of the cans had 135 g of juice to maintain the measured headspace. The pouches were filled with only 100 g of liquid.

All products were processed at the pilot facility of FMC Central Engineering Laboratories, located in Santa Clara, California. The cans, whether receiving an agitated or a still cook, were processed in an FMC Steritort Pressure Sterilizer. Thermocouples in the cans were connected to a potentiometer to provide a strip chart record of the complete product heating and cooling cycle. Thermocouples were also placed at strategic locations throughout the retort in order to monitor the medium temperature.

The canned products were processed at either 220°F, 250°F or 260°F. All cans sterilized in the Steritort were pressure cooled with a city water medium and an overriding air pressure.

All pouches were processed in a FMC Model 500 Laboratory Retort at 220°F or 250°F which is the temperature limit of the pouch material. This sterilizer is specifically for the thermal processing of products without agitation in containers of unconventional shape or material combination (Anon. 1979).

The sterilization trays used for this study were designed for pouches with a maximum thickness of ¾ in., with 10 containers per tray. Within the retort, temperature sensing thermocouples were placed at a number of locations during a process run in addition to those in the pouches. "Dummy pouches" filled with a 7% bentonite solution were used to fill the retort car trays in order to simulate a full retort load.

The concentration of reduced ascorbic acid in the fruit and vegetable samples was determined by using a modification (Swartz 1978) of spectrophotometric methods developed by Pearson (1970) and Dietrich (1977).

The processed samples were evaluated by color measurements using a Hunter

Lab Color/Difference Meter D25D2. The Hunter meter permits measurements of lightness, redness and yellowness.

A wire mesh screen, No. 6 Tyler screen (0.132 in. openings), was used to drain the particulates. The product was allowed to drain for 1½ min.

All the details of the experimental procedure, data organization and analysis, and related arguments and discussion for this project cannot be included here because of length constraints. This paper contains the important highlights. The original report (Snyder 1985) should be consulted for all the details.

RESULTS AND DISCUSSION

Pumpkin Puree

Process lethality (F_0) was calculated by the General Method using a center temperature at two minute intervals. A computer program was used to calculate the lethal rates and the total interval lethalties based on a Z value of 18°F and a reference temperature of 250°F. Two test runs were made for each container type. The processing times used during the pumpkin can runs, were 54.5 min and 53.2 min at 250°. Process times for the pouch runs were 19.8 min and 17.5 min at the same temperature. Typical heating curves for pureed pumpkin processed in the pouch and can are presented in Fig. 1. The large processing time advantage provided by the pouch can easily be seen.

Quality assessments of the pumpkin puree packed in the can and retort pouch were made after approximately 3 months of refrigerated (40°F) storage. Multiple color readings were taken from the two pouches and the two can runs.

The pouched pumpkin puree was found to be considerably lighter ($P < 0.01$) than the canned product as indicated by a higher Hunter lightness value. This result was not surprising considering that the can received a process that was nearly 3 times as long as that received by the pouched pumpkin.

Peas Packed in Brine

For the peas packed in brine, process lethality (F_0) was calculated by the General Method using processing temperature data collected from each test container at 2 min intervals for the still processes and at 1 min intervals for the agitated cooks. As with the pureed pumpkin, a computer program was used to calculate the lethal rates and total interval lethalties based on a Z-value of 18°F and a reference temperature of 250°F. Two still cook runs for cans, two agitated cook runs for cans, and three runs for pouches were made.

Figure 2 illustrates typical heating curves for pouched and canned peas receiving still cooks. Figure 3 compares the pouch still process with the can processed in the rotary cooker. Although the F_0 values calculated for the agitated cook are higher than the values obtained with the still processes, the processing

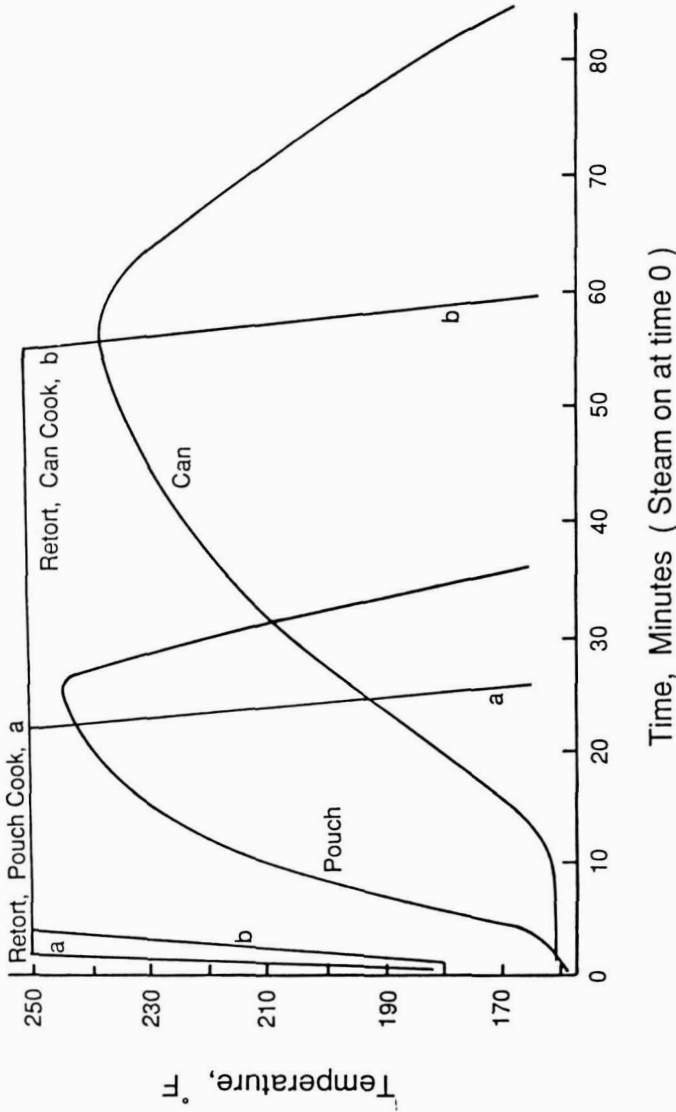


FIG. 1. TYPICAL HEATING CURVES OF 500 G PURED PUMPKIN STILL PROCESSED IN A FLEXIBLE POUCH OR METAL CAN AT 250°F FOR 19.8 MIN AND 53.2 MIN, RESPECTIVELY; F_0 VALUES AT 3.59 MIN AND 3.62 MIN, RESPECTIVELY

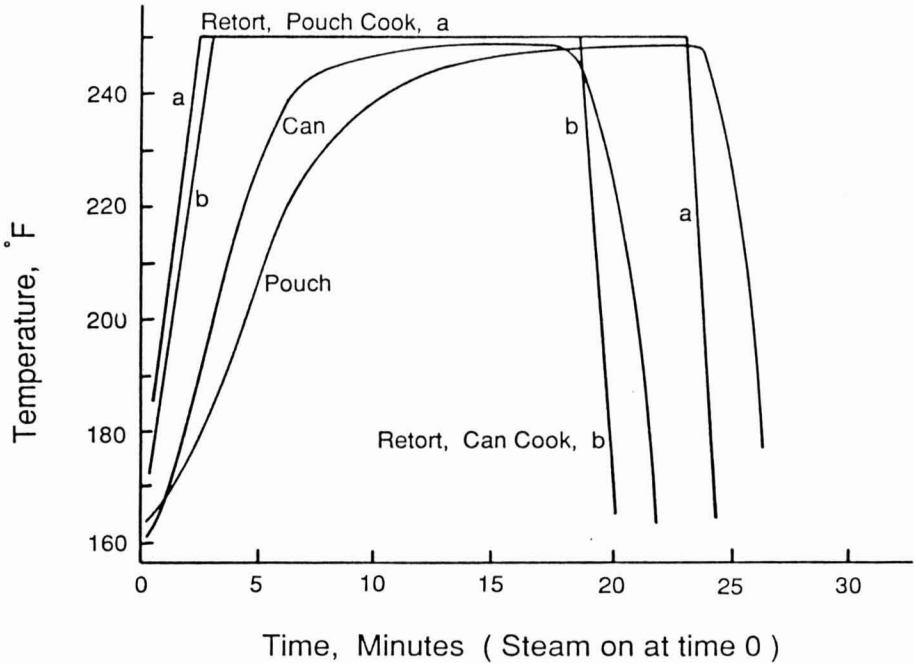


FIG. 2. TYPICAL HEATING CURVES OF 312 G OF PEAS PACKED IN BRINE STILL PROCESSED IN A FLEXIBLE POUCH OR METAL CAN AT 250°F FOR 22.7 MIN AND 16.7 MIN RESPECTIVELY; F_0 VALUES OF 11.00 MIN AND 11.01 MIN, RESPECTIVELY

time advantage achieved when processing particulates by agitation can easily be seen.

Taking the average processing time of the still processed pouches give a 33% processing time increase for the retort pouch over the still cooked cans. Comparing the still processing times for pouch and the can with the average processing time for the agitated-cooked cans yielded process reductions of 66.5% and 55.4%, respectively.

An agitated process temperature of 260°F was used so as to be in conformance with industrial practices. Additionally, the F values (18.0, 250.0) calculated for the agitated cooks were considerably higher than those calculated for the still cooks, thus, the processing time reduction of agitated over still processing would be further increased had similar F_0 values been obtained.

Color measurements, ascorbic acid analysis and drained weight determinations were taken after approximately 3 months of refrigerated (40°F) storage.

There was no significant ($P < 0.01$) difference in product lightness between the canned peas receiving an agitated or still process. There were differences

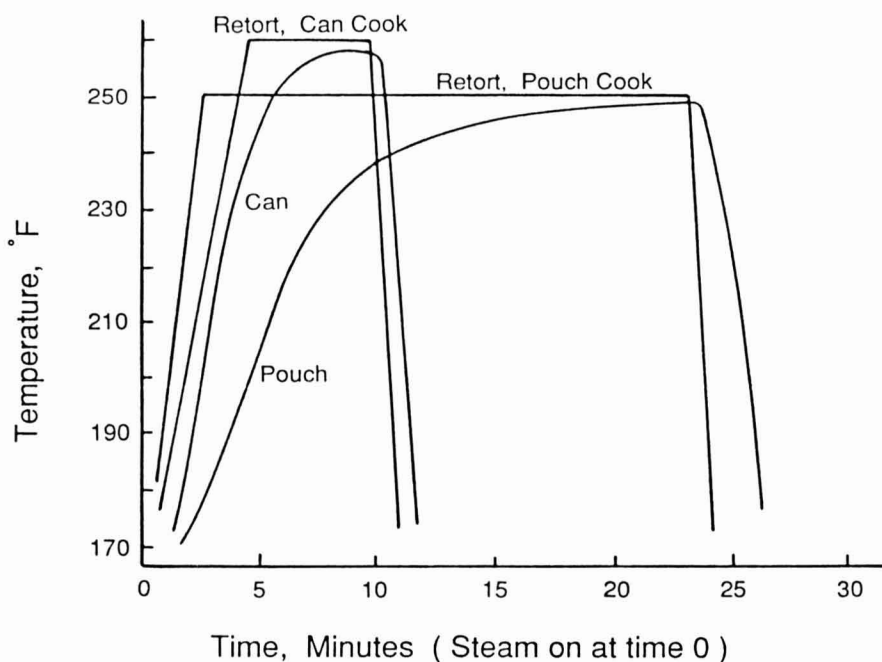


FIG. 3. TYPICAL HEATING CURVES OF 312 G PEAS PACKED IN BRINE STILL PROCESSED IN A FLEXIBLE POUCH AT 250°F FOR 22.7 MIN OR RECEIVING AN AGITATED PROCESS IN A METAL CAN AT 260°F FOR 7.2 MIN; F_0 VALUES 11.0 MIN AND 13.51 MIN, RESPECTIVELY

($P < 0.01$) in product lightness of approximately the same magnitude between the pouched peas and both types of canned peas.

While the lightness of the pouched pumpkin puree over the canned product was undoubtedly linked to the vast difference in processing times, the same reasoning cannot be used here to explain why the pouched peas exhibited a higher L-value. Perhaps the fact that there was less brine in the pouched products can explain this difference. The increased liquid in the canned product may have caused more leaching of pigments (chlorophylls) from the pea skins. Numerous factors influence chlorophyll loss in vegetables, including atmospheric oxygen and storage temperature (Peterson and Johnson 1978). For the agitated cans the color loss may, therefore, be explained by oxygen present in the headspace gases. Also, since there were no differences in product lightness for the two different can processes, the packaging material may have effected the product coloring, independent of the amount of heat received.

All Vitamin C readings were made on drained pea samples. The brine was not analyzed for ascorbic acid content. A lower amount of ascorbic acid was

found in the pouches. This may be attributed to the longer processing time for the pouch.

A significantly ($P < 0.01$) higher amount of brine was absorbed by the peas processed in cans than those processed in pouches. This can possibly be explained by the fact that there was more free liquid available for the canned peas to absorb as compared to that available to the pouched peas.

Pineapple Packed in Juice

The continuous rotating cooker is used almost exclusively for processing of pineapple. The canned pineapple was pressure sterilized with a 220°F steam processing medium. The pouches were processed using two different medium temperatures, 220°F and 250°F pressurized water.

The process lethality was calculated by the General Method using one minute temperature intervals. The computer program used to calculate the lethal rates and the total interval lethality used a reference temperature of 212°F and a Z-value of 18°F.

It was once again evident that an agitated can process offers considerable advantage over a still retort pouch cook in terms of processing time. Figure 4 presents the heating curves for pineapple packed in juice for the retort pouch and metal can processed at 220°F. The results of processing the pouched pineapple chunks with 250°F media indicate that the advantage of the can over the pouch appears to have diminished. Figure 5 illustrates the heating curves for pineapple chunks in juice processed in the pouch at 220°F and 250°F.

Color measurements, ascorbic acid values and drained weight determinations were made on the canned and pouched samples after approximately 2 months of 40°F storage.

It was found that the pouched products receiving a 220°F process were significantly ($P < 0.01$) lighter than the pineapple chunks processed in the cans with a similar retort temperature or in the pouches at 250°F. A comparison of these latter two processes yielded no significant ($P > 0.05$) difference in product lightness.

Averaging the calculated sterility values for the three run types resulted in F-values of 2.53, 2.66 and 3.46, for the pouch at 220°F, the can at 220°F and the pouch at 250°F, respectively. It is, therefore, surprising to find that pouched pineapple measured lighter than the can at the 220°F processing temperature, since the pouched received a process which was twice the time of that received by the canned product. The inferior product color of the canned pineapple was most likely due to the presence of oxygen in the headspace gas promoting an enzymatic browning reaction (Johnson and Peterson 1974).

A comparison of the Vitamin C content from pouched pineapple processed at retort temperatures of 220°F and 250°F indicated that no significant difference

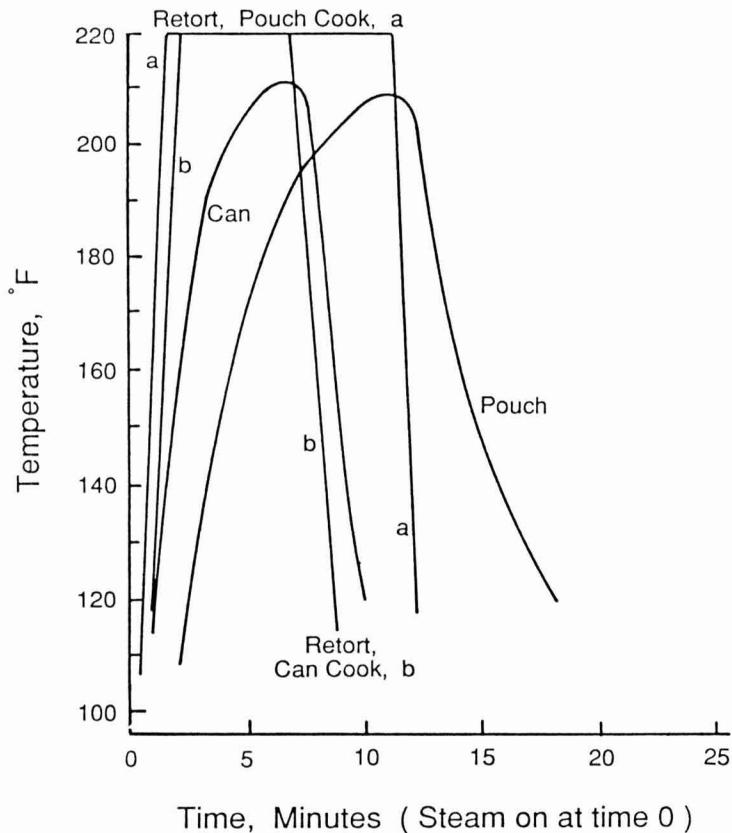


FIG. 4. TYPICAL HEATING CURVES OF 350 G PINEAPPLE CHUNKS IN JUICE RECEIVING A STILL PROCESS IN A FLEXIBLE POUCH OR AN AGITATED PROCESS IN A METAL CAN, AT 220°F FOR 10.4 MIN AND 5.1 MIN, RESPECTIVELY; F VALUES (18.0, 212.0) OF 2.59 MIN AND 2.51 MIN, RESPECTIVELY

($P > 0.05$) existed, despite the two-fold increase in processing time. Apparently, the heat sensitive Vitamin C underwent similar destruction, whether at the 220°F process for 10.5 min or the 250°F process for approximately 5 min.

The results obtained may be related to the type of process, the slight difference in the quantity of liquid medium or, more likely, the presence or absence of headspace gases. Comparing equal processing times of a still process with an agitated process, it must be realized that with the latter processing method each product particle sees more or less the same amount of heat during the process due to forced innermixing. With the still process, those pieces on the outer package edge were exposed to considerably more heat than those sheltered pieces in the container center. For sampling purposes, therefore, it was important to

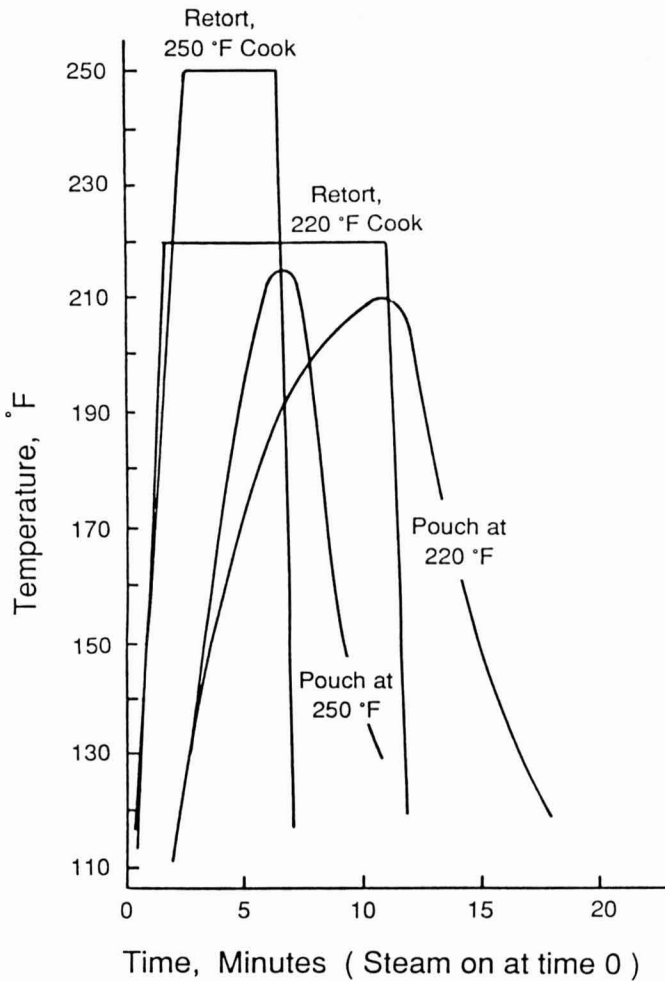


FIG. 5. TYPICAL HEATING CURVES FOR 350 G PINEAPPLE CHUNKS IN JUICE RECEIVING A STILL PROCESS IN A FLEXIBLE POUCH AT 220°F AND 250°F, FOR 10.4 MIN AND 4.7 MIN RESPECTIVELY; F VALUES (18.0, 212.0) OF 2.59 MIN AND 2.76 MIN, RESPECTIVELY

obtain a large sample of the still processed product pieces in order to be assured of obtaining a representative sample in terms of heat exposure. The greater amount of juice and the mechanical agitation of the rotary process may have lead to the leaching of Vitamin C out of the canned chunks into the surrounding fluid.

The largest contributing factor to the Vitamin C destruction of the canned pineapple, however, was undoubtedly the oxygen contained in the atmospheric

headspace. Ascorbic acid is very susceptible to destruction by oxidation in the presence of moisture, especially if promoted by heat, alkali, or dissolved copper and iron (Johnson and Peterson 1974).

The lowest drained weights were obtained with the pouched pineapple processed in the 250°F retort. The pineapple chunks processed at 220°F had similar values, with the can drained weights slightly lower.

At the higher processing temperature, the greater decrease in the drained weight may be due to increased softening of the fruit tissues, which in turn leads to a greater sloughing of particles off of the pineapple chunks.

Comparing the drained weights of the product receiving the 220°F process, a significant ($P < 0.05$) difference was realized, with the pouched pineapple chunks showing a greater drained weight loss. This can probably be attributed to the longer processing time of the pouch.

It appears that the process temperature has the greatest negative effect on drained weight. The processing time also seems to effect these values, though to a lesser extent.

CONCLUSIONS

The most dramatic heat processing difference observed during this study occurred with the processing of the pureed pumpkin. The pouch gave a 67.5% reduction in processing time over the can.

For particulate products in a thin liquid media, an agitated can process appears to provide a considerable advantage in terms of processing times over comparable still processes, whether can or pouch. Until a method is developed for the agitation of pouches during processing it is unlikely that the pouch will offer any processing advantages over the agitated can. Even if this type of retort is designed, the agitated can will probably possess many processing advantages over the pouch due to a combination of the pouch dimensions and the incorporation of less brine. It is likely that when agitated, these factors will stifle or reduce the development of forced convection currents.

There were no clear cut quality advantages to packing particulate containing products, similar to those studied, into the retort pouch. It is quite possible, however, that particulate containing products in a heavy sauce or syrup may benefit by the thin profile the pouch offers due to the approaching of conduction type heating characteristics. Until additional research work is done it appears that the greatest successes with the pouch will be made for the thermal processing of homogeneous, conduction heating products.

For the pineapple and pea products, the validity of the ascorbic acid content for those cans receiving an agitated process was questionable due to the atmospheric gases incorporated into the container headspace at closing. There is little doubt that this played a significant role in indicating ascorbic acid levels in favor

of the pouch, despite longer processing times. Future work would be beneficial in this area.

For the peas in brine still processed in the pouch and can, a safe ascorbic acid comparison can be made as there was no headspace in either of the containers. The canned drained peas were found to have significantly more Vitamin C than the pouch. This follows, as the pouches were processed 33% longer than the still processed cans.

Another Vitamin C comparison can be made between the pouched pineapple processed at 220°F and 250°F. Here, no significant difference was found despite the two-fold increase in processing time for those pouches processed at 220°F. These results indicated that equivalent Vitamin C destruction occurred with processes of 220°F for 10.5 min and 250°F for 5 min. Again, this is another area which could be further explored.

Unfortunately, the inclusion of oxygen into the headspace of those cans receiving an agitated cook also affected product color, most notably the pineapple. The pouched pineapple chunks, whether processed at 220°F or 250°F, were found to be lighter and yellower in color than the canned samples (all received an agitated process). This is surprising since the pouches processed at 220°F had a processing time twice that of the cans, and the other set of pouches, while possessing a similar processing time, were processed at a much higher retort temperature. The color comparison which is meaningful was that made between the two sets of pouch data. Here, it was found that the lower processing temperature had a less detrimental affect on product lightness than the higher retort temperature of 250°F.

A valid comparison can safely be made with the peas still processed in the can and pouch. Here, the pouched peas were found to be lighter than the canned. The higher ratio of brine in the canned peas may have caused leaching of selected color pigments from the pea skin.

The pouched pureed pumpkin was found to possess a color which was significantly lighter and more yellow than the canned product. The results of the color analysis may indicate that for slow heating conduction products the pouch would offer advantages over the can if the product's color was susceptible to color degradation due to extended heat exposure.

It was found that the canned peas had a greater increase in drained weight than the pouched product. This was thought to be attributed to the greater amount of brine available for absorption into the canned peas. With the pouched pineapple, it was found the higher the processing temperature, the greater the drained weight decrease. This might be related to the softening of the fruit at higher temperatures. The agitated canned pineapple had the lowest drained weight loss. The reason that the nonagitated pouch had a greater drained weight decrease than the agitated canned product was thought to be due to the difference in their

processing times, with the pouch receiving a heat process approximately twice as long as the can.

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