ISSN 0022–1163

Volume 18 Number 5 October 1983

IFST

Journal of Food Technology

Published for the Institute of Food Science and Technology (U.K.) by Blackwell Scientific Publications
Oxford London Edinburgh Boston Melbourne

JOURNAL OF FOOD TECHNOLOGY

Institute of Food Science and Technology (U.K.)

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The Journal of Food Technolgoy is published bimonthly, six issues form one volume. The annual subscription is £72.00 (U.K.), £86.00 (Overseas), $195.00 (N. America, including cost of airfreight). Copies of the journal are dispatched by airfreight to New York for forwarding by surface mail throughout the Western Hemisphere, and by airfreight to Singapore for forwarding by surface mail to subscribers in India, Australasia and the Far East. Back volumes are still available. This journal is covered by *Current Contents*, *ASCA* and *Science Citation Index*.

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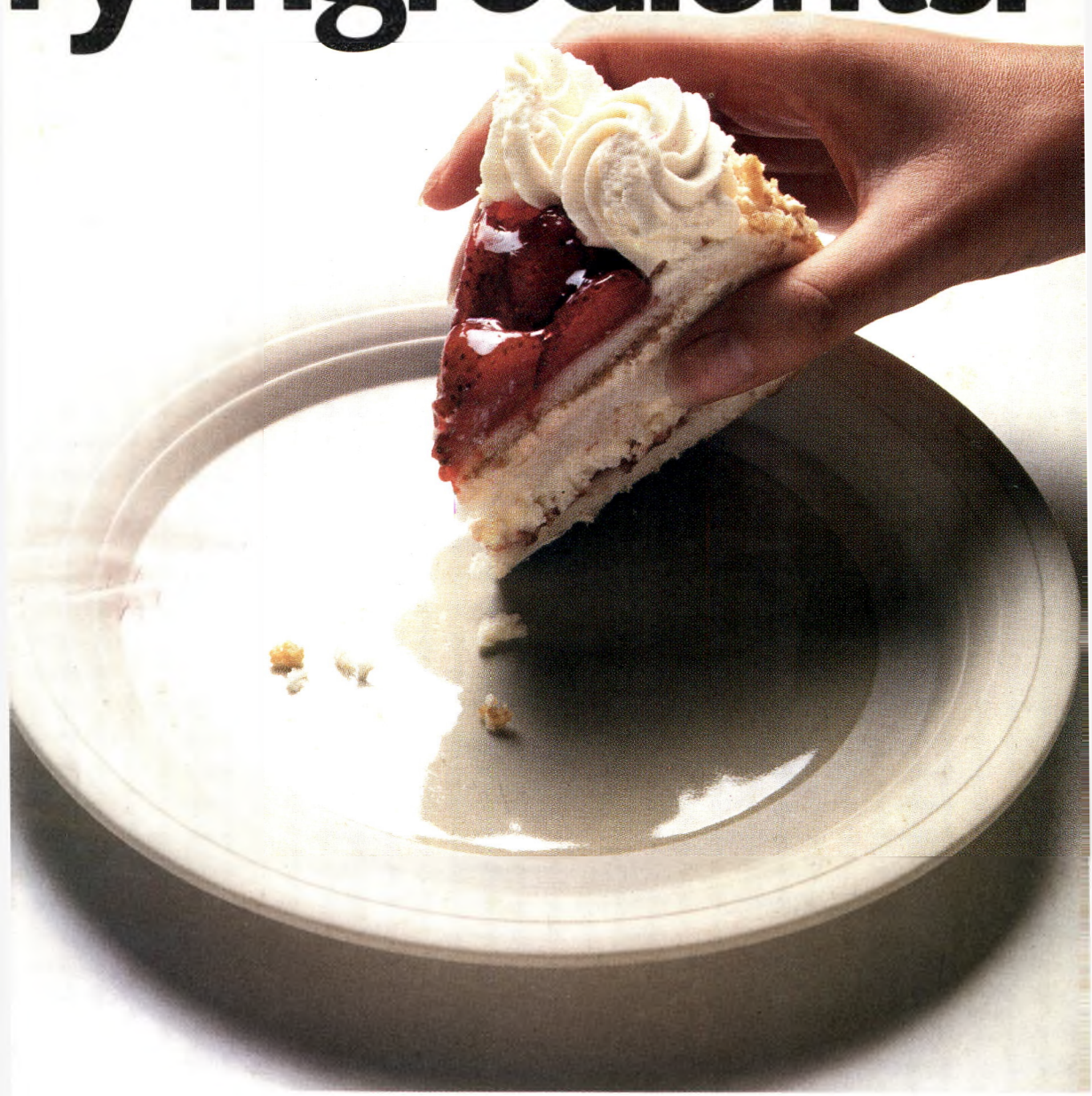

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Modelling of multiple-effect falling-film evaporators

SANDRO ANGELETTI and MAURO MORESI

Summary

The classic mathematical model of multi-effect evaporators operating in forward flow was combined with an accurate estimation of the overall heat transfer coefficient (HTC) in falling-film evaporators. For this correlation developed by Narayanamurthy & Sarma (1977) has been used. It was possible also to estimate the order of magnitude of the fouling factor (R_d). Finally, the design of an orange-juice double-effect falling-film evaporator (DEFFE) system was carried out according to two different strategies.

Introduction

Falling-film evaporators (FFE) have recently found wide application in the food industry, as they can handle heat sensitive materials (e.g. fruit and vegetable juices, milk, skim milk and whey, beet and cane sugar juices, coffee and tea extracts, etc.,) because of their particular characteristics, such as residence times no longer than a few seconds; high values of the heat transfer coefficients even at low boiling temperatures; minimum loss of the available temperature difference (hydrostatic head, friction and acceleration effects are negligible when the liquid level in the bottom of the evaporator and vapour velocity in the cores of the tubes are kept low).

However, FFEs are generally unsuitable for salting, scaling, and fouling liquors, as boiling, occurring in the tubes, may facilitate fouling, thus making heat transfer more difficult. Similarly, feeds with viscosity greater than 200 mPas cannot be handled, as such viscous solutions do not spread over the tubes.

The performance of FFEs can be easily described as follows: feed enters the top distributor and flows down the tube walls as a thin film by gravity. The heat flux issued from the wall makes a portion of the falling film evaporate, thus producing a liquid-vapour flow which enhances the overall heat transfer coefficient. Then, the liquid-vapour mixture is separated by using a type of

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cyclone, thus keeping liquid entrainments low even when foaming liquors are processed. At low ratios between input flow rate and heat transfer surface partial drying of the tubes may occur, thus reducing the heat transfer effectiveness. This is usually avoided by recirculating a portion of the liquid.

The modelling of multiple-effect evaporation has been recognized as a valuable tool in process design as well as in the analysis of existing plants. Recently, many rigorous mathematical models were proposed to optimize the evaporation process in general (Holland, 1975; Stewart & Beveridge, 1977) or in the sugar industry (Radović *et al.*, 1979).

In this study a modelling of multiple-effect, falling-film evaporators operating in forward flow was undertaken by combining the classic mathematical model of multiple-effect evaporators, which is based on a constant value of the overall heat transfer coefficient in each effect, with a reliable estimation of such a coefficient in falling-film evaporators. To achieve such a goal, the reliability of the available correlations for the prediction of the heat transfer coefficient in turbulent falling films was tested by analysing the performance of a few industrial systems, commonly encountered in the citrus industry. Finally, two different strategies (such as equal or different heat transfer surfaces) were compared to optimize the process under study, as far as the minimum value of the overall heat transfer surface of the system, or primary steam and cooling water requirements, is concerned.

Heat transfer rate in falling-films

Vapour evolution from a thin liquid film flowing downward on a vertical surface can take place by bubble forming at the heating wall or by direct evaporation at the liquid–vapour interface. In particular, the latter mechanism prevails when the total temperature difference is less than 10°C (Narayanamurthy & Sarma, 1977). However, the heat transfer rate in falling films was found to show a large variation according to the type of flow within the film (laminar, wavy laminar, transitional, and fully turbulent regimes), as shown by Oosthuizen & Cheung (1977).

For this reason, a simple model as the Nusselt model (1916), based on direct evaporation at the free surface and on steady-state laminar flow only, is unable to give a thorough reconstruction of the experimental asymptotic dimensionless values of the film heat transfer coefficient (H^+) within a wide range of the Reynolds number (Re). In fact, according to the Nusselt model, H^+ should decrease as Re increases:

$$H^+ = 1.1\ \mathrm{Re}^{-1/3} \tag{1}$$

On the contrary, this trend was experimentally confirmed only for Re less than a critical value ranging from 1600 to 3200, and better correlated by means of the following empirical expression by Chung & Seban (1971):

$$H^+ = 0.606\ \mathrm{Re}^{-0.22} \tag{2}$$

A complete description of the phenomenon under study might be carried out by describing the hydrodynamic and thermal behaviour of the film by means of

Table 1. Available equations for the prediction of the heat transfer rates in turbulent falling films

No.	Equation	Flow characteristics	Ref.
1	$H^+ = 0.01\ (\mathrm{Re\ P})^{1/3}$	Turbulent motion down the inner walls of pipes	McAdams *et al.* (1940)
2	$H^+ = 0.02\ \mathrm{Re}^{1/3}\ (\sin\theta)^{0.2}$	Turbulent motion down flat plates with different angles (θ) of inclination	Garwin & Kelly (1955)
3	$H^+ = 8.7 \times 10^{-3}\ \mathrm{Re}^{0.4}\ \mathrm{Pr}^{0.34}$	Turbulent motion outside of a metal rod heated internally by hot water	Wilke (1962)
4	$H^+ = 6.92 \times 10^{-3}\ \mathrm{Re}^{0.345}\ \mathrm{Pr}^{0.4}$	Turbulent motion down the inside surface of tubes	Ahmed & Kaparthi (1963)
5	$H^+ = 8.54 \times 10^{-4}\ \mathrm{Re}^{0.65}$	Turbulent motion of water only down the inner walls of pipes	Herbert & Stern (1968)
6	$H^+ = 3.8 \times 10^{-3}\ \mathrm{Re}^{0.4}\ \mathrm{Pr}^{0.65}$	Turbulent flow down the surface of an electrical heated vertical tube	Chun & Seban (1971)
7	$H^+ = 0.89\ \delta^{1/3}/\{5 + [\tan^{-1}(2.73\sqrt{\mathrm{Pr}}) - \tan^{-1}(0.455\sqrt{\mathrm{Pr}})]/0.091\sqrt{\mathrm{Pr}} + \ln(\delta/30)/(0.36\ \mathrm{Pr})\}$ with $\ln\delta = 0.786 + 0.103\ln\mathrm{Re} + 0.041\ (\ln\mathrm{Re})^2$*	Theoretical expression applicable for $\mathrm{Pr} > 1$ and $\delta > 30$	Narayamamurthy & Sarma (1977)

* This regression was derived from the results of Dukler & Bergelin (1952), Belkin *et al.* (1959); and Narayanamurthy & Sarma (1977).

combined laminar and turbulent mechanisms, although suitable expressions for eddy diffusivity of momentum and heat are not yet available (Dukler, 1959; Seban & Faghri, 1976; Brumfield & Theofanous, 1976; Narayanamurthy & Sarma, 1977).

From the chemical engineering point of view a rational design of FFEs might even be carried out by using a simple, but reliable correlation of H^+ under turbulent flow conditions, since the wave structure of the turbulent film involves a reduction of the average film thickness, thus increasing H^+ and reducing the heat transfer surface.

Table 1 reviews the main equations reported in the literature for heat transfer rates in turbulent falling films. The reliability of the only equations which take into account the effect of the Prandtl number (Pr) and refer to turbulent flow down tubes and pipes are examined below.

Mathematical model

Modelling of multiple-effect falling-film evaporators (MEFFE) operating in forward flow was carried out by taking into account the following:

1 for all practical purposes the solute is quite non-volatile at the prevailing conditions, thus allowing a complete vaporization of the solvent without removal of the solute;

2 the useful temperature difference effective upon the heat transfer surface is reduced by the effect of boiling point rise only; and
3 incondensable gases (such as, air leakage and air released from the feed liquors) do not affect the overall pressure and the effectiveness of heat transfer in the system, whereas their amount has to be estimated in order to design the vacuum equipment (e.g. ejector or vacuum pump, barometric or surface condensers, extraction pump or barometric leg), as shown further below.

With reference to the typical scheme shown in Fig. 1, the system can be described by imposing the overall and solute material balances, and the heat balance across each generic effect j:

$$S_{j-1}=S_j+V_j \tag{3}$$

$$S_{j-1}x_{j-1}=S_jx_j \tag{4}$$

$$S_{j-1}h_{j-1}+V_{j-1}(H_{j-1}-h_{j-1})(1-\beta_j)=V_jH_j+S_jh_j \tag{5}$$

where all the symbols are reported in the Nomenclature section (page 561).

The performance of each falling-film evaporator is also described by the following heat transfer equation:

$$V_{j-1}\,(H_{j-1}-h^*_{j-1})(1-\beta_j)=U_{Dj}A_j(T_{j-1}-t_j) \tag{6}$$

where T_{j-1} is the condensation temperature of the steam, which comes from the $(j-1)$th effect and condenses in the shell of the jth evaporator:

$$T_{j-1}=t_{j-1}-\Delta\ T_{b,j-1} \qquad j=2,3,\ldots,n \tag{7}$$

and T_0 is the condensation temperature of live steam.

Provided that the equilibrium conditions are achieved in each effect, the temperature of the boiling liquor and solvent vaporized are equal (t_j). Owing to the rise in boiling point (ΔT_{bj}) of the liquor, the vapour leaving the jth effect is superheated, while the overall pressure (P_j) in the separator j is equal to the vapour pressure of the solvent:

$$P_j=p_s(T_j) \tag{8}$$

where p_s is a known function of the system under study. In the case of food materials the solvent is usually water and its vapour pressure–temperature relationship is readly available.

When the liquor recirculates through the evaporator, the weight flow rate (F_j), composition (y_j), and temperature ($\bar{t}_j$) of the solution entering the jth evaporator can be easily determined by solving the following overall- and solute-material and heat balances:

$$F_j=S_{j-1}+E_jS_j \tag{9}$$

$$F_jy_j=S_{j-1}x_{j-1}+E_jS_jx_j \tag{10}$$

$$F_j\bar{h}_j=S_{j-1}h_{j-1}+E_jS_jh_j \tag{11}$$

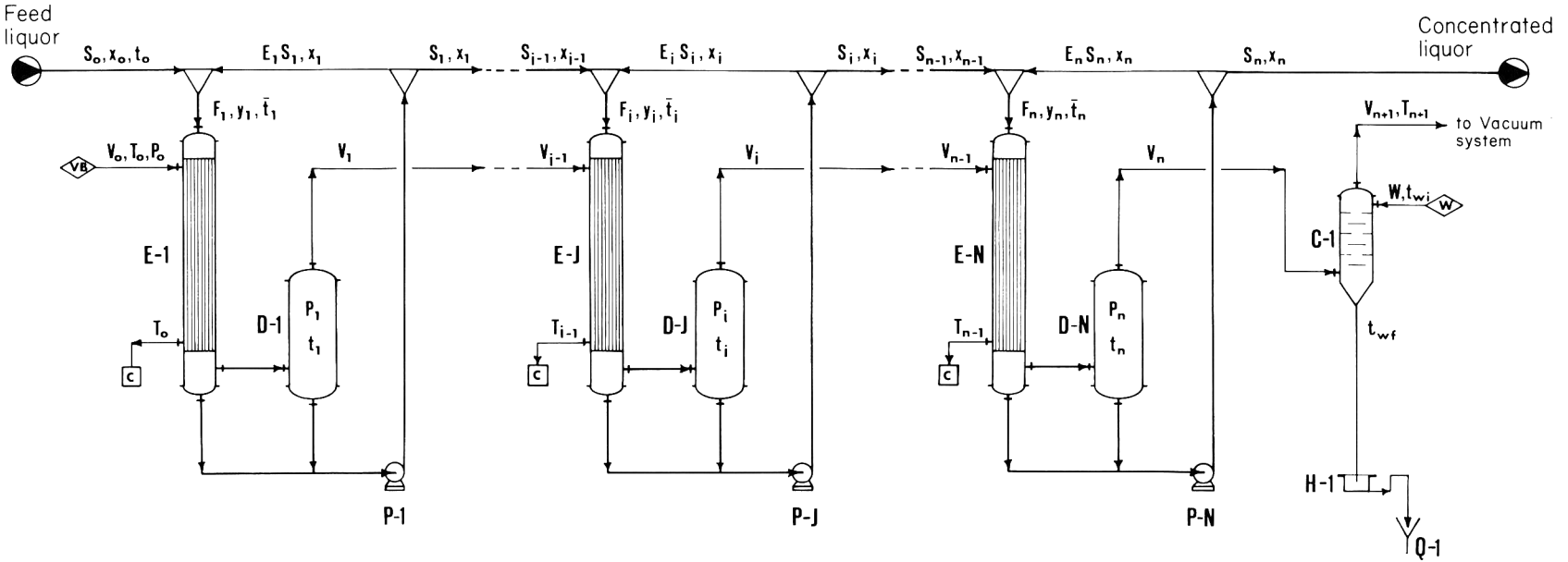

Figure 1. Typical flow diagram of a forward multiple-effect falling-film evaporator system. Item list: C=barometric condenser; D=liquid–vapour separator; E=falling-film evaporator; H=hydraulic seals; P=centrifugal pump; Q=sewage discharges. Utility identification items: C=condensed steam; VB=low pressure steam; W=cooling water. Stream symbols as in the Nomenclature section.

where E_j is the recirculation ratio. When single pass FFEs are considered, E_j is equal to zero.

Procedure of calculation

The previous system of non-linear equations describing MEFFEs leads to a determinate solution provided that the number of degrees of freedom (d.f.) pertaining to this problem is obviously zero. With reference to Figure 1, the independent variables are the following:

S_j; x_j; t_j; V_j; P_j; T_j $\quad j=0, 1, \ldots, n$

and

A_j; U_{Dj}; β_j; E_j; F_j; y_j; ℓ_j $\quad j=1, 2, \ldots, n$

while their overall number is

$N_v=13\ n+6$

As the number of independent equations (N_e)—see equations 3–11—is

$N_e=9\ n$

the degrees of freedom of the system become

$\text{d.f.}=N_v-N_e=4\ n+6$

For practical purposes the following parameters are usually specified to make the problem determinate: weight flow rate (S_0), composition (x_0), and temperature (t_0) of the feed liquor; solute composition of the final product (x_n); operating conditions (T_0, P_0, P_n); and thermal losses, overall heat transfer coefficients, and recirculation ratios (β_j, U_{Dj}, and E_j for $j=1, 2, \ldots, n$).

However, at this stage the problem is still indeterminate with $(n-1)$ degrees of freedom and a determinate solution can be obtained as follows:

1 Identical heat transfer surfaces in all the evaporators:

$$A_j=A=\text{const} \qquad j=1,2,\ldots,n \tag{12}$$

This assumption gives the advantage of designing a single evaporator whatever the number of effects, thus reducing the engineering, construction, and installation costs of the evaporation unit.

2 Different heat transfer surfaces in all the evaporators:

$$A_j \neq A_{j+1} \qquad j=1,2,\ldots,n \tag{13}$$

This working hypothesis has to be coupled with some other condition, being a problem of optimization as indicated by Holland (1975). For instance, the required $(n-1)$ values of A_j might be assigned by minimizing the overall costs of the evaporation unit or assuming identical temperature differences in all the evaporators:

$$T_{j-1}-t_j=\Delta T=\text{const} \qquad j=1,2,\ldots,n \tag{14}$$

With forward flow condition (14) *a priori* involves that the more concentrated and viscous the solution, the greater the heat transfer surface becomes.

All the mentioned assumptions will be further analysed and compared with several industrial applications.

Once given the mentioned process and design parameters, the number of effects (n), and some geometrical parameters (size—d_o, d_i, L—and closeness—P_T—of tubes and their type of pitch), the design procedure of MEFFEs may be summarized as follows:

1 Calculate the first trial values of solvent flow rate vaporized in each effect:

$$V = \frac{S_0(1 - x_0/x_n)}{n} \qquad j = 1, 2, \ldots, n \tag{15}$$

2 Evaluate the liquor flow rate (S_j) in each stage by using equation 3.

3 Estimate the solute weight fraction (x_j) in each stage by using equation 4.

4 Calculate the condensation temperature of the solvent vaporized in the nth effect from equation 8:

$$P_n = p_s\,(T_n) \tag{16}$$

5 Assume a tentative set of t_j values for all the n effects.

6 Calculate the boiling point rise (ΔT_{bj}) of liquor in each stage. This parameter is in general a function of temperature (t_j) and solute concentration (x_j):

$$\Delta T_{bj} = f(t_j, x_j) \tag{17}$$

Unfortunately, this function is available only for a limited number of liquors, for instance, orange (Moresi & Spinosi, 1980) and lemon juices (Varshney & Barhate, 1978).

7 Calculate the specific enthalpy of each stream in each effect:

$$h_j = c_p t_j \tag{18}$$

$$h_j^* = c_{w1} T_j \tag{19}$$

$$H_j = \lambda_w + c_{wv} T_j \tag{20}$$

where T_j can be evaluated by using equation 7, while the reference temperature for enthalpy calculation is 0°C.

8 Calculate the live steam flow rate (V_0) from equation 5 with $j=1$.

9 Evaluate the condensation temperatures (T_1, T_2, . . . , T_{n-1}) of the solvent vaporized in all the effects, except the last one estimated at step 4, by solving the following system of $(n-1)$ linear equations (obtained by substituting equation 7 into equation 6 and dividing by $U_{Dj}A_j$):

$$V_{j-1}(H_{j-1} - h_{j-1}^*)(1-\beta_j)/(U_{Dj}A_j) = T_{j-1} - T_j - \Delta T_{bj} \tag{21}$$

If the heat transfer surfaces are equal ($A_j = A = \text{const}$), A can be evaluated by summing up both sides of equation 21 for j ranging from 1 to n, thus

yielding:

$$A = \frac{\sum_{1}^{n_j} V_{j-1}\,(H_{j-1} - h^*_{j-1})(1-\beta_j)/U_{Dj}}{T_0 - T_n - \sum_{1}^{n_j} \Delta T_{bj}} \tag{22}$$

If all the heat transfer surfaces are different ($A_j \neq A_{j+1}$), $(n-1)$ values of A_j are to be prefixed in a certain way, while the remaining one (for instance, A_1) can be calculated by applying the same procedure used to derive equation 22:

$$A_1 = \frac{V_0(H_0 - h^*_0)(1-\beta_1)/U_{D1}}{T_0 - T_n - \sum_{1}^{n_j} \Delta T_{bj} - \sum_{2}^{n_j} V_{j-1}\,(H_{j-1} - h^*_{j-1})(1-\beta_j)/(U_{Dj}A_j)} \tag{23}$$

In both cases the condensation temperatures (T_j) can be easily determined by solving equation 21 for $j=1, 2, \ldots, (n-1)$.

If the temperature differences in all the evaporators are equal (ΔT=const), ΔT can be calculated by substituting equation 7 into the design condition (14) and adding up the two sides of equation 14 for $j=1, 2, \ldots, n$:

$$\Delta T = \frac{T_0 - T_n - \sum_{1}^{n_j} \Delta T_{bj}}{n} \tag{24}$$

while each condensation temperature (T_j) can be determined by solving in sequence equation 14:

$$T_j = T_{j-1} - \Delta T_{bj} - \Delta T \qquad j=1,2,\ldots,(n-1) \tag{25}$$

Each heat transfer surface (A_j) can finally be estimated by using equation 21.

10 Calculate the boiling temperature (t_j) of the liquor in each effect from equation 7.

11 Estimate the specific enthalpies of all the liquid and vapour streams by using equations 18–20 for $j=1, 2, \ldots, (n-1)$.

12 Determine the algebric solution ($V_0, V_1, \ldots, V_n, S_1, \ldots, S_{n-1}$) of the $2n$ linear equation—(3) and (5)—system:

$$\begin{vmatrix} \Delta H_0 & -H_1 & 0 & \cdots & 0 & 0 & -h_1 & 0 & \cdots & 0 \\ 0 & \Delta H_1 & -H_2 & \cdots & 0 & 0 & h_1 & -h_2 & \cdots & 0 \\ \cdot & \cdot & \cdot & \cdots & \cdot & \cdot & \cdot & \cdot & \cdots & \cdot \\ 0 & 0 & 0 & \cdots & \Delta H_{n-1} & -H_{n-1} & 0 & 0 & \cdots & h_{n-1} \\ 0 & 1 & 0 & \cdots & 0 & 0 & 1 & 0 & \cdots & 0 \\ 0 & 0 & 1 & \cdots & 0 & 0 & -1 & 1 & \cdots & 0 \\ \cdot & \cdot & \cdot & \cdots & \cdot & \cdot & \cdot & \cdot & \cdots & \cdot \\ 0 & 0 & 0 & \cdots & 0 & 1 & 0 & 0 & \cdots & -1 \end{vmatrix} \begin{vmatrix} V_0 \\ V_1 \\ \cdot \\ V_n \\ S_1 \\ S_2 \\ \cdot \\ S_{n-1} \end{vmatrix} = \begin{vmatrix} -h_0 S_0 \\ 0 \\ \cdot \\ h_n S_n \\ S_0 \\ 0 \\ \cdot \\ -S_n \end{vmatrix} \tag{26}$$

where

$$\Delta H_j = (H_j - h_j^*)(1 - \beta_{j+1}) \qquad j = 0, 1, \ldots, (n-1) \tag{27}$$

by using the method of Gaussian elimination (Lapidus, 1962).

13 Estimate a new set of solute weight fractions (x'_j) by using equation 4 for $j=1, 2, \ldots, (n-1)$.

14 Check if the difference between the old (x_j) and new (x'_j) sets of solute weight fractions in each effect is less than a prescribed tolerance (ε):

$$|x'_j - x_j| \leq \varepsilon \qquad j=1, 2, \ldots, (n-1) \tag{28}$$

If the above difference is greater than ε, the calculation procedure is repeated from step 6 by using the method of direct substitution modified to speed the convergence as follows:

$$x_j = (x_j + 0.5\, x'_j)/1.5 \qquad j=1, 2, \ldots, (n-1)$$

If condition 28 is satisfied for $j=1, 2, \ldots, (n-1)$, the iterative process is said to have converged and the calculation procedure can continue.

15 Calculate the following parameters (F_j, y_j, $\bar{h}_j$, and $\bar{t}_j$) of each solution entering each evaporator (see Fig. 1) by using equations 9–11 and equation 18, respectively, for $j=1, 2, \ldots, n$.

As soon as the liquid stream F_j is forced through the film-forming device of the jth evaporator, flashing may occur provided that $\bar{t}_j$ is greater than the equilibrium boiling temperature (t_{Bj}) at the prevailing pressure P_j in the same evaporator (for the sake of simplicity the pressure drop through each heat exchanger was assumed to be negligible). Of course, for $\bar{t}_j < t_{Bj}$ no flashing occurs and the calculation procedure continues from step 17.

16 Estimate the inlet temperature (t'_j) of the liquid stream after flashing according to the following iterative process. A first estimate of $t'_j = \bar{t}_j$ is made to calculate the composition:

$$y'_j = \frac{\lambda_w + c_{wv}\, t'_j - h'_j}{\lambda_w + c_{wv}\, t'_j - \bar{h}_j}\, y_j \tag{29}$$

and the boiling-point rise $\Delta T'_{bj} = f(t'_j, y'_j)$ of the flashed liquor, then a new estimate of t'_j is calculated by summing up the mentioned boiling point rise to the saturation temperature of solvent at P_j and resubstituted into equation 29, thus repeating the process until the difference between two subsequent estimations of t'_j is smaller than 0.5°C.

17 Evaluate the design heat transfer coefficient (U_{Dj}) by combining the clean heat transfer coefficient (U_{Cj}) and the fouling factor (R_{dj}) to take into account the additional resistance to heat transfer due to adhesion of suspended matter or incrustating components present in the thin juice inside and outside the tube:

$$1/U_{Dj} = R_{dj} + 1/U_{Cj} \tag{30}$$

where U_{Cj} is the clean overall heat transfer coefficient referred to the temperature difference $(T_{j-1}-t_j)$ in each evaporator. This coefficient can be evaluated by using equations A.16, A.22 and A.24–A.27, as shown in greater detail in the Appendix.

18 Check if the difference between the old (U_{Dj}) and the new (U'_{Dj}) values of the design heat transfer coefficient in each evaporator is less than a prefixed tolerance (ε')

$$|U'_{Dj}-U_{Dj}|\leq\varepsilon' \qquad j=1,2,\ldots,n \tag{31}$$

If the above difference is greater than ε', the process is repeated starting from step 9, by using the method of direct substitution; otherwise, the design of the MEFFE system may be retained satisfactorily and the cooling water required at the barometric condenser is evaluated in the following manner:

19 Estimate the amount of non-condensables of the process (G) by taking into account the air leakage occurring at piping connections (flanges, valves, etc.), stuffing boxes, mechanical equipment seals, etc., and the air released from the inlet liquor, which is assumed to be saturated at the input temperature (t_0). By using the average air leakage per each type of fitting, extracted from Ludwig (1964), the former may be quoted as 1.1×10^{-3} kg air leakage/sec/each effect (heat exchanger and liquid–vapour separator), while the latter can be assumed to be 1.9×10^{-5} kg air/kg liquor (Ludwig, 1964, thus obtaining

$$G=1.1\times10^{-3}\,n+1.9\times10^{-5}\,S_0 \tag{32}$$

20 Fix the temperature of the exit gaseous stream from the barometric condenser 3°C greater than the inlet cooling water temperature (t_{wi}):

$$T_{n+1}=t_{wi}+3 \tag{33}$$

while set the outlet cooling water temperature (t_{wf}) as that of steam corresponding to vacuum less 3°C:

$$t_{wf}=T_n-3 \tag{34}$$

21 Calculate the cooling water consumption (W) by writing the heat balance across the barometric condenser:

$$W=\frac{(V_n-V_{n+1})(H_n-h_f^*)+(G\,c_G+V_{n+1}\,c_{wv})(T_n-T_{n+1})}{h_f^*-h_i^*} \tag{35}$$

where

$$V_{n+1}=\frac{M_w}{M_a}\cdot\frac{p_s(T_{n+1})}{P_n-p_s(T_{n+1})}\,G \tag{36}$$

and M_w and M_a are the molecular weight of water and air.

Results and Discussion

Analysis of industrial evaporator plants

The performance of a few industrial double-effect falling-film evaporators for citrus juice concentration manufactured by Officine Metalmeccaniche Santoro (OMS) SpA (Messina, Italy) was simulated by means of the mathematical model previously described using only the most reliable heat transfer correlations shown in Table 1.

Table 2. Input data required for the analysis of two industrial citrus-juice double-effect falling-film evaporator plants. (Courtesy of Officine Metalmeccaniche Santoro SpA, Messina, Italy.)

	Water removal capacity (kg h^{-1})		
Input data	1000	2000	Unit
(1) Feed flow rate (S_0)	1200	2400	kg h^{-1}
(2) Feed temperature (t_0)	18	18	°C
(3) Feed concentration (x_0) a	10	10	°Brix
b	8	8	°Brix
(4) Output concentration (x_2) a	60	60	°Brix
b	40	40	°Brix
(5) Live steam temperature (T_0)	95	95	°C
(6) Live steam pressure (P_0)	83.8	83.8	kPa
(7) Second effect pressure (P_2)	6	6	kPa
First effect			
(8) Thermal loss (β_1)	0.03	0.03	—
(9) Recirculation ratio (E_1)	0	0	—
(10) Design HTC (U_{D1})	c	c	W m^{-2} $°C^{-1}$
(11) Heat transfer surface (A_1)	38	65	m^2
Second effect			
(12) Thermal loss (β_2)	0.03	0.03	—
(13) Recirculation ratio (E_2)	0	0	—
(14) Design HTC (U_{D2})	c	c	W m^{-2} $°C^{-1}$
(15) Heat transfer surface (A_2)	71	95	m^2

a, orange juice;
b, lemon juice;
c, calculable using equations 30, A16, A24–27 and the heat transfer correlations shown in Table 1.

The input data required for this analysis are given in Table 2 and refer to two different plant sizes (i.e. 1000 and 2000 kg of water removal per hour). Among them, the values of the thermal losses β_1 and β_2, being not given by OMS SpA, were estimated as 3% of total heat load.

In particular, the concentration of each lot of citrus juice entering the first stage and leaving the second one was determined by means of a standard refractometer at 25°C, while the operating temperatures and pressures in both

Table 3. Regression equations of the physical properties of citrus juices

Physical property	Regression	Unit	Ref.
Boiling-point rise	a $\Delta T_b = 3.2x - 2.42x^2 + 14x^3$	K	1
	b $\Delta T_b = 11.22x$	K	2
Viscosity	a $\mu = \alpha T_K^{\beta}$	mPa s	1
	$\alpha = \exp(34.67 - 20.24x + 162x^2)$	(mPa s). K $^{\beta}$	1
	$\beta = -6.11 + 3.96x - 26.8x^2$	—	1
Density	a $\rho = 0.9944 + 0.307x + 0.282x^2$	g cm $^{-3}$	1
Specific heat	a $c_p = 4.186 - 2.679x$	J (g K)$^{-1}$	1
Thermal conductivity	c $k = 0.213 + 1.316 \times 10^{-3} T_K - 0.339x$	W (m K) $^{-1}$	3

a, Orange juice; b, lemon juice; c, aqueous sucrose solutions; 1, Moresi & Spinosi (1980); 2, Varshney & Barhate (1978); and 3, Honig (1953).

Table 4. Analysis of an industrial orange-juice double-effect falling-film evaporator (water removal capacity of 1000 kg h^{-1}) manufactured by OMS SpA (Messina, Italy) using different heat transfer correlations (A, McAdams, Drew & Bays (1940); B, Wilke (1962); C, Ahmed & Kaparthi (1963); D, Chun & Seban (1971); E, Narayanamurthy & Sarma (1977)). Comparison between the calculated and recorded output data. (Input data are given in Table 2).

Output data	A	B	C	D	E	OMS SPA	Unit
Live steam consumption (V_0)	644	645	645	634	629	600–650	kg h^{-1}
Cooling water consumption (W)	24.5	24.5	24.5	24.4	24.3	23–27	m^3 h^{-1}
First effect							
Liquor concentration at outlet (x_1)	17.09	17.08	17.08	17.13	17.18	16–18	°Brix
Pressure (P_1)	34.61	36.6	36.1	26.5	20.4	15–18	kPa
Temperature (t_1)	73.1	74.4	74.1	67.0	61.1	55–58	°C
Boiling-point rise (ΔT_{b1})	0.55	0.55	0.55	0.55	0.55	—	°C
Clean heat transfer coefficient (U_{C1})	1544	1900	1308	1235	2763	—	W (m^2 °C)$^{-1}$
Second effect							
Liquor concentration at outlet (x_2)	60	60	60	60	60	60	°Brix
Pressure (P_2)	6	6	6	6	6	6	kPa
Temperature (t_2)	40.4	40.4	40.4	40.4	40.4	40	°C
Boiling-point rise (ΔT_{b2})	4.1	4.1	4.1	4.1	4.1	—	°C
Clean heat transfer coefficient (U_{C2})	171	162	158	254	655	—	W (m^2 °C)$^{-1}$
Fouling factor (R_d)	0.0015	0.0015	0.0013	0.0019	0.0030	—	m^2 °C W^{-1}

stages were measured by using standard thermocouples and vacuum gauges, respectively.

The physical properties of orange and lemon juices necessary for this study (e.g. boiling-point rise, ΔT_b; viscosity, μ; density, ϱ; specific heat, c_p; and thermal conductivity, k), were expressed as shown in Table 3. In particular, k, being unknown for both juices, was assumed to be equal to that of aqueous sucrose solutions: the k values extracted from Honig (1953) and referred to different temperatures (0–80°C) and sugar contents (0–60°Brix) were correlated by means of the method of least squares (mean s.e.<1.5%) (see Table 3). As far as lemon juices are concerned, the ΔT_b regression was derived by smoothing the results of Varshney & Barhate (1978), while their physical properties, being unknown, were supposed to be similar to those of orange juices for the sake of simplicity.

In the case of a double-effect evaporator (n=2) the degrees of freedom are 14, while the number of the input variables shown in Table 2 is 15, thus allowing the calculation of an unknown parameter of this problem. Provided that identical fouling factors might be expected in all the evaporators, a generic

Table 5. Analysis of an industrial orange-juice double-effect falling-film evaporator (water removal capacity of 2000 kg h^{-1}) manufactured by OMS SpA (Messina, Italy) using the heat transfer correlations as in Table 4: comparison between the calculated and recorded output data. (Input data are given in Table 2.)

Output data	A	B	C	D	E	OMS SpA	Unit
Live steam consumption (V_0)	1303	1305	1306	1283	1268	1200–1300	kg h^{-1}
Cooling water consumption (W)	48.9	49.0	49.0	48.7	48.5	46–54	m^3 h^{-1}
First effect							
Liquor concentration at outlet (x_1)	17.04	17.03	17.03	17.10	17.15	16–18	°Brix
Pressure (P_1)	40.9	46.8	46.9	32.2	24.6	18–20	kPa
Temperature (t_1)	79.3	80.3	80.4	71.4	65.3	58–60	°C
Boiling-point rise (ΔT_{b1})	0.54	0.54	0.54	0.55	0.55	—	°C
Clean heat transfer coefficient (U_{C1})	1752	2159	1491	1352	2862	—	W (m^2 °C)$^{-1}$
Second effect							
Liquor concentration at outler (x_2)	60	60	60	60	60	60	°Brix
Pressure (P_2)	6	6	6	6	6	6	kPa
Temperature (t_2)	40.4	40.4	40.4	40.4	40.4	40	°C
Boiling-point rise (ΔT_{b2})	4.1	4.1	4.1	4.1	4.1	—	°C
Clean heat transfer coefficient (U_{C2})	192	187	179	293	657	—	W (m^2 °C)$^{-1}$)
Fouling factor (R_d)	0.0007	0.0008	0.0005	0.0012	0.0022	—	m^2 °C W^{-1}

expression of R_d can be derived by substituting equation 30 into equation 21 and summing up both sides of equation 21 for $j=1, 2, \ldots, n$:

$$R_d = \frac{T_0 - T_n - \sum_1^n{}_j \Delta T_{bj} - \sum_1^n{}_j V_{j-1} \Delta H_{j-1}/(A_j U_{Cj})}{\sum_1^n{}_j V_{j-1} \Delta H_{j-1}/A_j} \quad (37)$$

In Tables 4 and 5 the values of the process variables in each effect, primary steam and cooling water requirements, and fouling factor calculated by using different heat transfer correlations are compared to those recorded in the two industrial plants previously mentioned. These comparisons made it possible to underline that the heat transfer correlation developed by Narayanamurthy & Sarma (1977) allowed a better reconstruction of the operating variables (e.g. solute fraction, liquor temperature and working pressure) in the first effect of both plants. The simulation of other two lemon-juice double-effect falling-film evaporators based on the input data of Table 2 allowed a further confirmation of the goodness of the correlation mentioned above (see Table 6).

The calculated values of R_d (see Tables 4–6) were found to be much greater

Table 6. Analysis of two industrial lemon-juice double-effect falling-film evaporators manufactured by OMS SpA (Messina, Italy) using the heat transfer correlation developed by Narayanamurthy & Sarma (1977): comparison between the calculated and recorded output data. (Input data are given in Table 2.)

	Water removal capacity (kg h^{-1})				
	1000		2000		
Output data	Calc.	OMS SpA	Calc.	OMS SpA	Unit
Live steam consumptions (V_0)	605	600–650	1219	1200–1300	kg h^{-1}
Cooling water consumption (W) (temperature range: 18–34 °C)	23.4	23–27	46.7	46–54	m^3 h^{-1}
First effect					
Liquor concentration (x_1)	13.3	13–15	13.3	13–15	°Brix
Pressure (P_1)	18.2	12–15	21.1	12–15	kPa
Temperature (t_1)	59.7	50–55	62.9	50–55	°C
BPR (Δt_{b1})	1.5	—	1.5	—	°C
Clean heat transfer coefficient (U_{C1})	2888	—	2958	—	W m^{-2} $°C^{-1}$
Second effect					
Liquor concentration (x_2)	40	40	40	40	°Brix
Pressure (P_2)	6.0	6.0	6.0	6.0	kPa
Temperature (t_2)	40.8	40	40.8	40	°C
BPR (Δt_{b2})	4.5	—	4.5	—	°C
Clean heat transfer coefficient (U_{C2})	1320	—	1332	—	W m^{-2} $°C^{-1}$
Fouling factor (R_d)	0.0033	—	0.0025	—	m^2 °C W^{-1}

than those commonly used for a variety of processes in the chemical industry: i.e. $R_d=(0.15-1.5)\times10^{-3}$ m^2 °C W^{-1}. This parameter, in fact, is intended to protect any heat exchanger from delivering less than the required process heat load for a period of about a year to a year and a half before carrying out a cleaning operation. On the contrary, in the food industry periodic daily or weekly cleanings are set up to hold the change in the heat transfer due to scale formation within fixed limits. For instance, in a multiple-effect sugar evaporator the scale formed in 2½ days involved a reduction of the overall heat transfer coefficient from 1200 to 730 W (m^2 °C)$^{-1}$ (Kerr, 1950), which is equivalent to $R_d=0.5\times10^{-3}$ m^2 °C W^{-1}. Similar results may be extracted from the figures collected by Honig (1963).

In our specific case, it is undeniable that the R_d values derived from the performance of the evaporators concerned, being twice or three times greater than those usually encountered in the chemical industry services, unduly swamp the clean overall heat-transfer coefficient and heat transfer effectiveness of FFEs. In fact, as personally communicated by the manufacturer, the larger units were less overdesigned than the smaller ones (the R_d values reported in Table 5 were about two-thirds of those shown in Table 4) in order to fit the typical outputs of the citrus industry in the south of Italy. Therefore, such values of R_d appear to apply to small-size evaporators only, especially when high levels of flexibility and reliability in treating different kind of citrus juices are expected.

Design of a new evaporator plant

As pointed out in the Section 'Procedure of Calculation', the design of a multiple-effect evaporator can be carried out by assuming identical or unequal heat transfer surfaces in all the effects. However, the latter case may be solved by simply imposing identical temperature differences in all the effects, or, in a more complex manner, minimizing the overall costs of the evaporation unit.

Table 7 reports the results for the design of an orange-juice double-effect falling-film evaporator system according to the strategies previously mentioned and to the input data shown in Table 2, by varying the values of R_d as shown in the previous section.

As expected, primary steam and cooling water requirements (see Tables 5 and 7) do not depend upon design strategies at a constant number of effects. In fact, the differences between the responses of the model, which are less than 2% and therefore fall within the precision range of this study, do not show any significant advantage of the design based on equal temperature difference over that based on equal area. On the other hand, as far as the overall heat transfer surface is concerned, the design of DEFFEs based on identical heat transfer surfaces seems to be more expedient, especially if the engineering, construction, and installation costs are taken into account. Moreover, it is worthwhile pointing out that the estimated steam and cooling water requirements are in good agreement with those claimed by the manufacturer (see Tables 5 and 7).

Table 7. Design of an orange-juice double-effect falling-film evaporator (water removal capacity of 2000 kg h^{-1}) on the basis of identical heat transfer surfaces (A = const) and identical temperature differences (ΔT = const) in all the effects, using the correlation of Narayanamurthy & Sarma (1977), the input data shown in Table 2, and different levels of the fouling factor (R_d)

Output data		$R_d \times 10^4$ (m^2 °C W^{-1}) 0.9	4.3	12.9	21.5	Unit
Live steam consumption (V_0)	a	1308	1297	1285	1280	kg h^{-1}
	b	1274	1274	1274	1274	
Cooling water consumption (W)	a	49.0	48.8	48.7	48.7	m^3 h^{-1}
(temperature range: 21.3–33.3 °C)	b	48.6	48.6	48.6	48.6	
First effect						
Liquor concentration (x_1)	a	17.0	17.1	17.1	17.1	°Brix
	b	17.1	17.1	17.1	17.1	
Pressure (P_1)	a	48.9	40.7	33.4	30.6	kPa
	b	27.7	27.7	27.7	27.7	
Temperature (t_1)	a	81.4	76.9	72.2	70.2	°C
	b	68.0	68.0	68.0	68.0	
BPR (ΔT_{b1})	a	0.55	0.55	0.55	0.55	°C
	b	0.55	0.55	0.55	0.55	
Design heat transfer coefficient (U_{D1})	a	2461	1323	619	403	W (m^2 °C) $^{-1}$
	b	2129	1259	611	402	
Heat transfer surface (A_1)	a	23.9	33.1	55.7	78.1	m^2
	b	13.5	22.8	47.0	71.5	
Second effect						
Liquor concentration (x_2)	a					
	b		60.0			°Brix
Pressure (P_2)	a					
	b		6.0			kPa
Temperature (t_2)	a					
	b		40.4			°C
BPR (ΔT_{b2})	a					
	b		4.07			°C
Design heat transfer coefficient (U_{D2})	a	633	519	357	273	W (m^2 °C) $^{-1}$
	b	628	516	357	273	
Heat transfer surface (A_2)	a	23.9	33.1	55.7	78.1	m^2
	b	37.0	45.0	65.1	85.1	
Overall heat transfer surface	a	47.8	66.2	111.4	156.2	m^2
	b	50.5	67.8	112.1	156.6	

a, A = constant;
b, ΔT = constant.

Since R_d exerts a significant influence upon the overall heat transfer surface and, consequently, upon the investment costs of the evaporation unit under study, one of the designer's main tasks should be to consider very carefully how dirty an evaporator should be allowed to become between two subsequent cleaning operations.

Conclusions

In this study, a mathematical model of multiple-effect falling-film evaporators was developed by combining the general structure of classic multiple-effect evaporator models with an accurate estimation of the overall heat transfer coefficient (HTC) in each effect based on the assumption that the local clean HTC varied linearly with the temperature of the liquid stream and exponentially with the solute fraction in the pre-heating and evaporating sections of the FFE, respectively. In this way, by using the correlation of Narayanamurthy & Sarma (1977) to predict the clean HTC in falling films, it was possible to obtain a fairly good simulation of the operating variables of several industrial orange and lemon juice double-effect falling-film evaporator plants.

Finally, two different design strategies based respectively on equal temperature difference and equal area in each effect were tested at different levels of the fouling factor (R_d). Keeping in mind that no heat transfer equipment with a heat transfer surface larger than that required to fulfil process requirements can wrongly operate unless partial drying of the tubes in falling-film evaporators occurs (see for instance Table 7), the design of FFE's for clarified citrus juice *a priori* scheduled to be daily or weekly cleaned is likely to be based on R_d values ranging from 0.4×10^{-3} to 1.3×10^{-3} m^2 °C W^{-1}, whereas that of highly flexible, small-size evaporation units will probably be carried out on the basis of the R_d values shown in Tables 4–6. Generally speaking, it would however appear to be wiser to advise and design for more frequent cleaning than to overdesign the equipment. In fact, the larger the equipment, the greater the investment and operating costs of the evaporation unit become, especially when higher recirculation ratios have to be maintained to allow an adequate wetting of the larger-than-necessary heat transfer surface.

A later paper will deal with the effect of the design parameters and number of effects on the overall operating costs of MEFFE systems at different water removal capacities.

Acknowledgment

The authors are grateful to Prof. Norman Blakebrough (NCFT, University of Reading, U.K.) for his helpful criticism.

Appendix

Calculation of the overall heat transfer coefficient in falling-film evaporators

The estimation of the overall heat transfer coefficient (HTC) in falling-film evaporators (FFEs) is not an easy task, even when a reliable correlation of the local HTC is available.

Since the inlet temperature t_j of the liquid phase F_j at the top of the jth evaporator may or may not coincide with the equilibrium boiling temperature

(t_{Bj}) at the prevailing pressure P_j in the same evaporator (for the sake of simplicity the pressure drop through the heat exchanger was assumed to be negligible), two different situations may arise.

For $\bar{t}_j > t_{Bj}$, F_j is adiabatically flashed, thus varying its concentration and temperature from y_j to y'_j and from $\bar{t}_j$ to t'_j, respectively (see step 16 in the section 'Procedure of Calculation').

For $\bar{t}_j < t_{Bj}$, the heat flux issued from the wall will make the falling film preheat up to the bubble point before producing a liquid–vapour flow.

In the latter situation the energy balance referred to the differential volume of FFE and heat flow across the tube wall yield the following:

$$dw\,[(H(T)-h^*(T)]=F_j\,dh=U_j\,(T-t)\,dA_j=dQ_{jph} \tag{A1}$$

where A_j is the outside heat transfer surface of FFE, U_j is the local clean HTC referred to the external surface of FFE, that is:

$$\frac{1}{U_j}=\frac{1}{h_{Tj}(d_i/d_o)}+\frac{d_o}{2k_m}\ln(d_o/d_i)+\frac{1}{h_{Toj}} \tag{A2}$$

where k_m is the thermal conductivity of the tube material, h_{Toj} is the condensing film HTC on the steam side referred to the tube outside diameter (d_o) and evaluated in accordance with the Nusselt's theory (Kern, 1950), h_{Tj} is the falling-film HTC calculable as shown in Table 1, and d_i is the inside diameter of the tubes.

As the falling film is heated from $\bar{t}_j$ to t_{Bj}, the viscosity of liquid phase exhibits a large variation, thus affecting h_{Tj}.

By integrating equation A1, the heat transfer surface (A_{jph}) required to pre-heat the liquor up to t_{Bj} can be derived:

$$A_{jph}=F_j\int_{\bar{t}_j}^{t_{Bj}}\frac{(\partial h/\partial t)_{x=y_j}\,dt}{U_j(T_{j-1}-t)} \tag{A3}$$

while the corresponding heat flow (Q_{jph}) is

$$Q_{jph}=F_j\,[h(t_{Bj})-\bar{h}_j] \tag{A4}$$

with

$$t_{Bj}=t_{Bw}(P_j)+\Delta T_b(y_j) \tag{A5}$$

where $t_{Bw}(P_j)$ is the boiling point of pure water at P_j, and $\Delta T_b(y_j)$ is the boiling point rise corresponding to a solute concentration y_j.

As soon as the temperature of liquor is equal to, or greater than, t_{Bj}, a different heat transfer mechanism has to be taken into account.

With reference to Figure A1 the solute material and heat balances across the differential volume, and total heat flow across the tube wall can be written as follows:

$$x\,ds=s\,dx \tag{A6}$$

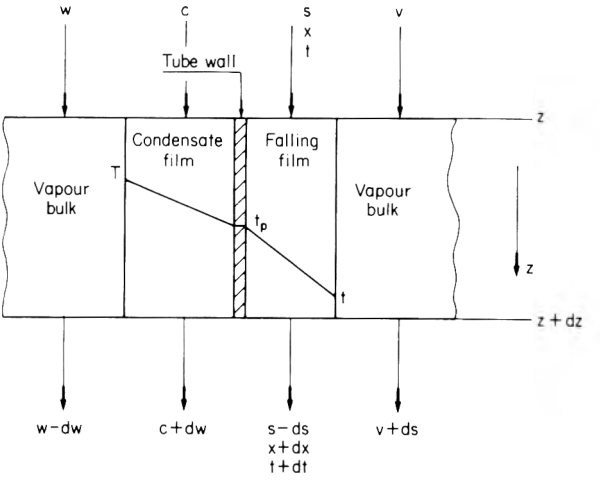

Figure A1. Model for heat transfer description in falling-film evaporators.

$$\mathrm{d}w\,[H(T) - h^*(T)] = \left[s\frac{\partial h}{\partial t} + v\frac{\mathrm{d}H}{\mathrm{d}t}\right]\mathrm{d}t + [H(t) - h(t,x)]\,\mathrm{d}s$$

$$= U_j(T_{j-1} - t)\,\mathrm{d}A_j = \mathrm{d}Q_{jev} \tag{A7}$$

while the overall- and solute-material balances from $A_j = A_{jph}$ to a generic $A_j > A_{jph}$ involve:

$$F_j = s + v \tag{A8}$$

and

$$F_j y_j = sx \tag{A9}$$

As the falling-film evaporates, the solute fraction (x) in the liquor increases, thus greatly affecting the physical properties of the liquid phase and therefore the inside HTC (h_{Tj}).

Since the falling-film generally exerts the controlling resistance to heat transfer and the thermal resistance of the tube wall is practically negligible with respect to that of either inside or outside films, U_j appears to be a complex function of the solute fraction—that is, $U_j(x)$—the boiling temperature of liquor being dependent on solute concentration only—see for instance equation (A5).

By combining equations A6–A9 and integrating modified equation A7 between the inlet and outlet solute fractions of liquor in the jth effect, it was possible to evaluate the heat transfer area required to concentrate the liquor between the limits given above, that is:

$$A_{jev} = F_j y_j \int_{y_j}^{x_j} \left\{\left[\frac{\partial h}{\partial t} + \left(\frac{x}{y_j} - 1\right)\frac{\mathrm{d}H}{\mathrm{d}t}\right]x\frac{\partial \Delta T_b}{\partial x} + (H - h)\right\}\frac{\mathrm{d}x}{x^2 U_j(x)(T_{j-1} - t)} \tag{A10}$$

with

$$t = t_{Bw}(P_j) + \Delta T_b(x) \tag{A11}$$

The associate heat flow is

$$Q_{jev}=F_j\{[H_j-h(t_{Bj},y_j)]-y_j/x_j(H_j-h_j)\} \tag{A12}$$

In general, the overall heat transfer surface (A_j) of the jth evaporator can be determined by simply summing up equations A3 and A10, while the clean overall HTC in FFE(U_{Cj}) can be conventionally related to the difference between the temperatures of condensing steam (T_{j-1}) and boiling liquor at the exit of each FFE(t_j):

$$U_{Cj}=\frac{Q_{jph}+Q_{jev}}{A_j(T_{j-1}-t_j)} \tag{A13}$$

By substituting equations A3–A4, A10 and A12 into equation A13, the reciprocal of U_{Cj} yields the following:

$$\frac{1}{U_{Cj}}=\frac{(T_{j-1}-t_j)}{(H_j-\bar{h}_j)-y_j/x_j(H_j-h_j)}\left\{\int_{\bar{t}_j}^{t_{Bj}}\frac{\partial h/\partial t\,dt}{U_j(T_{j-1}-t)}\right.$$
$$\left.+y_j\int_{y_j}^{x_j}\frac{[\partial h/\partial t+(x/y_j-1)(dH/dt)]\,x(\partial\Delta T_b/\partial x)+(H-h)}{x^2U_j(x)(T_{j-1}-t_{Bw}-\Delta T_b)}\,dx\right\} \tag{A14}$$

The application of equation A14 appears to be troublesome, especially if it is inserted into an iterative routine, such as that described in the section 'Procedure of Calculation'. Therefore, the following series of simplifying hypotheses will be assumed and then checked to make its application easier:

1 Boiling point of liquor approximately coincides with that of pure water, boiling-point, rise of fruit and vegetable juices being usually smaller than 5°C and therefore negligible with respect to t_{Bw}, that is

$$\Delta T_b \ll t_{Bw}$$

In these circumstances, it is possible to extrapolate that even the derivative of ΔT_b with respect to x is practically equal to zero:

$$\frac{\partial\Delta T_b}{\partial x}\approx 0 \tag{A15}$$

2 Specific enthalphy of liquor and vapour phases is constant over the entire length of FFE, the pressure in each effect being constant.

By substituting the above assumptions into equation A14, the following is derived:

$$\frac{1}{U_{Cj}}=\alpha+\gamma \tag{A16}$$

with

$$\alpha=\frac{(T_{j-1}-t_j)(\partial h/\partial t)_{x=y_j}}{(H_j-\bar{h}_j)-y_j/x_j(H_j-h_j)}\int_{\bar{t}_j}^{t_{Bj}}\frac{dt}{U_j(t)(T_{j-1}-t)} \tag{A17}$$

and

$$\gamma = \frac{y_j}{(H_j - \bar{h}_j)/(H_j - h_j) - y_j/x_j} \int_{y_j}^{x_j} \frac{dx}{x^2 U_j(x)} \tag{A18}$$

where the first term (α) on the right-hand side of equation A16 refers to the eventual pre-heating section of FFE, which is omitted if $\bar{t}_j$ is equal to, or greater than, t_{Bj}; while the second one (γ) refers to the evaporating section of FFE.

As far as the eventual pre-heating section of FFE is concerned, it appears to be reasonable to assume the following expression for the local variation of $U_j(t)$:

$$U_j(t) = a_0 + b_0 t \tag{A19}$$

being the solute fraction constant and equal to y_j throughout this section.

Over the evaporating section of FFE two different approximated expressions of $U_j(x)$ will be firstly assumed and then tested below:

$$U_j(x) = a_1 + b_1 x \tag{A20}$$

and

$$U'_j(x) = a_2 \exp(-b_2 x) \tag{A21}$$

where a_i and b_i are empirical constants.

By substituting equation A19 into equation A17, it is possible to derive the following analytical expression of α:

$$\alpha = \frac{(\partial h/\partial t)_{x=y_j}(T_{j-1} - t_j)(t_{Bj} - \bar{t}_j)}{(H_j - \bar{h}_j) - y_j/x_j(H_j - h_j)} \cdot \frac{\ln\left[U_j(t_{Bj})(T_{j-1} - \bar{t}_j)/U_j(\bar{t}_j)/(T_{j-1} - t_{Bj})\right]}{U_j(t_{Bj})(T_{j-1} - \bar{t}_j) - U_j(\bar{t}_j)(T_{j-1} - t_{Bj})} \tag{A22}$$

By respectively substituting equations A20 and A21 into equation A18, two different analytical expressions of γ can be obtained:

$$\gamma_{Lin} = \frac{y_j}{(H_j - \bar{h}_j)/(H_j - h_j) - y_j/x_j} \cdot \frac{x_j - y_j}{x_j U_j(y_j) - y_j U_j(x_j)} \cdot \left[\frac{U_j(y_j) - U_j(x_j)}{x_j U_j(y_j) - y_j U_j(x_j)} \cdot \ln \frac{x_j U_j(y_j)}{y_j U_j(x_j)} + \frac{x_j - y_j}{x_j y_j}\right] \tag{A23}$$

and

$$\gamma_{exp} = \frac{y_j}{(H_j - \bar{h}_j)/(H_j - h_j) - y_j/x_j} \left\{\frac{b_2}{a_2}[Ei(b_2 x_j) - Ei(b_2 y_j)] + \frac{1}{y_j U_j(y_j)} - \frac{1}{x_j U_j(x_j)}\right\} \tag{A24}$$

Table A1. Comparison between rigorous and approximated values of U_{Cj} in falling-film evaporators at different operating conditions

							U_{Cj} ($W\,m^{-2}\,°C^{-1}$)				
								$U_{Cj}=1/(\alpha+\gamma_{Lin})$		$U_{Cj}=1/(\alpha+\gamma_{exp})$	
y_j (−)	x_j (−)	t_j (°C)	T_{j-1} (°C)	P_j (kPa)	F_j (kg h^{-1})	n_t (−)	Rigorous calculation	Value	Error (%)	Value	Error (%)
0.10	0.17	18.0	95.0	16.5	1200	67	2582	2679	−3.8	2673	−3.5
0.10	0.17	18.0	95.0	19.0	2400	114	2647	2745	−3.7	2739	−3.5
0.17	0.60	36.9	60.5	6.0	1200	124	845	1036	−22.5	680	19.5
0.17	0.60	36.9	64.7	6.0	2400	166	844	1043	−23.6	685	18.9
0.10	0.30	18.0	95.0	16.5	1200	67	2189	2263	−3.4	2210	−0.9
0.10	0.40	18.0	95.0	16.5	1200	67	1990	2060	−3.5	1926	3.2
0.10	0.50	18.0	95.0	16.5	1200	67	1803	1900	−5.4	1636	9.3
0.17	0.30	36.9	60.5	6.0	1200	124	1522	1562	−2.7	1529	−0.5
0.17	0.40	36.9	64.7	6.0	2400	166	1294	1343	−3.8	1236	−4.5
0.17	0.50	36.9	60.5	6.0	1200	124	1061	1155	−8.8	934	12.0
0.30	0.40	37.4	60.5	6.0	1200	124	931	964	−3.5	939	−0.8
0.30	0.50	37.4	64.7	6.0	2400	166	715	778	−8.8	687	3.9
0.30	0.60	37.4	60.5	6.0	1200	124	511	627	−22.6	458	10.5
0.40	0.50	38.1	64.7	6.0	2400	166	519	546	−5.2	524	−0.9
0.40	0.60	38.1	60.5	6.0	1200	124	353	405	−14.5	338	4.3
0.50	0.60	39.1	64.7	6.0	2400	166	245	261	−6.6	247	−0.8
Overall percentage error (%)									11.5		9.0

with

$$b_2 = \frac{\ln\left[U_j(y_j)/U_j(x_j)\right]}{x_j - y_j} \tag{A25}$$

$$a_2 = \left[U_j(y_j)\right]^{x_j/(x_j-y_j)} / \left[U_j(x_j)\right]^{y_j/(x_j-y_j)} \tag{A26}$$

and

$$Ei(u) = \int_{-\infty}^{u} \frac{e^{\zeta}}{\zeta}\,d\zeta = 0.5772157 + \sum_{1}^{\infty} k\,\frac{u^k}{k\,k!} + \ln|U| \tag{A27}$$

With reference to concentrated orange-juice production, Narayanamurthy & Sarma's heat transfer correlation and several operating conditions, equation A14, equations A16, A22, A23, and A16, A22, A24–A27, allowed the rigorous, linear- and exponential-approximated values of U_{Cj} to be calculated in order to check the validity of the previous simplifying assumptions. Table A1 shows the main results of several simulation runs. The smaller the ratio of evaporation (x_j/y_j), the smaller the deviation between rigorous and approximated values of U_{Cj} will be.

Nevertheless, equation A21 not only yielded a better reconstruction of U_{Ci} than equation A20 whatever the ratio of evaporation examined, but also exhibited a maximum deviation in good agreement with the mean s.e. of a large number of heat transfer correlations. For the sake of simplicity it would therefore appear that the best thing one can do is design and analyse multiple-effect falling-film evaporators by replacing equation A14 with the set of equations A16, A22, and A24–A27.

Nomenclature

A	heat transfer surface, m^2;
A_{ph}	heat transfer surface for pre-heating, m^2;
A_{ev}	heat transfer surface for evaporation, m^2;
a_i, b_i	empirical constants;
c	local weight flow rate of condensate, kgs^{-1};
c_G, c_p	
c_{wl}, c_{wv}	specific heat of non-condensable gases, solution, and liquid and vapour water, $J(g°C)^{-1}$;
df	degree of freedom, dimensionless;
d_i	tube inside diameter, m;
d_o	tube outside diameter, m;
E	recirculation ratio, dimensionless;
$Ei(u)$	exponential integral function defined by equation A27;
$f(x, t)$	generic function of x and t;
F	flow rate of the solution entering each effect, kgs^{-1};
g	acceleration due to gravity, ms^{-2};
G	flow rate of non-condensable gases, kgs^{-1};
h	specific enthalpy of liquid phase from each effect, Jkg^{-1};
h'	specific enthalpy of liquid at the top of each evaporator after flashing, Jkg^{-1};
$\bar{h}$	specific enthalpy of liquor entering each effect, Jkg^{-1};
h^*	specific enthalpy of condensed steam or cooling water, Jkg^{-1};
h_T	falling-film heat transfer coefficient, $W(m^2°C)^{-1}$;
h_{T0}	condensing film heat transfer coefficient referred to d_0, $W(m^2°C)^{-1}$;
H	specific enthalpy of vapour phase, Jkg^{-1};
H^+	dimensionless heat transfer coefficient $(=h_T(\mu^2/\varrho^2/g)^{1/3}/k)$;
k	thermal conductivity of liquor, $W(m°C)^{-1}$;
k_m	thermal conductivity of tube material, $W(m°C)^{-1}$;
L	tube length, m;
M_a, M_w	molecular weight of air and water, $gmol^{-1}$;
n_t	number of tubes in each evaporator, dimensionless;
n	number of effects, dimensionless;
N_e	number of independent equations, dimensionless;
N_v	number of independent variables, dimensionless;

p_s vapour pressure of solvent, kPa;
P overall pressure in each effect, kPa;
P_T closeness of tubes, m;
Pr Prandtl number ($=c_p\,\mu/k$), dimensionless;
Q_{ph} heat flow for pre-heating, W;
Q_{ev} heat flow for evaporation, W;
R_d combined inside and outside fouling factor, $m^2°CW^{-1}$;
Re Reynolds number ($=4\Gamma/\mu$), dimensionless;
s local weight flow of evaporating liquor, kgs^{-1};
S flow rate of the liquid phase leaving each effect, kgs^{-1};
t temperature of the vapour and liquid phases leaving each effect, °C;
$\bar{t}$ temperature of the liquid phase entering each effect, °C;
t' temperature of the liquid phase at the top of each evaporator after flashing, °C;
t_B boiling temperature of liquor, °C;
t_{Bw} boiling temperature of pure water, °C;
t_p temperature of tube wall, °C;
t_w temperature of cooling water, °C;
T condensation temperature of the vapour phase, °C;
U_j, U'_j local clean heat transfer coefficient, $W(m^2°C)^{-1}$;
U_C clean overall heat transfer coefficient, $W(m^2°C)^{-1}$;
U_D design overall heat transfer coefficient, $W(m^2°C)^{-1}$;
u limit of integration for equation A27, dimensionless;
v local weight flow of water evaporated, kgs^{-1};
V flow rate of vapour leaving each effect, kgs^{-1};
w local weight flow of condensing steam, kgs^{-1};
W flow rate of cooling water, kgs^{-1};
x mass fraction of solute in each S stream, dimensionless or °Brix;
y mass fraction of solute in the solution entering each effect, dimensionless or °Brix;
y' mass fraction of the solute at the top of each evaporator after flashing, dimensionless;

Greek symbols

α parameter defined by equation A17, $m^2°CW^{-1}$;
β thermal loss of each evaporator, dimensionless;
γ parameter defined by equation A18, $m^2°CW^{-1}$;
Γ weight flow rate per unit width ($=S/\pi\, d_i n_t$), $kg(ms)^{-1}$;
δ dimensionless film thickness;
ΔH parameter defined by equation 27, Jkg^{-1};
ΔT temperature difference in each effect, °C;
ΔT_b boiling-point rise (BPR), °C;
ε, ε' prefixed tolerances for the convergence procedure, dimensionless;

θ angle of inclination of tube, degree;
λ_w latent heat of vaporization of water at 0°C, Jkg^{-1};
μ liquor viscosity, mPas;
ϱ liquor density, gcm^{-3}.

Subscripts
b refers to the bottom section of each evaporator;
f refers to cooling water leaving the barometric condenser;
i refers to cooling water entering the barometric condenser;
j refers to a generic effect;
o refers to feed liquor and live steam at the inlet of the plant;
t refers to the top section of each evaporator;
K expressed in degree Kelvin.

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(Received 20 November 1981)

J. Fd Technol. (1983) **18,** 565–572

Consumer reaction to processed Suntan apples

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Summary

A pilot consumer survey showed apple rings prepared from Suntan apples to be liked, particularly with respect to appearance and taste. People, on the whole, preferred a whiter product; texture was acceptable, although respondents would have preferred it to be firmer. Age appeared to be the major demographic factor influencing response, the colour of the product being more favourable to the 45 and over age group, with the 16–24 age group preferring a less sweet product.

Introduction

Over recent years the apple processing industry has been reluctant to accept apples for processing which do not produce a similar product to Bramley's Seedling. Laboratory tests (Williams, Warrington & Arnold, 1982; Williams, Arnold & Warrington, 1982, 1983), however, indicate that cultivars such as Suntan (Cox's Orange Pippin×Court Pender Plat) and East Malling A3022 (Cox's Orange Pippin×Northern Spy) produce an equally acceptable if not superior product to Bramley. These cultivars retain their structure better than Bramley's Seedling. Thus, both are much more suitable than Bramley for making products requiring a more rigid structure, such as apple rings or slices. The flesh of both cultivars, however, is much more yellow in colour than that of Bramley's Seedling. Consumer reaction to such products and to the more intense yellow colour was surveyed at the British Growers Look Ahead agricultural show during 1980.

Materials and methods

Material

Apples of the cultivar Suntan (25 kg) were obtained in February from controlled atmospheric storage (1.2–2.0% oxygen<1% CO_2 and 3–5°C) from

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0022–1163/83/1000–0565 $02.00

Luddington Experimental Station and processed immediately on arrival at Long Ashton.

Preparation of apple rings

Apple rings were prepared as previously described (Williams *et al.*, 1983) using a blanching time of 2 min and a steeping solution of 30% sucrose and 2.5% malic acid. Samples were then frozen and kept at −18°C until transferred to the site of the agricultural show, where they were held in a conventional refrigerator (7°C) until 15 min before being presented to the consumers.

Assessment

Visitors to the show were approached at random and asked to sit at tasting booths illuminated by normal fluorescent strip lighting. They were presented with a whole apple ring and asked to complete a questionnaire in which they were instructed to give their opinion on the product's appearance, colour (questions 1–5), taste (questions 6–8) and texture (questions 9–11) by ticking appropriate response categories. The actual questions posed and categories in which responses could be given can be seen in Table 2. Respondents were also asked a number of questions on their eating habits (questions 19–20, see Table 1) and the use to which they would put the product (questions 12–16, see Table 3), together with some general questions for use in making demographic classifications (questions 21–27, see Table 1). Although the Suntan rings were presented in a monadic situation, to get some indication of how they would fare against Bramley's Seedling, the normal U.K. culinary apple, participants were asked to comment on the acceptability of the rings in comparison with this latter cultivar (Table 3, question 17). To enable any effect of ordering of possible reply categories on the responses to be evaluated, alternatively distributed questionnaires had the reply categories for questions 1–17 in reverse order (for example, from 'Disliked' to 'Liked very much' alternating with categories running from 'Liked very much' to 'Disliked').

Analysis of data

Response patterns for each question, both for the data as a whole and for the various sub-groups defined by questions 18–27, were tabulated. Differences in patterns of response within a set of sub-groups were tested for by means of χ^2 contingency table analysis.

Results and discussion

Population

Two hundred and twenty two people were interviewed. Respondents were coded into the eleven areas of residence listed in Table 1 according to their

Table 1. Distribution of response to general questionnaire

		People ticking a particular category		National figs %
		No.	%	
19*	Frequency of of eating apples as fresh fruit			
	More than once a week	169	79	—
	Twice a month	32	15	—
	Less frequently	14	7	—
20	Frequency of eating apples in a cooked dessert			
	More than once a week	62	29	—
	Twice a month	103	48	—
	Less frequently	48	23	—
21	Age group			
	16–24	65	30	19
	25–34	42	20	16
	35–44	48	22	15
	45–54	35	16	16
	55–64	16	7	17
	65+	8	4	17
22	Sex			
	Male	157	73	49
	Female	58	27	51
23	Marital status			
	Married	128	60	—
	Single	83	39	—
	Other	4	2	—
24	Community			
	City	42	20	79
	Town	53	25	
	Village	120	56	20
25	Area of residence			
	South West	15	7	39
	South East	28	13	
	Home Counties	27	13	
	East Anglia	30	14	
	Midlands	29	13	24
	North West	26	12	35
	North East	40	19	
	Scotland	5	2	—
	Wales	3	1	—
	Ireland	8	4	—
	Non-specific (U.K., G.B., England)	4	2	—
26	Social class			
	A I	15	8	3
	B II	72	39	16
	C1 IIIN	66	35	—
	C2 IIIM	5	3	25
	DIV/+V	4	2	25
	V Unclassified non earners	25	13	12
26A	Occupation associated with horticulture			
	Yes	57	29	—
	Partial	31	16	—
	No	51	26	—
	Unclassifiable	59	30	—
27	Number of wages coming into household			
	More than one wage	93	45	—
	Not more than one wage	115	55	—

* Numbers refer to question Nos in Assessment Sheet.

answer to question 25. The occupation of the chief wage earner given in question 26 was used to classify respondents into the Registrar Generals six social classes, and also into groups defined by degree of association with the horticultural industry.

Where possible, figures from national statistics (Anon, 1971) are given alongside the demographic data in Table 1. Compared to these statistics, the sample examined was biased towards males, the younger age range, the higher social classes and county rather than town dwellers. The data were, however, examined as they stood, and interactive effects reported where they were found.

Effect of questionnaire design on response

Examination of the responses of the two sub-groups obtained by presentation of the two types of questionnaire indicated very little effect of order on response

Table 2. Distribution of response to sensory questions

		People ticking a particular category				People ticking a particular category	
		No.	%			No.	%
1*	Appearance			7	Acidity		
	Liked very much	16	7		Nothing like sharp enough	7	3
	Liked	114	51		Not quite sharp enough	33	15
	Fair	59	27		About the right sharpness	156	71
	Just acceptable	24	11		Too sharp	22	10
	Disliked	9	4		Much too sharp	3	1
2	Colour			8	Sweetness		
	Liked very much	24	11		Nothing like sweet enough	3	1
	Liked	78	35		Not quite sweet enough	29	13
	Fair	68	31		About the right sweetness	118	55
	Just acceptable	36	16		Too sweet	59	27
	Disliked	15	7		Much too sweet	6	3
3	Preferred whiter			9	Texture		
	Yes	141	64		Liked very much	19	9
	No	79	36		Liked	61	27
4	Preferred more yellow				Fair	64	29
	Yes	20	10		Just acceptable	41	18
	No	190	90		Disliked	37	17
5	Shape			10	Firmness		
	Very attractive	15	7		Nothing like firm enough	26	12
	Attractive	102	46		Not firm enough	112	50
	Neutral	96	43		About the right firmness	81	36
	Unattractive	9	4		Too firm	2	1
6	Taste				Much too firm	1	—
	Liked very much	52	24	11	Preferred structure		
	Liked	99	45		Completely destroyed as in stewed product		
	Fair	28	13		No	200	91
	Just acceptable	27	12		Yes	19	9
	Disliked	13	6				

* Numbers refer to question Nos in Assessment Sheet.

except for questions 6 and 7 (taste and acidity). People marked nearer the top of the scale irrespective of the direction of the scale ($P<0.01$).

Overall responses to sensory aspects of product

Table 2 lists the overall responses to the appearance, flavour and textural aspects of the apple rings. In common with all category scales with a centre point there exists the possibility that respondents could have marked the centre of the scale as an easy option without seriously considering their replies. Nevertheless, the returns indicated that the appearance and shape of the rings were generaly well received although the colour was liked less with the majority of the respondents indicating a preference for a whiter product. When assessing taste, about 70% of the sample population also scored in the top two categories ('Liked' or 'Liked very much'), replies as a whole indicating that the product had the right degree of acidity and sweetness, although there was some indication that a slightly less sweet product may have proved more acceptable. The response to the texture of the rings although indicating it to be acceptable was less favourable, a firmer product generally being preferred.

Uses to which the product may be put

Table 3 lists the responses in respect of the uses to which the apple rings could be put. About two-thirds of the respondents would be prepared to use the rings in a variety of products.

When asked to give an opinion on the use of the apple rings for culinary purposes in general in comparison with Bramley's Seedling, 43% showed no

Table 3. Distribution of responses as to what use apple rings could be put

		People ticking a particular category				People ticking a particular category	
		No.	%			No.	%
12*	As a dessert as it stands			15	In fritters		
	No	67	31		No	68	35
	Yes	136	67		Yes	124	65
				16	Use as a garnish for meals		
13	In fruit salads				No	76	35
	No	74	36		Yes	143	65
	Yes	129	64	17	Preferred for culinary use to Bramley's Seedling		
14	In fruit pies				No preference	95	43
	No	64	32		No	93	42
	Yes	138	68		Yes	33	15

* Numbers refer to question Nos in Assessment Sheet.

preference whereas 42% still showed a preference for Bramley's Seedling and only 15% preferred Suntan. Information from comparisons relying on the memory and experience of the person being asked to make the comparison, must be viewed cautiously. Although it is such a blind comparison that must generally be made when a consumer purchases apples or apple products, people's memories may be distorted and unreliable. The fact that in this case they have also just been presented with one partner in the comparison in the form of apple rings may also influence the response.

Interaction between demographic data and response

Using the responses to questions 19–27, various sub-groups of the respondents were identified. By means of the χ^2 contingency table analysis, it was found that the majority of the responses on the sensory characteristics of the apple rings were independent of demographic information. Those for which significant effects were found are discussed below. With seventeen two-way tables being considered for each of the factors, the probability of getting a significant result ($P<0.05$) by chance cannot be overlooked so such results should be treated with caution.

Questions 19 and 20 sub-divided respondents with respect to their frequency of eating fresh and cooked apples respectively. Those eating fresh apples most frequently preferred a more white and less sweet product than less frequent fresh apple eaters ($P<0.05$). However, frequent eaters of cooked apples appeared to be more satisfied with the colour of the apple rings than the less frequent eaters of cooked apples ($P<0.05$).

The age division was the factor to show the most effect on response patterns. As the numbers of respondents in the upper age groups were limited, the sub-divisions were reduced to four, i.e. 16–24, 25–34, 35–44 and >45. The 45 and older group responded more favourably to the appearance of the product than the youngest age group ($P<0.001$), and also to the yellow colour, with the rating getting progressively less favourable with decreasing age ($P<0.001$). The youngest age group (16–24) preferred a less sweet product than the older groups ($P<0.01$) and were also less inclined to be prepared to use the rings in a dessert ($P<0.05$). When comparing the Suntan rings with their recollection of Bramley's Seedling, the youngest age group showed less indication of any preference. The older groups, possibly because of greater familiarity with Bramley's Seedling, had a more definite preference for this latter cultivar ($P<0.05$). As mentioned previously, the results of this question must be treated with caution.

Some evidence could also be found that men were more favourably disposed towards the appearance of the slices than the women ($P<0.05$). Similarly, married respondents were also more favourably disposed to both the appearance ($P<0.01$) and colour ($P<0.001$) of the slices than single respondents. A greater proportion of the former were also prepared to use the

rings in a dessert ($P<0.05$) and they were more likely to state a distinct preference for Bramley's Seedling for culinary purposes ($P<0.01$).

Conclusions

The results indicate that apple rings prepared from Suntan apples usually received a favourable response from the 222 people interviewed, in particular with regard to appearance and taste. For many people, however, the texture could have been firmer. Responses were also, in general, independent of social class, area and type of environment in which people lived. The main demographic factor influencing the results was age, with the 45 and over age group being more in favour of the yellow colour and appearance of the product than younger people.

The data also indicates that scores were independent of sex and that older poeple seemed to prefer sweeter products than the 16–24 age group, both these results tying in with the general conclusions for wine and fruit juice reported in the literature (Amerine, Pangborn & Roessler, 1965).

How far responses to all questions were influenced by the presentation procedure and how the acceptance of the rings compared with that of other products cannot be estimated without similar test data on other products of known acceptance. In this context, direct comparison tests with the product of known acceptance as carried out when comparing fresh Suntan apples with Cox's Orange Pippin apples (Williams & Langron, 1983) would have provided more reliable data.

Despite the reservations, the results of question 17 seem to indicate that the majority of people, particularly amongst the older married have a preference for culinary purposes for at least their image of Bramley's Seedling. Whether this bias can truly be attributed to sensory properties or is a result of the monadic presentation needs to be checked by direct comparison experiments. It is also difficult to understand why respondents in this test indicated that they would prefer a firmer product yet still indicate preference for Bramley's Seedling, an apple which readily breaks down on cooking. Possibly other factors such as flavour, which has been shown to differ between the two varieties (Williams *et al.*, 1983), may be involved and carry more weight in making an overall judgement. The fact, however, that people liked the appearance of Suntan rings and were prepared to use them in products such as desserts, fruit salads and fritters, for which the Bramley's Seedling apple is not particularly suitable, may enable this new cultivar to fill a useful slot in the apple market.

Acknowledgment

The authors would like to thank Mrs J. F. Holmes, Mr P. S. Weaver and other members of the Scientific Liaison Section for their assistance both in preparing samples and for interviewing people during the course of the survey.

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(*Received 17 July 1982*)

J. Fd. Technol. (1983) **18**, 573–585

Influence of nitrite addition and gas permeability of packaging film on the microflora in a sliced vacuum-packed whole meat product under refrigerated storage

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Summary

Three batches of cured, smoked and cooked pork loins were prepared without nitrite and with 100 or 200 ppm nitrite. The product was sliced, vacuum packed and stored at 2°C, 5°C and 10°C. Part of the product was packed in a conventional film, a polyamide/polyethylene film, and part of it in two films with lower gas permeabilities. Nitrite inhibited the growth of *Brocothrix thermosphacta* and *Enterobacteriaceae* at all temperatures. This resulted in a lower growth rate and/or lower maximum counts. Reducing the gas permeability of the film in the absence of nitrite did not influence the growth of *B. thermosphacta*. An effect of increasing anaerobic conditions was, however, seen in conjunction with 100 ppm added nitrite at both 5°C and 10°C. The Gram-positive cocci were not affected by the addition of nitrite but were soon overgrown by the competing flora. The insensitivity of the lactic-acid bacteria to nitrite and microaerophilic conditions resulted in these bacteria constituting a larger proportion of the total flora in batches with nitrite and/or packaging films with reduced oxygen permeabilities.

Introduction

Studies of nitrite addition to vacuum-packed minced-meat products have shown different results with respect to its influence on the microflora. An examination of surface counts of vacuum-packed frankfurters stored at 4.5°C showed no effect of up to 156 ppm added nitrite (Simon *et al.*, 1973), while Hallerbach & Potter (1981) observed a difference in counts of aerobically stored frankfurters with or without 140 ppm added nitrite. The difference amounted to one log unit after 1 week's storage at 7–9°C. A somewhat improved keeping quality of nitrite-containing frankfurters compared to frankfurters made without nitrite was observed at 20°C by Bayne & Michener

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0022–1163/83/1000–0573 $02.00

(1975). While no effect of nitrite addition was observed during storage of sliced vacuum-packed, dry-cured ham (Kemp *et al.*, 1975), lower microbial counts were obtained during storage of Braunschweiger sausages produced with 156 ppm nitrite compared with sausages made without nitrite (Chyr, Walker & Sebranek, 1980). Similarly studies with nitrite addition to sliced vacuum-packed bacon have shown inhibition or no effect of added nitrite. Wood & Evans (1973) observed an effect of nitrite addition down to 20 ppm in bacon, and Hansen & Riemann (1962) found decreased lactobacilli counts and increased counts of micrococci by increasing the nitrite addition from 10 to 50 ppm. Shaw (1974) found no effect on total counts by increasing the nitrite content in vacuum-packed sliced bacon from 34 ppm to 150 ppm, while increasing the nitrite oncentration from 17 ppm to 144 ppm inhibited both total counts as well as lactic-acid bacteria. Increasing the nitrite addition to vacuum-packed bacon from 20 ppm to 120 ppm (6 and 35 ppm respectively in finished bacon) had negligible influence on total counts in a study by Wierbicki & Heiligman (1980).

In a previous study of vacuum-packed sliced Bologna-type sausage, the addition of nitrite was shown to have a profound influence on the growth of *B. thermosphacta* and *Enterobacteriaceae*, an effect which was greater with increasing nitrite levels and/or decreasing temperature (Nielsen, 1982a). The present study was undertaken to provide information on the effect of nitrite addition to a vacuum-packed whole meat product under normal storage conditions. In addition, the importance of oxygen permeability of the packaging film was studied.

Materials and methods

Pork loin production

Three batches were produced each consisting of three pork loins. The muscles were multi-stitch pumped with a pickle containing sodium chloride, polyphosphate, sucrose and with or without nitrite. The nitrite addition resulted in a concentration of 100 or 200 ppm nitrite in the pork loins. The pork loins were massaged for 20 min, then stored overnight under refrigeration. After a further massaging for 10 min, the muscles were stuffed in casings and smoked and cooked to a centre temperature of 75°C in a cabinet. The product was stored overnight at 4°C, sliced and vacuum packed in one of three films: (1) Rilotene, a polyamide/polyethylene laminate with an oxygen permeability of 52 ml/m^2/24 hr/1 atm at 75% r.h. and 25°C; (2) Mylothene S, consisting of a polyvinylidene chloride lacquered polyester film laminated to polyethylene with an oxygen permeability of 10 ml/m^2/24 hr/1 atm at 0% r.h. and 22°C; and (3) Lam-o-foil, consisting of a laminate of polyester/aluminium foil/polyethylene, the oxygen permeability of this film being extremely low. Only a minor part of the pork loins were packed in Mylothene S and Lam-o-foil and therefore this part of the experiment ended before the studies with Rilotene.

All films were supplied by Otto Nielsen A/S, Denmark. Packs with 0, 100 or 200 ppm added nitrite were stored at 2°C±1°C, 5°C±1°C and 10°C±1°C.

Sample preparation, bacteriological analyses, identification and chemical analyses were performed as previously described (Nielsen, 1982a).

Organoleptic analyses

Sensory analyses were done on the same packs as the microbiological analyses. Trained panellists were used in assessing odour and freshness. An 11-point hedonic scale was used (−5, bad, 0, neither good nor bad and +5, ideal). Between five and seven panellists participated in the nine sessions. Differences in colours between batches with and without nitrite were masked by serving the samples under red light. The results were analysed using linear regression, and 95% confidence intervals are given for the parameters in the regression equation (Helwig & Council, 1979).

Results

The results of the chemical analyses are shown in Table 1 and the initial microbial level in Table 2. Growth curves for *Brocothrix thermosphacta,* the lactic-acid bacteria and *Enterobacteriaceae* are also shown (Figs 1−5), and the composition of the total flora is shown in Tables 3 and 4. The curves for total counts are similar in shape to those of *B. thermosphacta* and are therefore not shown.

Table 1. Results of the chemical analyses of the whole meat product

Nitrite addition (ppm)	Salt (% w/w)	Water (% w/w)	Salt/water	Nitrite (ppm)	pH
0	3.6	73.0	4.9	0	6.0
100	3.5	71.8	4.9	59.8	6.1
200	3.8	74.4	5.1	134.1	6.1

Table 2. Initial numbers of microorganisms in the product

Micro-organism (/g)	Nitrite added (ppm)		
	0	100	200
Aerobic plate count	27*	39	34
Lactic-acid bacteria	27	10	16
B. thermosphacta	9	19	14
Gram-negative bacteria	< 25	< 25	< 5
Yeast	< 25	< 25	< 5

* Colony forming units/g.

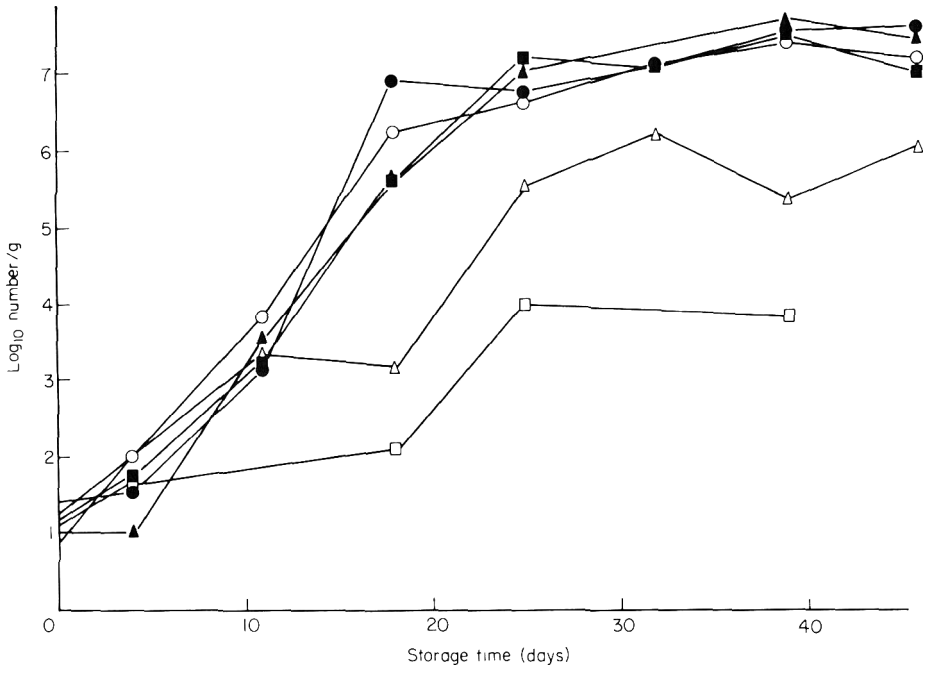

Figure 1. Growth of *B. thermosphacta* and lactic-acid bacteria at 2°C. ○, 0 ppm nitrite; △, 100 ppm; □, 200 ppm. Open symbols: *B. thermosphacta*, closed symbols: lactic-acid bacteria.

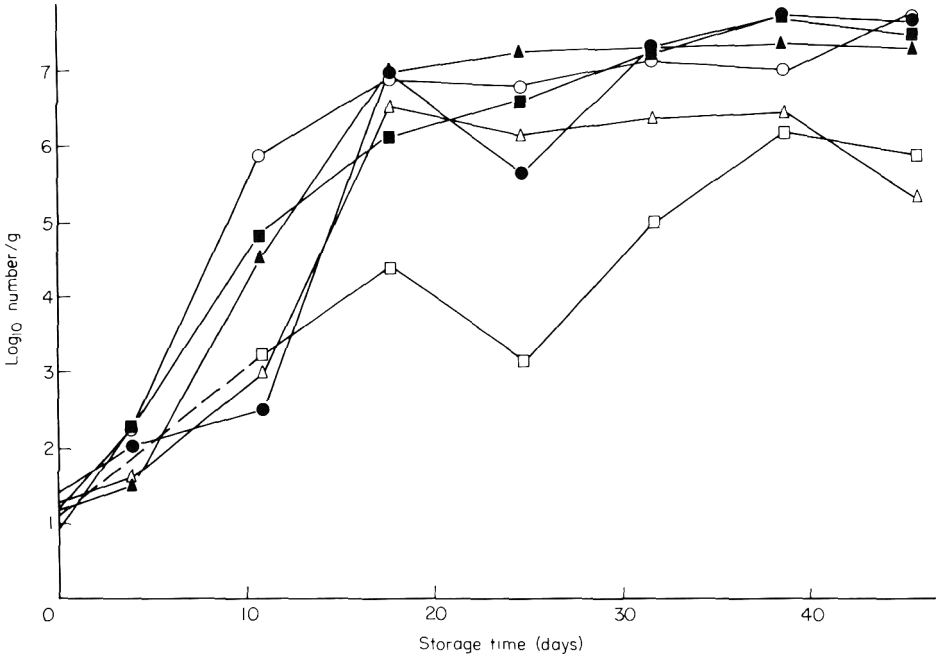

Figure 2. Growth of *B. thermosphacta* and lactic-acid bacteria at 5°C. ○, 0 ppm nitrite; △, 100 ppm; □, 200 ppm. Open symbols: *B. thermosphacta*, closed symbols: lactic-acid bacteria.

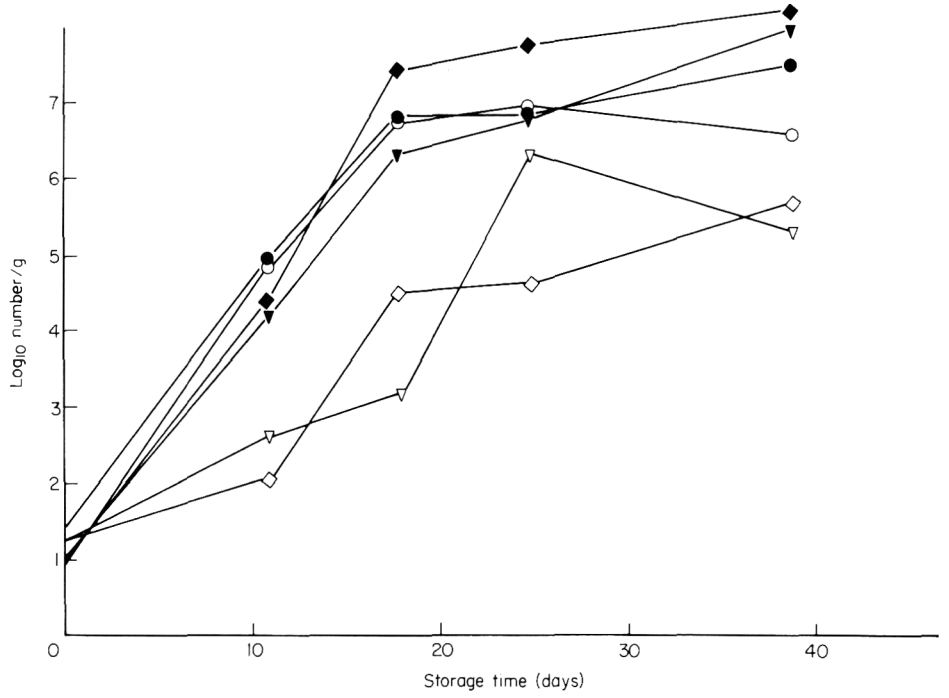

Figure 3. Growth of *B. thermosphacta* and lactic-acid bacteria at 5°C. ○, 0 ppm nitrite, Mylothene S; ▽, 100 ppm, Mylothene S; ◇, 100 ppm, Lam-o-foil. Open symbols: *B. thermosphacta*, closed symbols: lactic-acid bacteria.

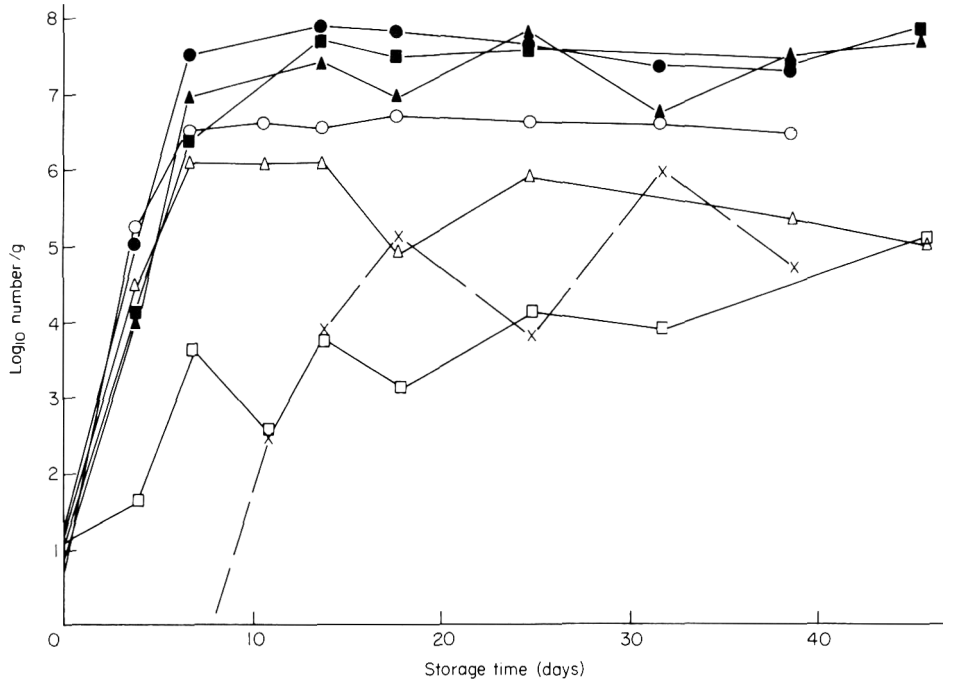

Figure 4. Growth of *B. thermosphacta*, lactic-acid bacteria and *Enterobacteriaceae* at 10°C. ○, 0 ppm nitrite; △, 100 ppm; □, 200 ppm. Open symbols: *B. thermosphacta*, closed symbols: lactic-acid bacteria. ×, 0 ppm nitrite *Enterobacteriaceae*.

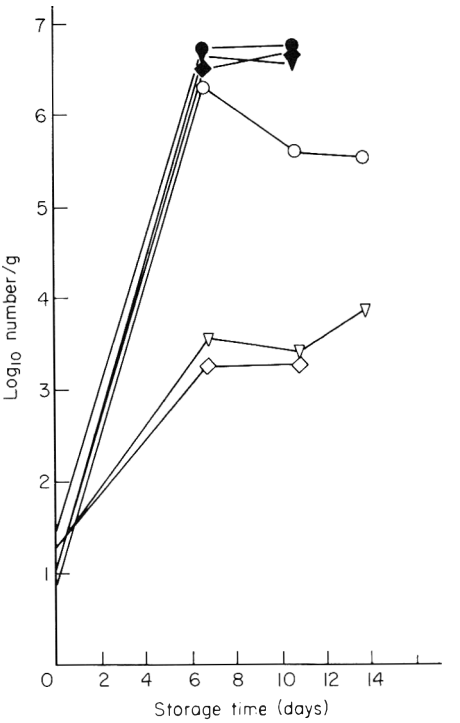

Figure 5. Growth of *B. thermosphacta* and lactic-acid bacteria at 10°C. ○, 0 ppm nitrite, Mylothene S; ▽, 100 ppm, Mylothene S; ◇, 100 ppm Lam-o-foil. Open symbols: *B. thermosphacta,* closed symbols: lactic-acid bacteria.

While the addition of 100 ppm nitrite did not influence the lag phase of the total aerobic bacteria (on Plate Counts Agar (PCA)) at 2°C, this being only a few days as in the batches produced without nitrite, 200 ppm of added nitrite extended the lag phase to about 3 weeks. Counts of approx. 10^6/g were obtained after approx. 2.5, 4 and 5.5 weeks storage in the product with 0, 100 and 200 ppm nitrite respectively. *B. thermosphacta* more or less constituted the total flora on PCA and was therefore influenced by the addition of nitrite as the aerobic plate count (APC) (Fig. 1). The influence of nitrite on the lactic-acid bacteria was small, counts of approx. 10^7/g being reached in all batches, without nitrite after 2.5 weeks and with nitrite after 3.5 weeks. The numbers stayed between 10^7/g and 10^8/g during the rest of the storage period. No growth of *Enterobacteriaceae* was seen at 2°C, but the numbers of *Moraxella*/*Moraxella*-like bacteria increased to 10^3-10^4/g after 4–6 weeks' storage, irrespective of nitrite addition. The pH value of the product in the vacuum packs did not change during storage at 2°C.

In spite of their numbers at the beginning of the storage period the *Micrococcaceae* were very rapidly overgrown by *B. thermosphacta* and the lactic-acid bacteria (Table 3). On the other hand the proportion of *B. thermosphacta* was less in batches with nitrite than in the product without added nitrite at any time during storage. The lactic-acid bacteria became the dominating group irrespective of nitrite addition.

Table 3. Composition of microbial flora in vacuum-packed sliced, cured and cooked pork loin during storage

		Nitrite added (ppm)																	
		0						100						200					
°C	Days	A*	B	C	D	E	F	A	B	C	D	E	F	A	B	C	D	E	F
2	0	37	15	44			4	17	46	25			12	30	26	31			13
	4		74	26				96		4				41	4	54			
	11		83	17					38	62						99			
	18		63	37						98	2					100			
	25		41	59					33	67						100			
	32		50	50					10	90						100			
	39		41	59					1	99					1	99			
	46		19	81					2	98						100			
5	4	9	56	32			1	94	3	3						100			
	11		68	32					2	98					2	98			
	18		45	56					16	84					2	98			
	25		93	7					4	96						100			
	32		18	82					2	85	13					100			
	39		13	87					6	94					1	99			
	46		28	72						100					3	97			
10	4		62	38					72	28				4	1	96			
	7		9	91					12	88				9		90		1	
	11		8	92					3	97						99			1
	14		3	97					3	97						99	1		
	18		4	96					1	99						100			
	25		5	95					1	99						100			
	32		3	97					5	95						100			
	39		4	96					1	99						100			
	46									100						100			

* (%): A, *Micrococcaceae*; B, *B. thermosphacta*; C, lactic-acid bacteria; D, atypical *Vibrio*, E, *Moraxella/Moraxella* like bacteria; F, 'other micro-organisms'. The isolates A, D, E and F were obtained from PCA, B from Streptomycin-Thallous Acetate-Actidione Agar, C from APT when possible for the competing flora, otherwise from the selective NAP.

Apart from a longer lag period (approx. 1 week) the increase in APC at 5°C was very much the same with addition of 100 ppm nitrite as in the product produced without nitrite, while the addition of 200 ppm resulted in a reduced growth rate throughout the storage period. Counts of approx. 10^6/g were reached in approx. 2, 2.5 and 5.2 weeks in the product produced with 0, 100 and 200 ppm nitrite respectively. *B. thermosphacta* again constituted the major part of the aerobic plate count on PCA (Fig. 2). The lactic-acid bacteria on the other hand were only affected to a limited extent as in the products stored at 2°C. In all batches, counts of about 10^7/g were obtained (Fig. 2) The *Enterobacteriaceae* did not grow at all at 5°C while the *Moraxella/Moraxella*-like bacteria increased in numbers in the batches without nitrite only, to 10^3-10^4/g within 2 weeks.

Table 4. Composition of microbial flora in sliced, cured and cooked pork loin vacuum packed in Mylothene S or Lam-o-foil film

		Mylothene S (0 ppm nitrite added)				Mylothene S (100 ppm nitrite added)				Lam-o-foil (100 ppm nitrite added)			
°C	Days	A*	B	C	D	A	B	C	D	A	B	C	D
5	0	37	15	44		17	46	25	12	30	26	31	13
	11		41	58	1		3	97			1	99	
	18		29	71				100			3	97	
	25		57	43			25	75				100	
	39		9	91				100				100	

A* (%): A, *Micrococcaceae*; B, *B. thermosphacta*; C, lactic-acid bacteria; D, 'other bacteria'.

The lactic-acid bacteria eventually dominated in all batches at 5°C (Table 3), and at an earlier stage in products produced with nitrite. Lactic-acid bacteria became the total flora in the product with 200 ppm nitrite after only 4 days, this was not the case until after approx. 3.5 weeks in the absence of nitrite.

Decreasing the gas permeability of the packaging film, i.e. using Mylothene S *versus* Rilotene, had no effect on the APC in product without nitrite, while addition of 100 ppm nitrite partially inhibited growth in Mylothene S compared with Rilotene. The even less permeable film Lam-o-foil further inhibited growth. In fact using Lam-o-foil and 100 ppm nitrite resulted in the same bacterial increase as in the packages of Rilotene with the addition of 200 ppm nitrite. The same situation holds for *B. thermosphacta* (Fig. 3). In contrast to this the lactic-acid bacteria were not affected by any combination of gas permeability and nitrite addition (Fig. 3).

Increasing the nitrite addition and/or decreasing the film permeability resulted in *B. thermosphacta* constituting a minor and the lactic-acid bacteria a major proportion of the total flora (Tables 3 and 4).

Only small differences in pH values between packages were recorded during storage, from pH 5.8–6.0.

Adding nitrite resulted in a slightly lower growth rate and a lower maximum total plate count at 10°C. The maximum counts in the product without nitrite were approx. 10^7/g, with 100 ppm nitrite 10^6/g and with 200 ppm 10^5/g. measuring the total counts by All Purpose Medium with Tween (APT), however, resulted in almost the same growth curves irrespective of nitrite addition (curves not shown). This is of course caused by the inclusion of the lactic-acid bacteria in the count and these bacteria were not affected by the addition of nitrite. On nitrite actidione polymyxin agar (NAP) (Davidson & Cronin, 1973) the lactic-acid bacteria counts were up to 10^7–10^8/g after 2 weeks (Fig. 4). Addition of 100 ppm nitrite had only a minor affect on *B. thermosphacta* at 10°C, with maximum counts approx. 0.5–1.0 $\log_{10}$ unit lower than in the product without nitrite. Addition of 200 ppm nitrite caused a considerable inhibition of *B. thermosphacta* at 10°C, after 3 weeks the counts

were still less than 10^4/g rising to 10^5/g after 6 weeks (Fig. 4). Addition of nitrite completely inhibited growth of *Enterobacteriaceae,* while the numbers reached 10^5/g after about 3 weeks' storage in product without nitrite (Fig. 4). The numbers of *Moraxella*/*Moraxella*-like bacteria increased to approx. 10^3/g after 2 weeks whether nitrite was present or absent, but no further propagation was observed after that time. Some growth of atypical *Vibrio* spp. was seen in all products. These bacteria were counted on PCA in which the sodium chloride (4% w/v) makes conditions for growth satisfactory. The *Vibrio* spp. could be counted on crystal violet kanamycin agar (Gardner, 1973) but this medium is not sufficiently selective to exclude the growth of *Enterobacteriaceae.* In all batches the atypical *Vibrio* spp. reached 10^5/g regardless of nitrite addition. After a couple of weeks, however, there bacteria could not be isolated in the batches with nitrite even on the selective medium, while they were observed during the whole 6 weeks' storage period in the product made without nitrite. The yeasts increased in numbers irrespective of nitrite addition to approx. 10^3-10^4/g within 2–3 weeks.

The lactic-acid bacteria eventually dominated in all products at 10°C. This happened after only a few days in the batches with 200 ppm nitrite and after approx. 1 week in the two other batches (Table 3). The domination of the lactic-acid bacteria was more evident using APT than by counting them on the selective medium NAP. The former media often gives counts of approx. 1 $\log_{10}$ unit higher than the latter. The differences were caused by chain-forming cocci, mostly *Leuconostoc mesenteroides* or *Leuc. dextranicum.* Only a few of these bacteria isolated from APT grew on NAP.

The counts on PCA were very strongly depressed by the addition of 100 ppm nitrite when using Mylothene S as packaging film (Fig. 5). After 2 weeks, the numbers were below 10^4/g, while the batches packed in Rilotene reached 10^6/g within a week. Further reducing the oxygen permeability by using Lam-o-foil did not enhance the nitrite inhibition. The lactic-acid bacteria were not affected by the packaging film (Fig. 5). No growth of *Enterobacteriaceae* or other Gram-negative bacteria was seen in the packages with lower oxygen permeabilities.

The lactic-acid bacteria very soon dominated in the batches packed in Mylothene S, and at an arlier stage than in the series where Rilotene was used as the packaging material. Complete dominance was seen after 7 and 11–14 days' storage in batches prepared with 100 ppm and without nitrite respectively (data not shown).

Organoleptic results

Linear regression of the results from the organoleptic assessments of odour (not shown) and freshness (Table 5) showed a linear decrease of these parameters with time and a trend to a faster deterioration in batches produced without nitrite. A disadvantage of the organoleptic analyses was the difference between the red colour of the nitrite-containing batches and the grey colour of

Table 5. Regression analyses of organoleptic assessment of freshness at 2 C, 5 C and 10 C for Rilotene packages

Temperature		Added nitrite (ppm)		
		0	100	200
2°C	a*	−0.07 ± 0.02	−0.05 ± 0.01	−0.05 ± 0.01
	b	1.39 ± 0.63	1.34 ± 0.46	1.47 ± 0.28
	R(n)†	−0.95‡	−0.95‡	−0.98‡
5°C	a	−0.08 ± 0.01	−0.05 ± 0.02	−0.05 ± 0.01
	b	1.62 ± 0.43	1.23 ± 0.59	1.51 ± 0.55
	R(n)	+0.98‡	−0.91‡	−0.93‡
10°C	a	−0.10 ± 0.05	−0.08 ± 0.02	−0.07 ± 0.01
	b	0.84 ± 1.12	1.46 ± 1.12	1.30 ± 0.47
	R(n)	−0.92‡	−0.94‡	−0.97‡

* Parameters in the equation, 'freshness = a days + b', 95% confidence intervals are shown.

† R = correlation coefficient, n = number of sessions (n = 7 for the series at 10 C without nitrite; for all other series, n = 9).

‡ Significant at 0.1% level.

the product without nitrite; it could not be eliminated completely by using red light at the evaluation sessions.

Discussion

The influence of nitrite addition on the growth of *B. thermosphacta* resembled the results obtained in the study of Bologna-type sausage (Nielsen, 1982a). At all temperatures the nitrite exerted an inhibitory effect which increased with increasing nitrite concentration. Contrary to the results obtained with the minced-meat product (Nielsen, 1982a), *B. thermosphacta* grew at 2°C with 200 ppm nitrite, but the numbers did not exceed 10^4/g. Addition of nitrite resulted in an increased lag phase and/or lower growth rates for *B. thermosphacta,* which therefore constituted lower proportions of the total flora in these batches. Using a packaging film with a lower oxygen permeability in comparison with Rilotene had no effect in the meat produced without nitrite. The combination of a lower permeability and nitrite addition, however, exerted a larger inhibition of *B. thermosphacta* at both 5°C and 10°C than nitrite alone. These results are in agreement with experiments on the influence of nitrite addition on *B. thermosphacta* under aerobic and anaerobic conditions in APT broth (Nielsen, 1982b) and with studies on total counts of vacuum- and non-vacuum-packed sliced bacon with 120 or 170 ppm added nitrite (Herring, 1973).

The Gram-positive cocci were hardly affected by nitrite addition had therefore made up a larger proportion of the total flora in batches with added nitrite; they were, however, quickly overgrown by other bacteria in all

products. It may be pointed out that no sodium ascorbate was added to the product in this experiment and that addition causes a faster depletion of nitrite (Bem, Hechelmann & Ramming, 1973) so that no effect of nitrite was observed (Simon *et al.*, 1973). However, in other studies where sodium ascorbate was added, an effect of nitrite has been noted (Hallerbach & Potter, 1981).

Addition of nitrite had only limited influence on the development of the lactic-acid bacteria and the same maximum counts were obtained whether nitrite was added or not. Increasing the temperature resulted in an earlier dominance of the lactic-acid bacteria and so did an increased nitrite addition. Decreasing the gas permeability of the film resulted in the lactic-acid bacteria constituting a larger proportion of the total flora although the effect was small. This might result in a better organoleptic quality of the product (Qvist & Mukherji, 1979) although this was not examined in the present study.

The effect of nitrite addition on *Enterobacteriaceae* may be surprising. Leistner, Hechelmann & Uchida (1973a, b) did not find any influence of 50–100 ppm added nitrite in 'Brühwurst' on the growth of a mixture of *Enterobacter- Klebsiella* at 5°C or 8°C, but Roberts, Britto & Schroff (1979) did observe an inhibition by 100 and 200 ppm nitrite compared with 50 ppm in broth cultures. Moreover the salt/water ratio is of importance for the growth of the *Enterobacteriaceae* (Hechelman, Bem & Alberts, 1974, Nielsen, 1982b) and the salt concentration in the present study was higher than in that of Leistner *et al.* (1973a, b). These bacteria made up mostly of *S. liquefaciens* and *E. aerogenes,* may be present in large numbers in vacuum-packed cured meat (Nielsen, 1982b) and not only during the later stages as quoted by Hechelmann *et al.* (1974b). They proliferate at a rate comparable with *B. thermosphacta* and may have just as much influence on the organoleptic quality of the product. An inhibition of these bacteria in some batches in the present study may be a combination of several factors, salt, nitrite, microaerophilic conditions and may be lactate concentration in combination with low initial counts. A lactate concentration of approx. 5–7 mg/g meat during storage is not uncommon (Nielsen, 1982b); with the present water content this corresponds to a concentration of approx. 76–106 mM in the water phase. Grau (1981) found growth of *S. liquefaciens* (the bacteria found in the present study) at 150 mM lactate at pH 6.1 under anaerobic conditions in broth culture. In that study there was, however a higher initial count (10^2–10^3/ml), a temperature of 25°C and no competition by other bacteria. In the present study, with lower initial counts and lower temperature as well as competition by other bacteria, the lactate might still be a factor of some importance.

As far as could be seen from countable plates the atypical *Vibrio* spp. and the *Moraxella*/*Moraxella*-like bacteria were unaffected by the nitrite addition. The atypical *Vibrio* (non-motile and sensitive towards penicillin) are quite often present in vacuum-packed cured meat products in high numbers (Gardner, 1980, Gardner & Patton, 1971, Nielsen, 1982b). Gardner (1980) found these bacteria in packs with sulphide spoilage and more than half of the isolates produced H_2S. Contrary to this, nearly all of the isolates in the present study

and in those of Nielsen (1982b) were sulphide negative, and had probably only limited influence on the organoleptic quality. Observation of *Moraxella*/*Moraxella*-like bacteria in vacuum-packed meat are few, but they proliferated in vacuum-packed fresh lamb during storage (Patterson & Gibbs, 1978, Shaw, Harding & Taylor, 1980), in high pH meat (beef) (Patterson & Gibbs, 1977) and in some of the experiments by Shaw & Harding (1978). That these bacteria were not found in packages of Mylothene S possibly reflects the lower gas permeability of this film in comparison with Rilotene.

The growth of yeasts was not influenced by the addition of nitrite, but highly affected by storage temperature. These organisms were only very seldom discovered at 2°C and 5°C but proliferated at 10°C unaffected by the nitrite concentration. This is in agreement with results by Hansen & Riemann (1962) with vacuum-packed bacon.

Differences in pH value were generally negligible between batches with and without nitrite and packed in Rilotene or Mylothene S. A lower pH value in a high barrier film in comparison with a normal barrier film has been observed (Amundson *et al.*, 1982). This results in a faster depletion of the added nitrite and might therefore enhance the growth of nitrite-sensitive bacteria.

In summary the study showed that the addition of nitrite to a vacuum-packed whole meat product exerted a profound effect on some important spoilage organisms, notably *B. thermosphacta* and the *Enterobacteriaceae*. The inhibition of these bacteria was reflected in a slower deterioration of the product in nitrite-containing products. In combination with low storage temperatures and/or use of packaging films with low oxygen permeabilities, nitrite is a significant additive in controlling the spoilage of vacuum-packed cured meat.

Acknowledgment

The research was supported by the Danish Council for Scientific and Industrial Research.

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Prediction of moisture gain and loss for packaged pasta subjected to a sine wave temperature/humidity environment

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Summary

The loss or gain of moisture through a package depends on the sorption isotherm of the food material, the conditions of temperature and relative humidity at which it is stored, and the water vapour permeability of the packaging material. The water vapour permeability (WVP) characteristics of paperboard, polypropylene, and polyethylene used for pasta were determined at 30 to 45°C and from 11 to 85% r.h. Both temperature and relative humidity affected the water vapour permeability of all the three films. Paperboard showed a greater WVP at 30°C compared to 45°C due to a higher moisture content which swells the matrix. Thus, it did not follow the typical Arrhenius relationship of a higher permeability at higher temperature. Polyethylene and polypropylene, on the other hand, showed a higher WVP as the temperature was increased. A second degree polynomial was fitted to predict the WVP of paperboard as a function of the ambient conditions. It was found that the creases and end openings on a typical paperboard box increase the overall transmission rate by two times over that of paperboard alone, therefore reducing the shelf life to half under adverse conditions.

A dynamic mathematical model to predict mixture transfer for pasta packages under controlled unsteady state conditions of temperature and r.h. was developed and tested; good predictions were obtained.

Introduction

Current dietary trends suggest an increase of complex carbohydrate intake with a consequent decrease in the consumption of refined sugars and foods from the meat and egg group of the basic four. These suggested changes could lead to low

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0022–1163/83/1000–0587 $02.00

intakes of essential nutrients such as the B vitamins if the replacing foods do not supply them in adequate amounts, either because the foods are naturally low in the specific nutrients or have significantly lost these nutrients during storage and distribution.

In this nutritional context and in a hungry and economics-conscious world, pasta would appear to present an ideal solution to several problems because of its unique combination of low price, palatability, versatility, ease of preparation, nutritive value (especially when enriched), and long shelf life (Antognelly, 1980). But pasta with a low water activity must be protected by packaging in such a way that it does not gain moisture. An increase in water activity increases the rate of loss of nutrients with the additional possibility of reaching an unsafe water activity which could permit the growth of microorganisms. Pasta also must be protected from moisture loss since below an a_w of 0.4 it becomes fragile. In addition, the package could become illegal to sell if the net weight is lower than the one declared on the label.

A food product can either lose or gain moisture through the package depending on its water activity, the condition of temperature and %r.h. at which it is stored, and the permeability of the film used. Oswin (1945a, b, 1946) developed solutions for prediction of moisture transfer in a packaged product under steady state conditions of constant external temperature and humidity. Heiss (1958) discussed the relationship between the moisture sorption properties of foods, the film permeability with respect to water vapour, and the shelf life of the product and developed a solution for constant temperature and humidity based on Fick's law of diffusion. Work on this area was further carried out at the Massachusetts Institute of Technology (Karel, Proctor & Wiseman, 1959; Karel & Labuza, 1969; Karel, 1967). Karel and his co-workers modified Heiss' mathematical models introducing computer solutions (Mizrahi, Labuza & Karel, 1970) and simpler mathematical models (Labuza, Mizrahi & Karel, 1972). Other studies have generated similar solutions for predictions of moisture transfer during storage of both foods and drugs (Veillard *et al.*, 1979; Lockhart, 1980; Peppas & Khanna, 1980). All of these works have basically used the same concepts originally introduced by Oswin (1945a, b, 1946), Heiss (1958) and Karel, Proctor & Wiseman (1959), utilizing studies done at constant humidity and temperature. No studies with food have been published on moisture transfer under unsteady state conditions simulating a real world situation where both temperature and humidity continuously vary. Work of this type was carried out for moisture gain/loss in packaged drugs by Nakabayashi, Shimamoto & Mina (1980). Reasonable agreement was found between predicted and actual values.

Assuming a linear isotherm for the enclosed food, Mizrahi, Labusa & Karel (1970) have shown that the gain or loss of moisture under constant environmental conditions is given by:

$$\ln \frac{m_e - m_i}{m_e - m} = \pm \frac{k}{x} \frac{A}{W_s} \frac{p_0}{b} \theta \qquad (1)$$

where:
m_e= moisture content of the food at equilibrium with the environment from the linear isotherm (dry basis);
m_i=initial moisture content;
m=moisture at time θ;
b=slope of the moisture sorption assuming it is linear—($m=b(a_w)+I$) where I is the intercept and b is the slope;
k/x=film water vapour permeability (WVP) in g H_2O/day m^2 mm of Hg;
A=area;
p_0=vapour pressure of pure water at temperature T of the environment; and
θ=time.

This equation assumes that k/x and b do not change with the environmental conditions which for paperboard and pasta is not true. For example, Labuza & Contreras (1981) showed that the WVP of plastic films varied with temperature and humidity.

The objective of this study was to develop and test a dynamic model for prediction of the gain or loss of moisture in pasta packaged in typical packaging materials under conditions of variable temperature and relative humidity, simulating those that might be found under actual storage and distribution systems.

Materials

Packaging materials

Folding cartons supplied by the Creamette Co. (Minneapolis, Minnesota) were used: these packages are currently manufactured by Champion International Corp. (St Paul, Minnesota). The cartons are made of 1.2 mm thick clay-coated newsback paperboard with dimensions of 18.5×10.00×3.6 cm. Boxes were stored at room temperature and 50% r.h. (average) before being tested. For sealing the ends of the boxes, a borated dextrin type glue (AJAX 7344-1) was used: this glue is manufactured by AJAX Adhesive and Chemical Co., Inc. (Chicago, Illinois). The flexible films chosen for this study were a 2 mil. low density polyethylene and a 1.6 mil. laminated polypropylene. These films were also obtained from the Creamette Co. (Minneapolis, Minnesota).

Methods

1. Permeability at constant temperature and r.h.

(a) Paperboard. The gravimetric method of Heiss (1958) as modified by Davis *et al.* (1960) was used to determine the WVP of complete packages. Paperboard boxes were filled with a known amount of pre-dried desiccant to maintain the package area to weight of solids ratio as in actual consumer pasta packages (3570 g of solids/m^2). The ends of the boxes were glued in the manner

in which pasta manufacturers seal them (i.e. open spaces at the upper and bottom seals: the open spaces at the end seals were covered with a stretched piece of pantyhose nylon in order to prevent physical loss of desiccant without building a barrier for moisture exchange.

Twing Albert cups (Philadelphia, Pa) were used to determine the permeability of paperboard. This permitted the separate determination of the WVP of the complete package and that of the material itself. Thus, it was possible to evaluate the effect of the creases and the poor end seals on the moisture transfer rate.

Paperboard is a hygroscopic material and either absorbs or gives off moisture. This can introduce error, depending on the humidity used, when moisture transmission rates are measured (Martin, 1962). Thus, in order to prevent errors in weight change, all the paperboard packages with one end opened, before being tested for permeability, were preconditioned using TAPPI standard T402 OS-70. It should be noted that the TAPPI (Technical Association of Pulp and Paper Industry) official test method for water vapour transmission rate (WVTR) determinations of sheet materials at normal and high temperature and humidity (T448 SU-71, 1941; T464 OS-79, 1973) state that no preconditioning of the materials is necessary. Open boxes and specimens of the appropriate dimensions to be used in the Twing Albert cups were first placed in a sealed chamber at 11% r.h. (saturated $LiCl_2$) at room temperature for 48 hr. The samples were then conditioned to each r.h. at which the permeability value was to be obtained. The time of exposure was determined for each humidity condition by weighing the specimens several times (~2 hr periods) until no differences greater than 0.01 g were found

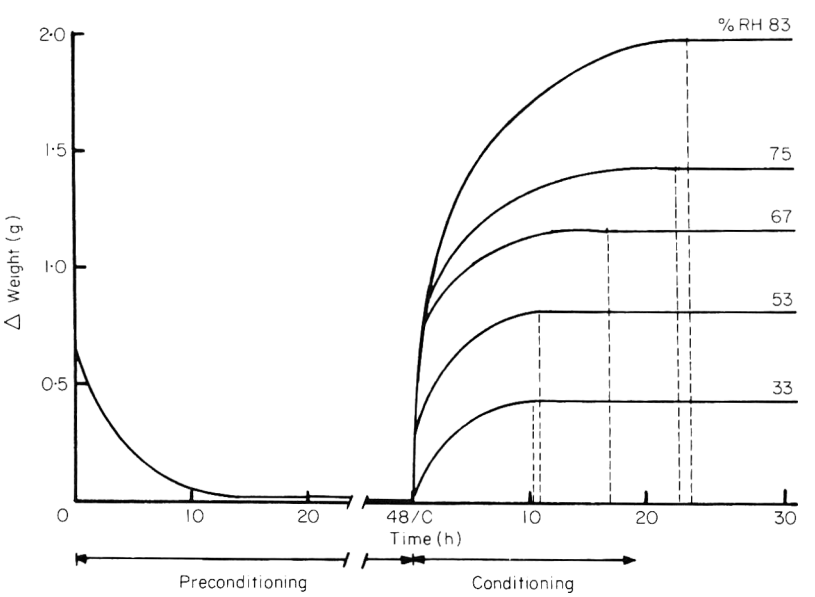


Figure 1. Preconditioning times at various % relative humidities for paperboard prior to permeability testing.

between subsequent weighings. The results are shown in Fig. 1. Basically, after 24 hr the boxes reached equilibrium at all relative humidities. It should be noted that a significant error would have occurred in the determination of the k/x value if preconditioning had not been done.

Once the samples had reached equilibrium, the boxes were filled with desiccant, sealed, the initial weight was recorded and they were transferred to the different temperatures and relative humidities (30, 37 and 45°C and 11% to 84% r.h.). Subsequent weighings of these packages were done every 2–4 hr in order to obtain the permeability data during the period of weight gain while keeping the weight gain of the desiccant within a 10% limit. The test was repeated three times. In addition, sheets of pre-equilibrated film were tested in the Twing Albert cups (Davis *et al.*, 1960).

(b) Flexible films. For each film, bags (9×9 cm) were filled with desiccant. The bags were sealed, weighed, and then stored in the same controlled r.h. chambers as for the paperboard. The weight *versus* time was recorded for triplicate samples of each film.

(c) Permeability calculations. Weight *versus* time were plotted for each of the packaging materials tested. By linear regression, the slope of the line was

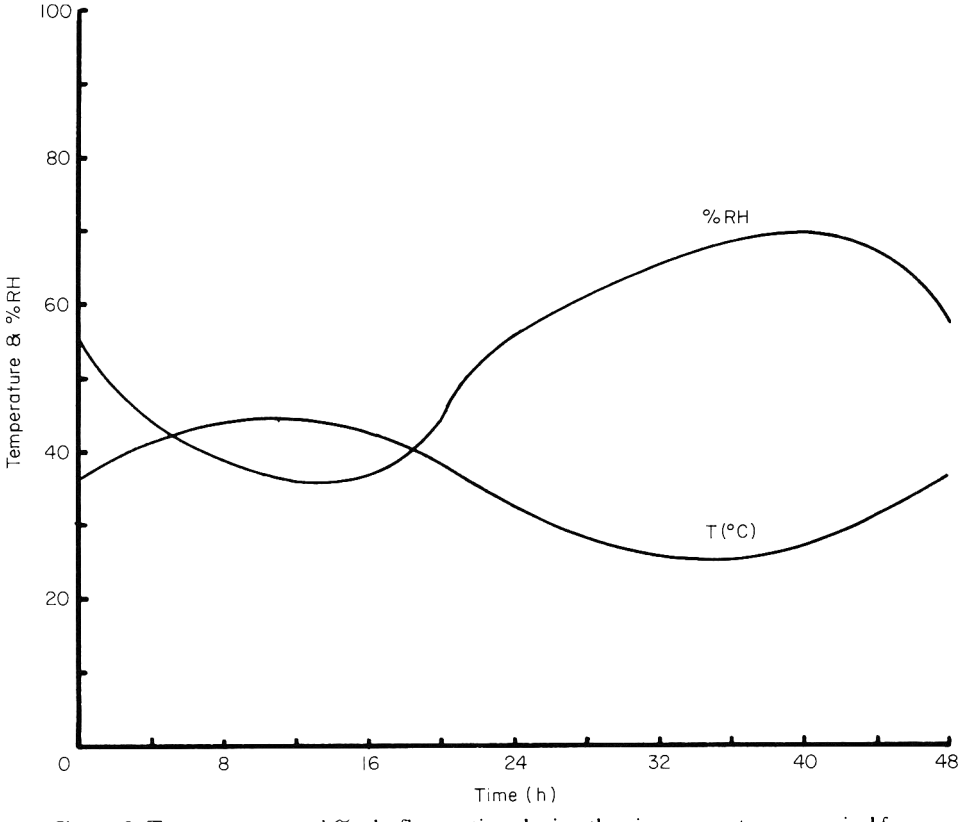


Figure 2. Temperature and %r.h. fluctuation during the sine wave storage period for pasta packages.

determined, and, from this k/x is:

$$\frac{k}{x} = \frac{\text{slope}}{(A)(P_{out} - P_{in})} \quad (2)$$

where:
P_{out}=outside vapour pressure: and
P_{in}=0=vapour pressure inside the package.

2. Isotherm determination

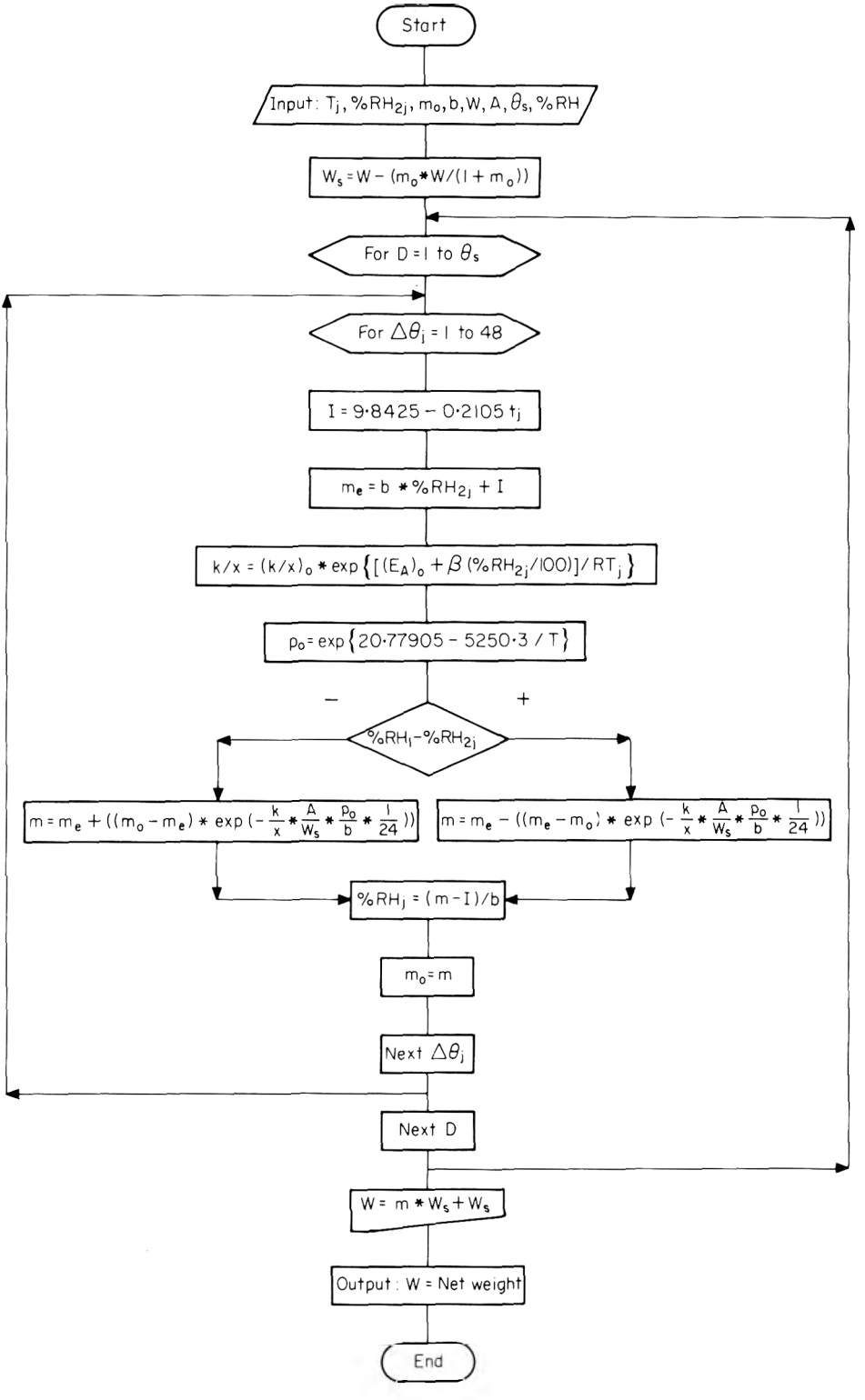
Isotherms were determined for pasta (25 and 45°C) and paperboard (30, 37 and 45°C) at each r.h. used above for the k/x determinations. The pasta was prepared on a laboratory extruder and dried in a laboratory drier. It was ground for 30 sec in a Micro-Mill manufactured by Technilab Instruments (*Pesuannock,* NJ) before testing. After 40 days the moisture content at each condition was determined by the Karl Fischer method

3. Evaluation of moisture gain or loss under conditions of variable temperature and r.h.

Triplicate samples of pasta packaged in the different packaging materials were stored in a walk-in environmental temperature and humidity-controlled chamber. The dry bulb temperature was cycled from 25 to 45°C by a sine wave scheme within a 48 hr period. The wet bulb temperature was kept constant at 30°C, thus the relative humidity cycled between 30 and 60% r.h. Samples were weighed periodically and actual moisture gain or loss was compared to the predicted gain or loss using a computer iterative technique. Figure 2 shows the actual storage condition to which the packages were subjected.

Actual gain or loss in weight was compared to the predicted values throughout the time of storage. To do this, equation 1, the kinetic data of WVP

Figure 3. Flow diagram of computer iterative calculation for net weight variation where: T=temperature at interval of time j (K); t=temperature at interval of time j (C); r.h._{2j}=outside relative humidity, given for each time interval j; $\text{r.h.}_{.j}=(a\times 100)$ of the product at any time; p_0=vapour pressure at temperature T, given for each time interval; m=initial moisture content of product at time interval j (the initial value is given); b=slope of the linear isotherm (given); I=intercept of the linear isotherm (function of T, given); θ_s=shelf life time or time after which the net weight of the product wants to be calculated (days); W=net weight of the product at any time (g); A=total surface area of the package (m^2); D=number of days, from 1 to end of shelf life; W=weight of solids in the product (g); k/x=WVP of the packaging materials (g of water/day m^2 mmHg); $(k/x)_0$=constant for each film; E_A=activation energy for each film (kcal/kmol); E_0=extrapolated activation energy at 0% outside relative humidity; β=slope of E *versus* %r.h. plot; R=gas constant (1.987 cal/mol K); m_e=moisture of the product in equilibrium with the outside environment (g H_2O/g of solids); and m=moisture of the product at any time (g H_2O/g of solids).


Start
Input: T_j, $\%RH_{2j}$, m_o, b, W, A, θ_s, %RH
$W_s = W - (m_o * W/(1 + m_o))$
For D = 1 to θ_s
For $\Delta\theta_j$ = 1 to 48
$I = 9{\cdot}8425 - 0{\cdot}2105\, t_j$
$m_e = b * \%RH_{2j} + I$
$k/x = (k/x)_o * \exp\{[(E_A)_o + \beta(\%RH_{2j}/100)]/RT_j\}$
$p_o = \exp\{20{\cdot}77905 - 5250{\cdot}3/T\}$
−
+
$\%RH_1 - \%RH_{2j}$
$m = m_e + ((m_o - m_e) * \exp(-\frac{k}{x} * \frac{A}{W_s} * \frac{p_o}{b} * \frac{1}{24}))$
$m = m_e - ((m_e - m_o) * \exp(-\frac{k}{x} * \frac{A}{W_s} * \frac{p_o}{b} * \frac{1}{24}))$
$\%RH_j = (m - I)/b$
$m_o = m$
Next $\Delta\theta_j$
Next D
$W = m * W_s + W_s$
Output: W = Net weight
End

of the packaging materials at different conditions of temperature and r.h., and the isotherm as a function of T and %r.h. were used.

This calculation was carried out as follows:

(a) The external conditions (T, %r.h.) at which the packages had been held were controlled and recorded at all times during the storage period (Fig. 2).
(b) The change in moisture after a certain time of storage was calculated by dividing the total time into 1 hr intervals.
(c) For initial time, the %r.h., T, and m_i are known.
(d) For that segment of time, the vapour pressure, p_0, of pure water was calculated by using the following equation:

$$\ln p_0 = 20.77905 - 5250.31/T = \text{mmHg} \quad (3)$$

where:

T=absolute temperature (°K).

Equation 3 was obtained by multiple regression using tabulated data of vapour pressure of water *versus* temperature in the range of 25–45°C. A correlation coefficient of 1 was found.

(e) An m_e was obtained from the isotherm for the external temperature and %r.h. at that time. If m_e is less than m_i, the loss equation was used; if it was greater than m_i, the gain equation was used to calculate m.
(f) This procedure was then repeated using the new m as the new initial m_i.

This calculation was made more simple and accurate by the use of a computer iterative technique. A flow diagram to calculate the net weight of the product after any period of time when held at known fluctuating temperature and r.h. conditions is presented in Fig. 3. A program to perform these calculations was written in the BASIC language and the outputs were compared with the experimental result. The program is available from the authors.

Results and discussion

Water vapour permeability as a function of temperature and r.h. in paperboard

Table 1 shows the average k/x values obtained for the complete paperboard packages. Table 2 shows those obtained for flat sheets of paperboard specimens by the Twing Albert cup method. One of the advantages of using the actual package to determine its permeability value is that this represents not only the k/x value of the material but also a composite value including the folding and sealing effects which in the case of paperboard packages are important. These tables show the large differences found between the WVP for paperboard specimens as measured by the cup method and the complete commercial package of the same material at similar conditions. On the average, the creases and end openings more than doubled (2.2) the permeability of the box. Although the effect of creases on the WVP of different materials (especially wax-coated paperboard) has been evaluated (Minifie, 1970) and tests for this

Table 1. WVP of paperboard boxes (k/x (g H_2O/day m^2 mm Hg))

	% Relative humidity (r.h.)					
T (°C)	11	32	52	67	75	85
30	7.58	7.87	11.03	12.28	16.04	16.90
37	10.78	11.01	11.56	10.53	14.82	15.98
45	11.59	10.57	10.00	12.24	12.11	12.39

Table 2. WVP of paperboard specimens (Thwing Albert cup method) (k/x (g H_2O/day m^2 mmHg))

	% Relative humidity (r.h.)					
T (°C)	11	32	51	67	75	84
30	4.95	4.71	5.60	6.47	7.03	6.64
37	3.72	4.52	4.11	6.12	7.51	6.44
45	4.94	3.98	5.13	5.10	5.44	5.48

purpose have been developed (British Standard Packaging Code, TAPPI Office Test Methods T533-PM-76, 1976; T512 OM-81, 1981), no reference has been found in the literature which addresses the effects of both the creases and openings left at the ends of the packages. Because of the number and types of creases, it is impossible to separately evaluate the crease effect from the end-opening effect for a whole box. These results show that the use of a liner with lower permeability (e.g. as in cereal breakfast packages) would significantly improve the shelf life of pasta in paperboard boxes.

The effect of temperature and r.h. on paperboard WVP

In order to analyse the behaviour of the material as a function of temperature, the log WVP *versus* the reciprocal of the absolute temperature was plotted at constant r.h. An example is shown in Fig. 4. The permeability values obtained indicate that paperboard behaves differently than most plastic films, in that it does not follow the typical Arrhenius plot with a negative slope. At humidities above 50%, paperboard shows a higher permeability value at the lower temperature tested (30°C) than at the higher temperature. For the whole box over the range of 11–85% r.h. the k/x is more than doubled at 30°C, increased by 50% at 37°C while increased by 10% at 45°C.

These observations suggest that the water sorption characteristics of paperboard play an important role in the WVP properties of the material. The moisture sorption isotherm of paperboard (Fig. 5) shows that at relative humidities of 30% and higher the material holds less moisture at lower than at

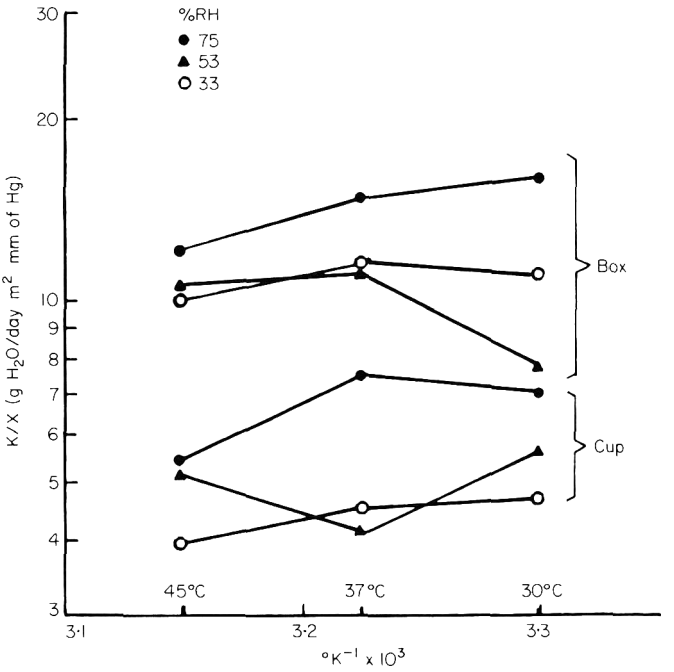


Figure 4. WVP for paperboard as a function of temperature and relative humidity measured for the whole box and by the cup method.

higher temperatures. Corte (1958) and Field (1973) showed that the mean pore size in paperboard increases over the range of 0–10% moisture content. An increased mean pore size should result in a higher diffusion rate through the pores since diffusion is in part proportional to the square of the pore radius when laminar flow is occurring. Theoretically, permeability is dependent both on the diffusivity (which increases with increasing temperature and increased pore size) as well as the solubility or concentration of the permeant in the film (Paine, 1962). Solubility of the vapour decreases with increasing temperature. Thus, an increased temperature causes both an increase and decrease in WVP. The magnitude of the pore size change, however, is greater and thus results in the non-Arrhenius-type behaviour of Fig. 4.

When the typical WVTR (g/day m^2) value is considered for the whole box (Fig. 6), it can be seen that the values increase as temperature and r.h. increase. A typical exponential type of relationship can be observed. However, when k/x values are used, the water vapour permeation through the material is corrected for the vapour pressure differential. At the higher temperature and relative humidities, paperboard has the higher vapour pressure differential which lowers the k/x value. Since WVTR values are not corrected for the vapour pressure differential, they cannot be used for shelf life studies under unsteady state external conditions (Mizrahi, Labuza & Karel, 1970). In order to solve for moisture gain/loss under variable conditions, the results for the whole box were fitted to a second degree polynomial using MULTREG (Weisberg, 1979).

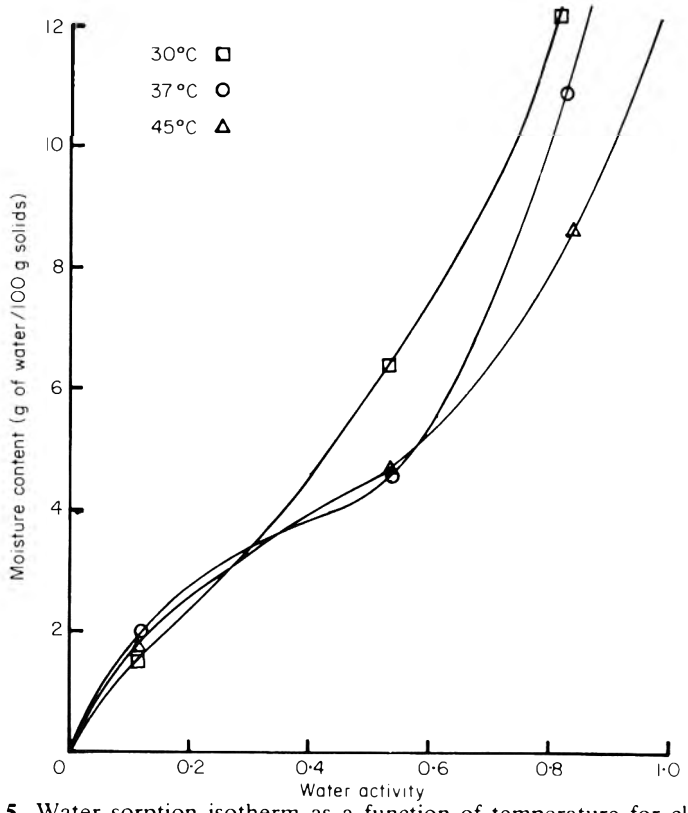


Figure 5. Water sorption isotherm as a function of temperature for clay-coated newsback paperboard.

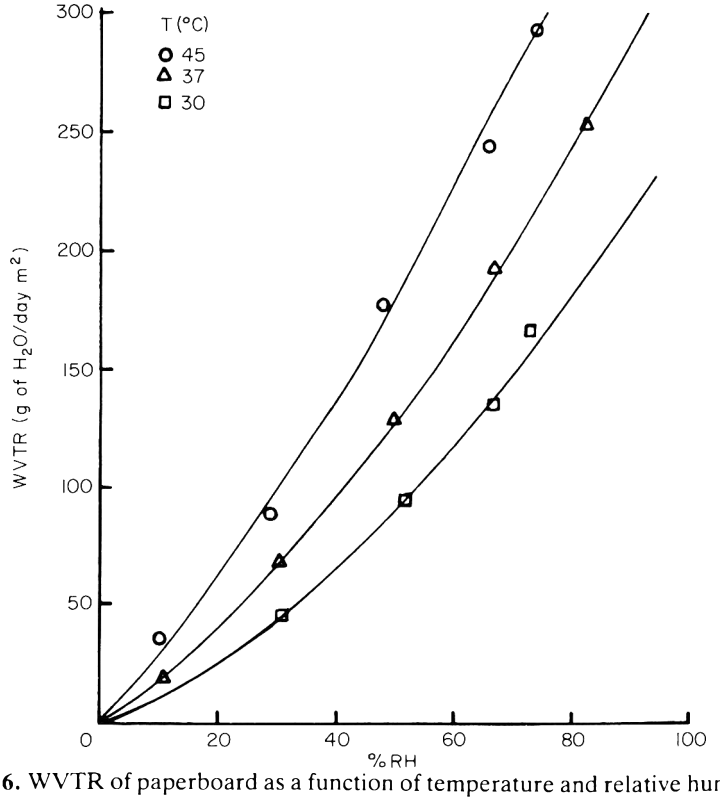


Figure 6. WVTR of paperboard as a function of temperature and relative humidity.

Equation 4 was found:

$$k/x = 2470.173 - 15.89532\ T + 99.91809\ \%\text{r.h.} + 0.0256059 \times 10^{-1}\ T^2 + 0.8456451 \times 10^{-3}\ \%\text{r.h.}^2 - 15446.17\ \%\text{r.h.}/T - 0.1616512\ \%\text{r.h.} * T \quad (4)$$

with $r^2 = 0.87$
where:
k/x = water vapour permeability (g of water/day m^2 mm of Hg);
T = absolute temperature (K); and
%r.h. = outside percent relative humidity.

The WVP obtained for polyethylene and polypropylene are presented in Tables 3 and 4. Figure 7 shows the typical Arrhenius relationship for polypropylene. These results confirm a similar behaviour found by several workers (Doty, Aiken & Mark, 1946; Hauster & McLaren, 1948; Kumins, Rolle & Rotman, 1957; Karel, Proctor & Wiseman, 1959; Labuza & Contreras, 1981). The activation energy of permeation (E_A) increased as the r.h. decreased as shown in Fig. 8 ($r^2 = 0.95$). Hilton (1957) suggested that the log of WVTR for plastic films *versus* r.h. results in a straight line for values of r.h. over 30%. Karel, Proctor & Wiseman (1959) confirmed this relationship to be true for polyethylene but this behaviour was not found for other films tested. Labuza & Contreras (1981) found a linear relationship when E_A *versus* (a_w − %r.h./100) was plotted on linear paper; however, an empirical relation between activation

Table 3. WVP for polyethylene at different temperatures and relative humidities (k/x (g H_2O/day m^2 mmHg)

	% Relative humidity (r.h.)					
T (°C)	11	32	53	67	75	83
30	0.187	0.196	0.201	0.221	0.228	0.231
37	0.143	0.239	0.232	0.195	0.228	0.222
45	0.336	0.347	0.262	0.263	0.247	0.252

Table 4. WVP for polypropylene at different temperatures and relative humidities (k/x (g H_2O/day m^2 mmHg) × 10^1)

	% Relative humidity (r.h.)					
T (°C)	11	32	53	67	75	83
30	0.473	0.476	0.445	0.471	0.516	0.709
37	0.517	0.522	0.495	0.540	0.505	0.517
45	0.893	0.825	0.664	0.645	0.631	0.532

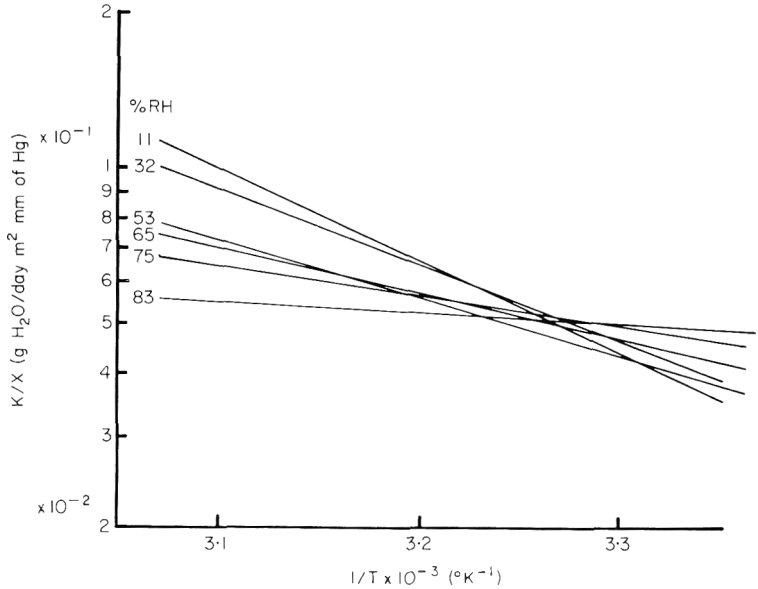


Figure 7. WVP of polypropylene as a function of temperature and %r.h. plotted as an Arrhenius function.

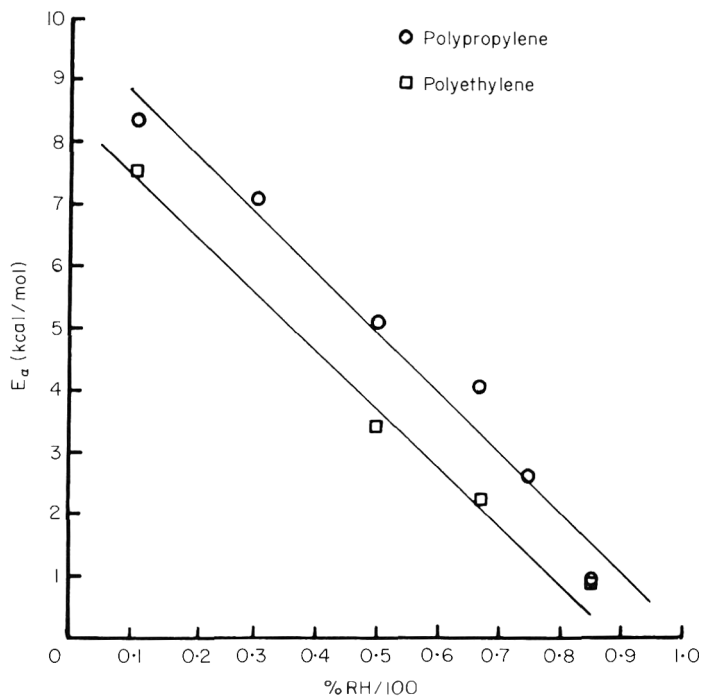


Figure 8. Activation energy for water vapour permeation through plastic films as a function of %r.h.

energy and the outside r.h. for polyethylene and polypropylene better fit the data where:

$$E_A=(E_0)+\beta\,(\%\text{r.h.})/100 \tag{5}$$

where:
E_A=activation energy of permeation (cal/mol);
(E_0)=extrapolated activation energy of permeation when outside %r.h. is zero (cal/mol);
β=slope when E_A is plotted *versus* outside %r.h./100; and
%r.h.=outside relative humidity.

The overall k/x is thus:

$$k/x=(k/x)_0 \exp-[(E_0)+\beta(\%\text{r.h.}/100)]/RT \tag{6}$$

where:
$(k/x)_0$=permeability pre-exponential constant (different for each external r.h.);
R=universal gas constant (1.987 cal/mol K); and
T=temperature. (All the other terms as previously defined.)

The values found from the experimental data for β were -9.547 for polyethylene and -9.802 for polypropylene. The $(k/x)_0$ values extrapolated from the experimental data are presented in Table 5. In order to correlate these values with %r.h., least squares computation was performed on the data, equation 7 for polyethylene and 8 for polypropylene were obtained with correlation coefficients of 0.99 and 0.96 respectively.

$$(k/x)_0=12.259-1.547\times10^{-1}\,\%\text{r.h.} \tag{7}$$

$$(k/x)_0=13.191-1.605\times10^{-1}\,\%\text{r.h.} \tag{8}$$

By using equations 6, 7 and 8, it is possible to calculate the k/x of the flexible films at any combination of temperature and r.h. These equations were used in the dynamic model to predict moisture transfer in pasta packages. The equations were included in the iterative computer calculation.

Table 5. Extrapolated $(k/x)_0$ constant for plastic films

% r.h.	Polyethylene	Polypropylene
11	10.788	10.609
32	—	8.663
53	4.003	5.393
17	2.144	3.612
75	0.229	1.333
83	−0.114	−1.504

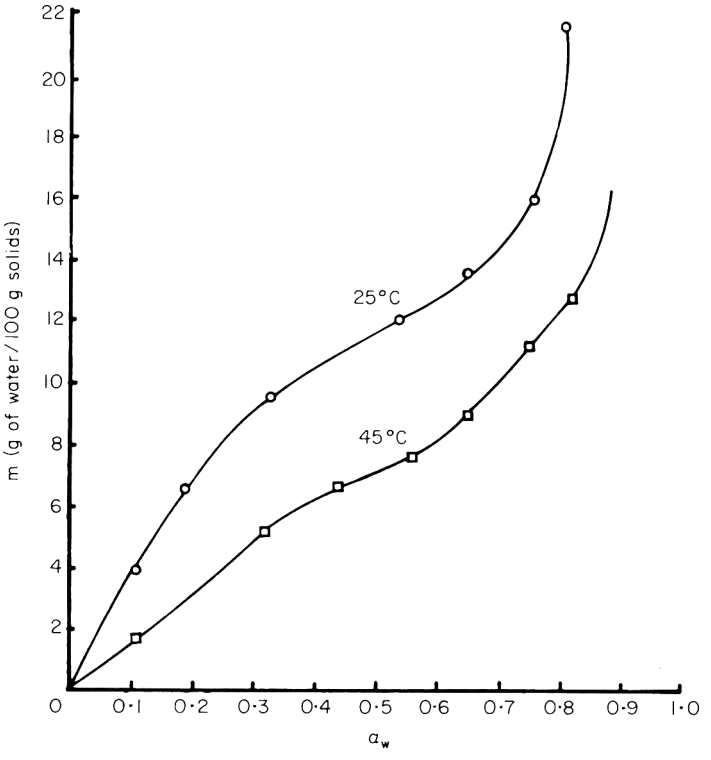


Figure 9. Water sorption isotherm of egg noodles at two temperatures.

The water sorption isotherm for pasta

Figure 9 shows the moisture sorption isotherm for pasta at 25°C and 45°C. The values are presented in Table 6. From Fig. 9 it can be observed that a considerable shift occurs in the isotherm at the higher temperature. In a closed environment such as a package of a food, when the temperature is raised the amount of water will be relatively constant but the water activity will be considerably higher.

Table 6. Moisture sorption isotherm data for pasta. (m (g H_2O/100 g solids)

a_w	25°C	45°C
0.11	3.9	1.7
0.32	9.5	5.2
0.44	10.7	6.6
0.56	12.0	7.6
0.65	13.6	8.9
0.75	15.9	11.2
0.83	21.5	12.8

It could be said that by using the isotherm obtained at 25°C for moisture gain calculations, a safety factor has been introduced as the water activity inside the package will be considered lower than it actually is; thus, an overprediction of moisture gain will occur. However, the opposite effect will be true for moisture loss. Thus, it was important in this study to consider this effect for the calculations of moisture gain/loss under the fluctuating conditions of temperature and r.h., as the low outside relative humidities are associated with the higher temperatures (Fig. 3). In order to calculate the moisture gain/loss, both the slope (b) and the m_c value are needed from the isotherm.

The slope for pasta in this study was found to be the same (b=0.141) for the 25 and 45°C isotherms which are the temperature limits in this study. To get m_c in addition to the slope (b), the intercept I is needed. A linear relationship was found for the intercept as a function of temperature:

$$I=9.84-0.21\ T \tag{9}$$

where:
I=intercept of the linear isotherm=g H_2O/100 g solids; and
T=temperature of the outside environment (°C).

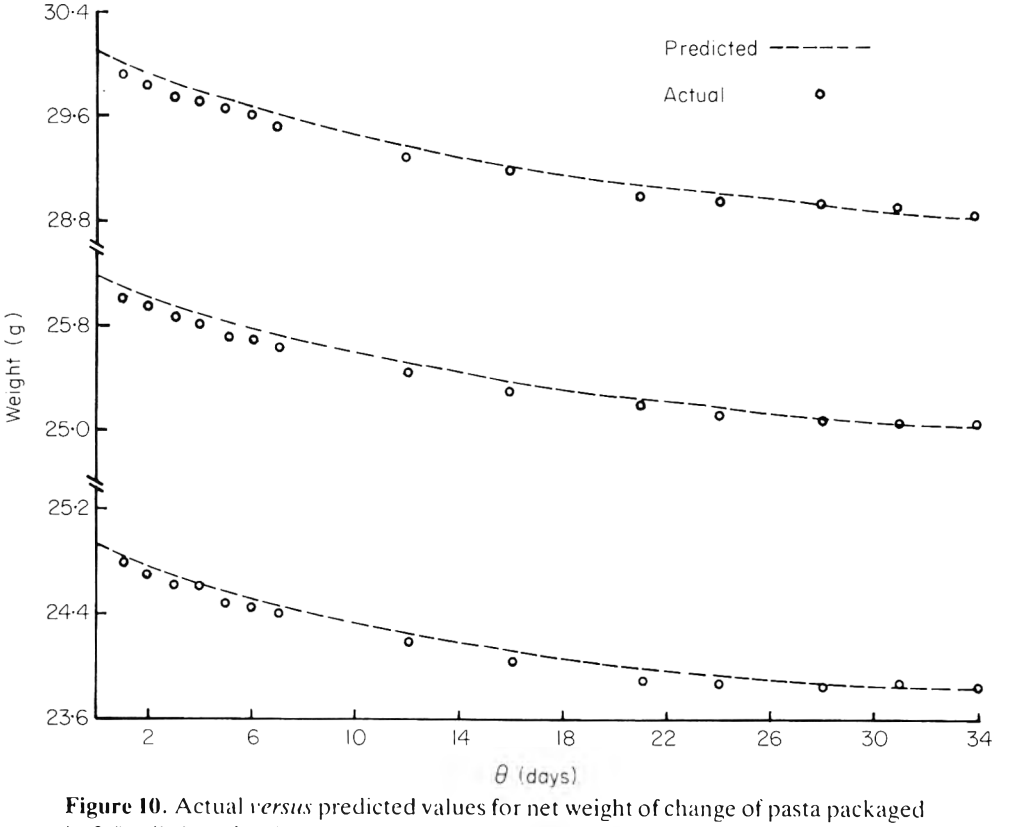


Figure 10. Actual *versus* predicted values for net weight of change of pasta packaged in 2.0 mil. low density polyethylene bags held in a sine wave condition.

Moisture transfer under variable temperature and r.h. conditions

A computer program written in the BASIC language was used to perform iterative calculations of moisture gain or loss in pasta under the unsteady state conditions of temperature and r.h. as previously described. Time intervals of 1 hr were used over a 34 day storage period using the two flexible films with the same size bag as before, containing between 25 and 30 g of pasta. The flow diagram for this program is presented in Fig. 3.

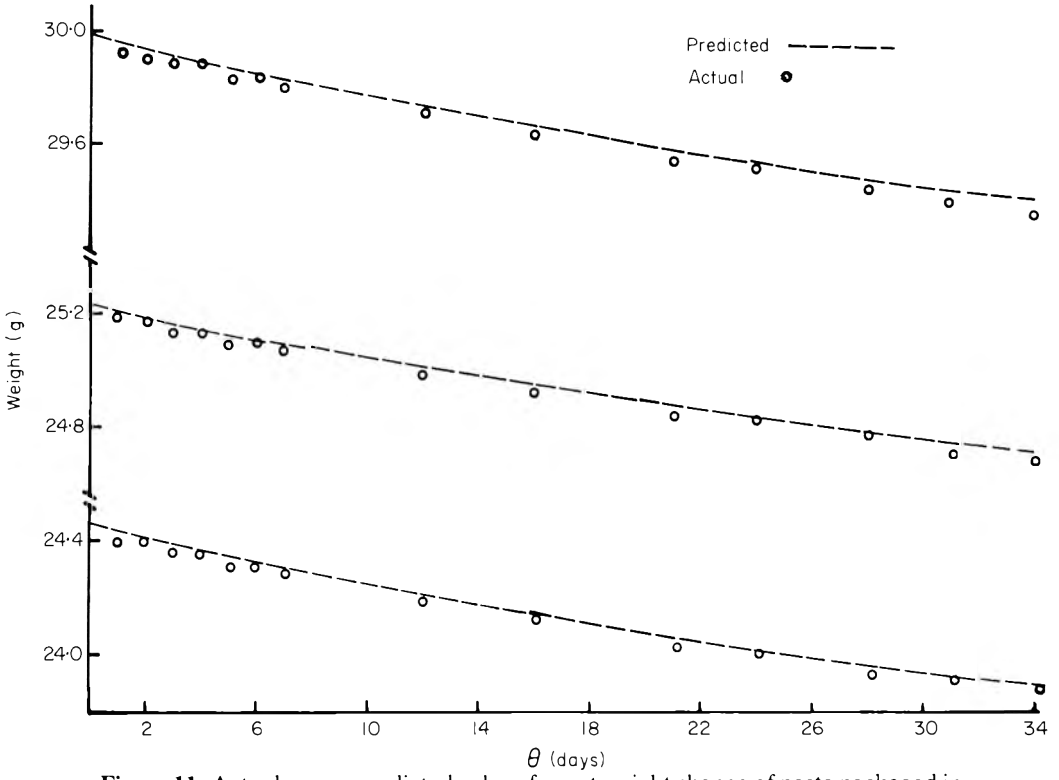


Figure 11. Actual *versus* predicted values for net weight change of pasta packaged in 1.6 mil. laminated polypropylene bags held in a sine wave condition.

Figures 10 and 11 present a comparison between the actual and predicted values. There was no significant difference (P=0.01) using Student's t test for all cases suggesting that the mathematical model used in this study predicts well the net weight variation in pasta packaged in flexible films, and the program could be applied to most dehydrated products whenever the variable storage conditions are known. This information may be used to determine the end of shelf life for the product if the rate of any reaction leading to unacceptability is known as a function of water activity and temperature. It should be noted that only the calculated value at the end of each 48 hr period was used in the plot and measured values were taken at the middle or the end of a full cycle. Since the driving force for loss was greater (larger ΔP difference), this results in a net overall loss although if the plot were expanded to show a 24 hr period the data

would follow an irregular sine wave pattern with a net decrease. It can be noted that the actual data do show a rise and fall when 24 hr intervals are used.

A similar storage study was done with whole paperboard boxes. The results were not as good, as shown in Fig. 12, with the actual net weight lagging behind the predicted weight and varying in a sine wave over a period of 6 days. This same pattern was followed for the whole 34 days. In Fig. 12 the graph is

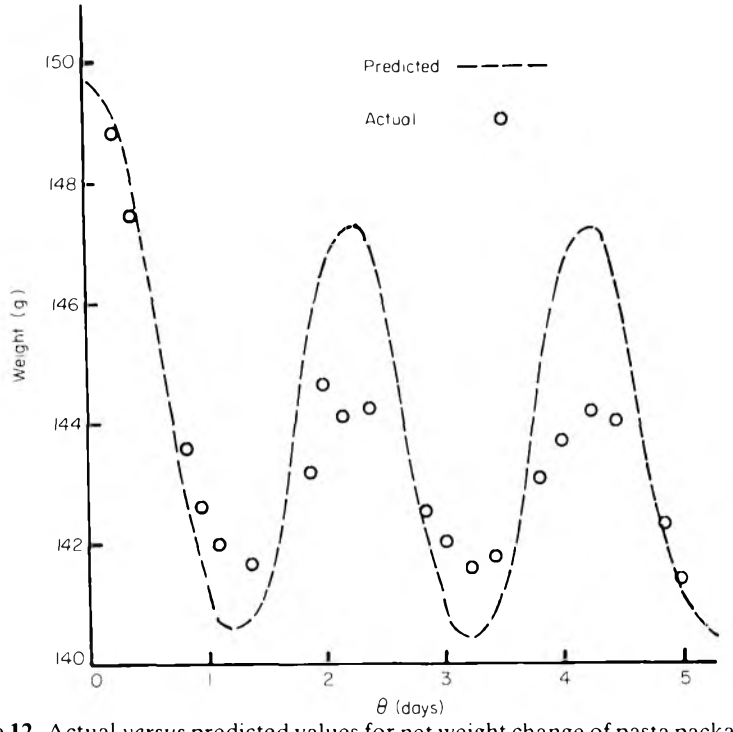


Figure 12. Actual *versus* predicted values for net weight change of pasta packaged in paperboard boxes held in a sine wave condition.

expanded to show the actual rise and fall in the 48 hr period which is confirmed by the actual data. In addition, the peaks of each wave decrease as the driving force decreases for moisture gain. The difference in predicted *versus* actual values is probably due to the fact that equation 1 assumes no internal resistance to moisture transport, that is, all the resistance is in the film and moisture equilibrates with the food instantly. In the short cycle used, however, the diffusion of moisture into the pasta must be limiting to some degree so the theoretical model is not true. In addition, the paperboard may not be quickly responding to the varying temperature and r.h. conditions and, thus, the k/x value used at each instant of time in the program may not be correct. A longer term study with a slower cycling of T and r.h. is needed.

This part of the study demonstrated that the principles and assumptions involved in the derivation of the mathematical model are somewhat valid, and that good simulations of net weight variations in packaged dehydrated products can be made by using a computer iterative technique.

Acknowledgments

This paper is scientific journal series No. 13136 from the University of Minnesota Agricultural Experiment Station. This project was supported in part by University of Minnesota Agricultural Experiment Station Grant Nos. 18–72 and 18–78, the University of Minnesota Computer Center and NSF Grant No. PFR 7910370 from the National Science Foundation. Mr Cardoso was supported in part by the Fulbright Commission through the Institute of International Education.

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(Received 26 October 1982)

J. Fd Technol. (1983) **18**, 607–617

Muscle fibre orientation and its effect on measurements of tenderness of bovine longissimus dorsi muscle

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Summary

The muscle fibre orientation within a particular cooked beef steak was found to vary considerably and to be markedly affected by vacuum packaging, freezing and/or cooking. Shear force values, initial-bite tenderness (panel) and cooking loss were highly dependent on, and overall tenderness (panel) was only slightly dependent on muscle fibre orientations. These measures were affected by fibre orientation to a greater extent in tough than in tender meat. At each of three fixed muscle fibre orientation, correlations between tenderness measures were significant ($P<0.05$), their magnitude depending on the fibre orientation. At a fixed fibre angle correlations of tenderness measures with cooking losses were not significant. However, on a within-animal basis, cooking loss was related to shear value and to initial-bite tenderness apparently through an effect of fibre orientation. This effect may constitute a major source of variation in the measurement of tenderness and cooking loss.

Introduction

Meat cores for the determination of Warner–Bratzler shear force values are usually obtained from cooked steaks in one of two methods—by coring parallel to the longitudinal axis of the muscle fibres or by coring perpendicular to the surface of the steak (AMSA, 1978). Francis *et al.* (1981) concluded, contrary to the earlier findings of Hostetler & Ritchey (1964), that for the longissimus dorsi (LD) muscle, the two methods result in identical shear values. This may indicate that the muscle fibre orientation need not be carefully considered and controlled when carrying out shear force determinations, but such is not the case. The first method above results in shearing muscle fibres at 90° to their longitudinal axis. With the second method the angle at which the muscle fibres are sheared depends upon their orientation in

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0022–1163/83/1000–0607 $02.00

the steak after cooking and this has been shown to dramatically affect shear force values (Murray & Martin, 1980).

The present study was initiated to ascertain the extent to which the orientation of the muscle fibres at the time shearing or biting influences: (1) the objective and subjective estimations of tenderness of the LD muscle and (2) the relationships which exist among shear force values, sensory measures of tenderness and cooking loss.

Materials and methods

Three steers, from each of three breed composites (100% Simmental, 75% Simmental and 75% Limousin) were assigned to each of three animal groups and were slaughtered at approximately 15 months of age and 430 kg live-weight. The longissimus dorsi (LD) muscle, from the fifth thoracic to the sixth lumbar vertebra, was removed from the right side of carcasses which had been aged for 6 days. Each muscle was then divided into three portions approximately equal in length which were designated locations 1 to 3, 1 indicating the anterior and 3 the posterior end of the muscle. In order to replicate muscle fibre angles equally across all locations, steaks were cut from locations 1, 2 and 3 at three angles (0°, conventional, 90°) with respect to the longitudinal axis of the muscle fibres in a manner indicated in Table 1. The 'conventional' angle was that obtained by cutting a steak perpendicular to the longitudinal axis of the muscle. Four 3 cm steaks were cut at each location. After trimming of fat the steaks were vacuum packaged, frozen and stored at −30°C for up to 2 months.

Steaks were thawed at 1°C and cooked at 177°C to an internal temperature of 75°C in a convection oven. Cooking losses were determined by weighing steaks before and after cooking. The steaks were then stored overnight in plastic bags at 1°C.

Two steaks from each location were used for shear force and panel assessment by normal procedures. Six 2 cm cores were removed from each steak with an Eberbach cork boring device by coring perpendicular to the steak surface. Three were assigned at random for shear force determination; the remaining three were used for panel tenderness evaluation. Cores were sheared or bitten (initial-bite tenderness) perpendicular to their longitudinal

Table 1. The angles at which steaks were cut with respect to the orientation of the muscle fibres within muscle location and animal group

Muscle location	Animal group		
	1	2	3
1	0°	Conventional	90°
2	Conventional	90°	0°
3	90°	0°	Conventional

axis with no attempt to control the angle of rotation about this axis. Shear values obtained by this approach are referred to subsequently as 'normal'.

The remaining two steaks were used to test the effect of the degree of rotation of the cores at time of presentation to the mechanical shear device. Four lines were drawn on the surface of each steak, two approximately parallel and two approximately perpendicular to the longitudinal axis of the steak (Fig. 1). The lines were alternately parallel and perpendicular with no attempt being made to distinguish between the medial and lateral ends of the steak. Four cores were removed as above such that the lines remained on the end of each core. They were then sheared perpendicular to their longitudinal axis after rotation of the cores so as to shear perpendicular to the lines. This resulted in shear force values either 'parallel' or 'perpendicular' to the longitudinal axis of the steak.

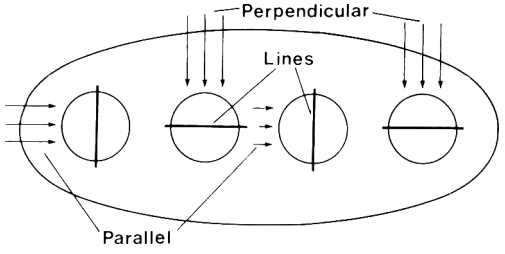

Figure 1. Diagrammatic representation of a steak to show how lines drawn on its surface were used to orient cores for shearing (arrows) either parallel or perpendicular to its longitudinal axis.

All tenderness estimates were made approximately 20 min after removing the cores from refrigeration temperatures according to AMSA guidelines (AMSA, 1978). A six-member trained panel was utilized to evaluate initial-bite tenderness (the perceived force required to bite through a core with the incisors) and overall tenderness on scales of 1–9 (1=extremely tough, 9=extremely tender). Shear force values were obtained using an Ottawa Texture Measuring System fitted with a Warner–Bratzler head (L'Hirondelle & Martin, 1975).

Total collagen and % salt-soluble collagen (Hill, 1966) were determined by the method of Woessner (1961) using meat samples from five locations along the LD muscle of the right side of the carcass. Intramuscular fat concentration of the LD muscle between the thirteenth rib and the sixth lumbar vertebra of the left side of each carcass was determined on a dry weight basis using a Soxtec (Tectator Co.) fat extraction apparatus.

The 'initial' angle of the muscle fibres with respect to the steak surface was estimated visually while cutting the raw steaks. The 'final' fibre angle was determined after coring and dissection of cooked steaks and involved four or five measurements per steak with a protractor.

Results and discussion

'Muscle fibre angle' is used subsequently to refer to the angle between the longitudinal axis of the muscle fibres and the steak surface, or, since cores were removed at 90° to the steak surface, to the angle between the longitudinal axis of the muscle fibres and the plane at which the shear blade or the teeth of the panelist penetrated the core.

Table 2. Simple means and standard errors of final fibre angle, tenderness measures and cooking loss at the three initial fibre angles

	Initial fibre angle					
	0°		Conventional		90°	
	Mean	s.e.	Mean	s.e.	Mean	s.e.
Final fibre angle (degrees)	5.4a	0.5	35.3b	1.0	51.8c	1.1
Shear force value (kg)						
normal	4.4a	0.1	6.6b	0.3	7.1b	0.3
perpendicular	4.8a	0.1	6.9b	0.3	7.2b	0.3
parallel	4.2a	0.1	6.5b	0.3	6.8b	0.3
perpendicular–parallel	0.6a	0.1	0.4b	0.2	0.4b	0.1
Initial-bite tenderness	7.3a	0.2	5.1b	0.3	4.8b	0.3
Overall tenderness	6.1a	0.3	5.5a	0.3	5.6a	0.3
Cooking loss (%)	16.6a	0.3	18.6b	0.4	18.9b	0.3

a, b: means within the same row with different letters differ significantly ($P < 0.05$).

One source of variation not considered in the initial design of this study was the influence(s) of vacuum packaging, freezing and/or cooking on the muscle fibre angle. Although steaks were carefully cut so as to approximate 0°, conventional and 90° angles initially, after cooking these angles had changed considerably. Table 2 shows both 'initial' (assigned when steaks were cut) and 'final' (assessed on cooked steaks) fibre angles. At initial fibre angles of 90°, final fibre angles were found to be considerably smaller than expected. From the data of Eisenhut *et al.* (1965) one can calculate that the fibre angle in a steak cut conventionally from the LD muscle of a vertically suspended side would vary from 65° (seventh rib) to 29° (third lumbar vertebra). The final fibre angle for conventionally cut steaks (35°, Table 2) was found in this study to be on the low end of this range. The forces applied during the vacuum-packaging technique were probably responsible for this discrepancy.

The effect of fibre angle

Simple means and standard errors for shear force values (three methods of presenting the core to the shear device), initial-bite tenderness, overall tenderness and cooking loss are presented by initial fibre angle in table 2. Shear force values (all methods) were higher, and initial-bite tenderness lower, at

higher muscle fibre angles. An increase of 46.4° (51.8−5.4) in fibre angle resulted in an increase in shear value (normal method) of 2.7 kg and a decrease in initial-bite tenderness of 2.5 panel units as well as an increase in cooking loss of 2.2%. Although in a preliminary study (Murray & Martin, 1980), the final fibre angles were not determined, the difference between the shear force at initial angles of 0° and 90° was similar to that reported above. Overall tenderness, however, appeared to be unaffected by fibre angle. The linear regression of shear force value, initial-bite tenderness, overall tenderness and cooking loss on final fibre angle yielded slopes of 0.058 (s.e.=0.004), −0.057 (s.e.=0.006), 0.013 (s.e.=0.005) and 0.051 (s.e.=0.007) kg/degree, respectively. All four were significantly ($P<0.05$) different from zero, although overall tenderness was only slightly affected by fibre angle. It is suggested that this small effect of fibre angle on overall tenderness may have resulted through an effect of fibre angle on cooking loss or because the shape of the core caused panelists to chew perpendicular to its longitudinal axis rather than in random directions as would have been the case if the sample had been presented as a cube. Inclusion of a quadratic term in addition to the linear term caused further significant ($P<0.05$) decreases in the error mean squares for shear value (normal method) and for initial-bite tenderness only, but increases in within-animal R^2 values (from 78 to 83% and from 60 to 63%, respectively) were small.

Inspection of the unanalysed data indicated that the relationship of shear force value and initial-bite tenderness with fibre angle varied with the inherent tenderness. Since overall tenderness was affected very little by fibre angle, its average values for the three initial fibre angles were arbitrarily divided into three tenderness categories: (1) toughest (scores 3.5−5.0, $n=8$); (2) intermediate (scores >5.0−6.5, $n=12$); (3) tenderest (scores >6.5−8.0, $n=7$). Although this categorization was not included in the original design, breeds and animal groups were reasonably equally represented in all three categories. Overall tenderness, initial-bite tenderness, shear force value and cooking loss are plotted *versus* final fibre angle for each of the three tenderness categories in Fig. 2. The plots indicate that shear value and initial-bite tenderness of the tenderest category were less affected by fibre angle than were the other two categories, and that differences in these tenderness measures were more pronounced at higher fibre angles.

The three tenderness categories, which represent differences among animals, fail to segregate cooking losses into distinct groups. Cooking loss was expected to be related to the fat content. Although the fat content (dry-weight basis) of the least tender category, 11.5% (s.e.=0.9), was less than that for the intermediate and most tender categories, 16.6% (s.e.=1.1) and 17.9% (s.e.=1.7), respectively, cooking losses were similar for all categories. This indicates that the between-animal variation in cooking loss was not related to tenderness variation or to variation in fat content.

The collagen content of the LD muscle of the animals in this study ranged from 0.38 to 0.75% (mean=0.52%, s.e.=0.035) on a wet-weight basis and

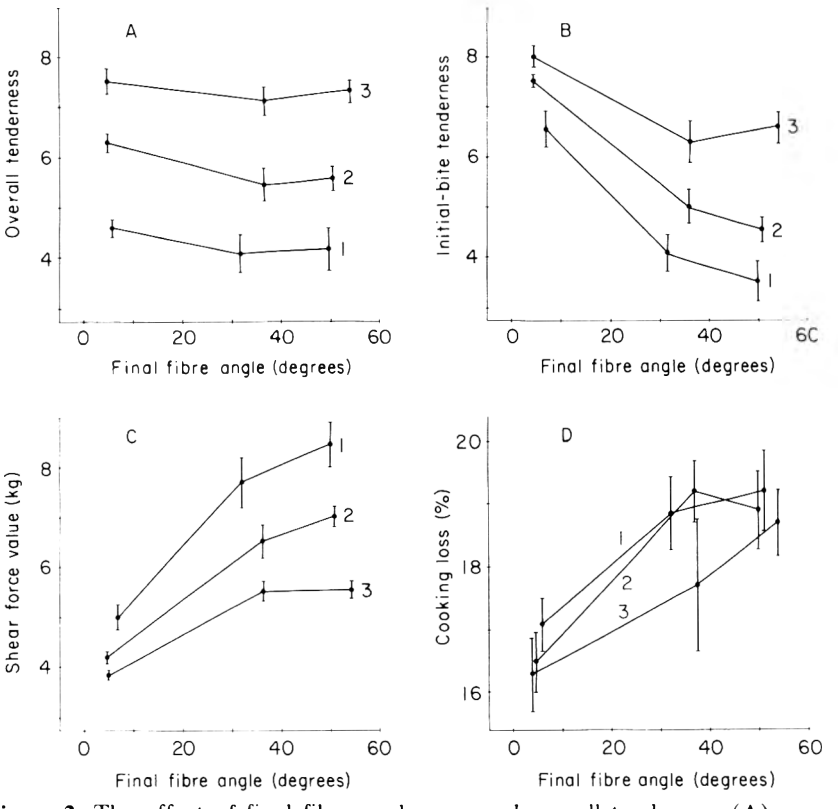

Figure 2. The effect of final fibre angle on panel overall tenderness (A), panel initial-bite tenderness (B), shear force value (C) and cooking loss (D) for each of three tenderness categories (1, toughest, 2, intermediate, and 3, tenderest). (Standard errors are indicated.)

equalled 0.51% (s.e.=0.022), 0.52% (s.e.=0.028) and 0.54% (s.e.=0.035) for categories 1, 2 and 3, respectively. The % solubility of the collagen of this muscle ranged from 12.4 to 22.5 (mean=16.2, s.e.=0.41) and equalled 15.8 (s.e.=0.65), 16.7 (s.e.=0.68) and 15.9 (s.e.=0.79) for categories 1, 2 and 3, respectively. These data suggest that neither total nor percentage soluble collagen was related to any tenderness measure or to the fibre angle times tenderness category interaction for shear force value or for initial-bite tenderness.

Orientation of the core in the shear device

Often steaks are cut from the LD muscle perpendicular to the longitudinal axis of the muscle. Cores for shear determination are then removed by coring perpendicular to the steak surface and are aligned in the shear device so as to shear perpendicular to the longitudinal axis of the core (Hedrick *et al.*, 1968). Rotation of the core about its longitudinal axis is random. In order to determine

the extent to which the degree of rotation of the core influenced shear values, cores were sheared 'parallel' and 'perpendicular' to the longitudinal axis of the steak as described above and in Fig. 1. These directions were chosen arbitrarily so as to have a reproducible frame of reference. 'Perpendicular' and 'parallel' shear values tended to be higher and lower, respectively, than those obtained by the 'normal' method, and the difference (0.4–0.6 kg) between 'perpendicular' and 'parallel' shear values was significantly ($P<0.05$) greater than zero at the three initial fibre angles studied (Table 2).

At the final fibre angle of 0°, the fibres were organized at an angle of approximately 0° with respect to the steak surface. In this case the fibres were oriented at approximately right angles to the longitudinal axis of the steak. Shearing of a core 'perpendicular' with respect to the longitudinal axis of the steak caused the shear blade to approach the core so as to first hit the ends of the fibres. Shearing of a core 'parallel' with respect to the longitudinal axis of the steak caused the shear blade to first hit the length of the fibres. In both cases the shearing is on the same plane and at the same fibre angle. The data in Table 2 indicate that for this example 0.6 kg more shear force was required to shear into a core hitting first the ends of the fibres then to shear into a core hitting first the length of the fibres. Little can be concluded concerning the other initial fibre angles (conventional and 90°) since the exact orientation of the muscle fibres with respect to the longitudinal axis of the steak was not evaluated. At present an explanation for this phenomenon is not apparent. It is possible that the force of the shear blade hitting the ends of the fibres caused them to change fibre angle. Thus instead of passing between the fibres the blade may have broken a portion of the fibres. If this effect was purely due to muscle fibre orientation, then one might expect it to be absent at a final fibre angle of 90°. In any case, such an effect will undoubtedly add to the amount of variation within data from a given steak. However, by using a random degree of rotation of the cores in the shear device (normal method) no bias should be introduced into the experimental result.

Variation within and between animals

An analysis of variance was completed (Table 3) to determine the relative contributions of animals, steaks and cores to the total variation. Since final fibre angle was recorded only as the mean and range of 4 or 5 protractor readings per steak, the within-steak variance could not be calculated. However the average ranges (twenty-seven animals at each initial angle) of 9.6°, 14.2° and 24.1° for initial angles of 0°, conventional and 90°, respectively, indicated that within each steak the final angle varied considerably. The extent of this variation increased with increasing fibre angle, as did that of shear value measured by each of the three methods. Within-steak variation is undoubtedly due to a combination of the heterogeneity in the distribution of intramuscular fat, connective tissue, muscle fibres, etc. in addition to variation in fibre angle. The variation between steaks within animal increased with increasing fibre angle for

Table 3. Analysis of variance of final fibre angle, shear force value and cooking loss at each of three initial fibre angles.

		Source of variation							
		Animal			Steak within animal			Core within steak within animal	
Variable	Initial fibre angle	DF_A	MS_A	F_A	DF_S	MS_S	F_S	DF_C	MS_C
Final fibre angle	0°	26	28	1.8	27	16	—	—	—
	Conventional	26	48	1.4	27	34	—	—	—
	90°	26	66	1.2	27	55	—	—	—
Shear force value									
Normal	0°	26	4.6	4.9*	27	0.9	1.5	108	0.6
	Conventional	26	11.8	6.7*	27	1.8	1.1	108	1.7
	90°	26	13.6	5.9*	27	2.3	1.4	108	1.7
Perpendicular	0°	26	2.1	3.3*	27	0.6	1.2	54	0.5
	Conventional	26	9.2	5.8*	27	1.6	1.0	54	1.6
	90°	26	8.3	4.6*	27	1.8	1.1	54	1.6
Parallel	0°	26	2.6	5.3*	27	0.5	0.8	54	0.6
	Conventional	26	7.6	7.0*	27	1.1	1.1	54	1.0
	90°	26	7.1	4.6*	27	1.5	1.6	54	1.5
Cooking loss	0°	26	9.7	3.3*	81	2.9	—	—	—
	Conventional	26	18.2	5.4*	81	3.3	—	—	—
	90°	26	12.0	3.0*	81	4.0	—	—	—

A, S, C: subscripts denote animal, steak within animal and core within steak within animal, respectively.

DF: degrees of freedom. MS: mean square. $F_S = MS_S/MS_C$, $F_A = MS_A/MS_S$.

* F ratios are significant ($P < 0.05$).

all variables examined and for shear value was, as expected, not larger than the variation within steaks. The between-animal variation was greater than that between steaks for shear value and for cooking loss only and it increased with increasing fibre angle. The magnitude of the F-statistic used to make this test suggested that the animal differences in shear value and cooking loss were determined with similar sensitivities at the three initial fibre angles. Final fibre angle was not significantly affected by animal. This suggests that within initial fibre angle, although the final fibre angle may affect within-animal variation, it is likely to contribute only marginally to the between-animal variation in these measures.

Correlations among tenderness measures, cooking loss and muscle components

Correlations involving tenderness measures and cooking loss at each of the three initial fibre angles are presented in Table 4. Shear value was negatively correlated with each of the panel estimates and the correlation coefficients increased significantly in magnitude as the initial fibre angle increased. For the

Table 4. Correlations involving tenderness measures and cooking loss at three initial fibre angles

	Initial fibre angle	Final fibre angle	Shear force value	Initial-bite tenderness	Overall tenderness
Shear force value	0°	0.35			
	Conventional	−0.19			
	90°	0.04			
	Pooled[A]	0.88*			
Initial-bite tenderness	0°	−0.34	−0.56		
	Conventional	0.02	−0.59*		
	90°	0.07	−0.76*		
	Pooled[A]	−0.77*	−0.86*		
Overall tenderness	0°	−0.28	−0.71*	0.76*	
	Conventional	0.19	−0.81*	0.82*	
	90°	−0.07	−0.89*	0.91*	
	Pooled[A]	−0.33*	−0.50*	0.70*	
Cooking loss	0°	−0.12	0.22	−0.21	−0.24
	Conventional	−0.11	0.19	−0.26	−0.25
	90°	0.17	0.21	−0.07	−0.19
	Pooled[A]	0.68*	0.71*	−0.61*	−0.33*

A: pooled over angles and within animal ($P < 0.05$).

* Correction coefficient differs significantly ($P < 0.05$) from zero.

three initial fibre angles shear values were more strongly correlated with overall tenderness than with initial-bite tenderness. Since both shear value and initial-bite tenderness would be expected to estimate the force required to cut through a core of meat, they were expected to yield the highest correlation coefficient. This finding is not as yet understood. Cooking loss was unrelated to any tenderness measure at each of the three initial fibre angles. This same conclusion was evident from Fig. 2 where segregation of carcasses into tenderness categories failed to segregate cooking loss into categories. Although cooking loss varied considerably among animals (Table 3), this variation appeared to be unrelated to tenderness. Where the pooled within-animal (angles pooled and the variation due to animal removed) correlation coefficient was greater than that at the individual initial fibre angles, the correlations (cooking loss *versus* either shear value or initial-bite tenderness) arose primarily from a fibre angle effect. If the converse were true, then the correlation (overall tenderness *versus* shear value) was due primarily to an animal effect. In certain situation correlations (initial-bite tenderness *versus* either shear value or overall tenderness) were due to a combination of angle and animal effects.

Intramuscular fat was significantly ($P<0.05$) related to shear value ($r=0.46-0.58$) and to overall tenderness ($r=0.40-0.53$) at all three initial fibre angles and to initial-bite tenderness ($r=0.49$) at an initial fibre angle of 90° but not to cooking loss ($r=0-0.35$). Certain objective measures of tenderness, such as Instron compression and adhesion (Bouton *et al.*, 1975), sense a variation in

the forces holding muscle fibres in register and are usually correlated with connective tissue properties. The magnitude of the shear (peak force) values obtained by shearing meat which had been cooked to 75°C at 90° to the direction of the muscle fibres, would probably reflect the toughness of the muscle fibres (Moller, 1980–81). One might then expect connective tissue properties to be related to the shear force at a fibre angle approaching 0° (Nottingham, 1956). Such was not the case. Total collagen content and percent solubility were not significantly correlated with any tenderness measure (r=0.13–0.22).

Conclusions

Within the range of muscle fibre angles used in this study, the angle of orientation of the fibres with respect to the steak surface had a substantial effect on measures of shear force value (0.058 kg/degree) and initial-bite tenderness (−0.057 panel units/degree) and a much lesser effect on overall tenderness (−0.013 panel units/degree). Shear force values and initial-bite tenderness were less affected by fibre angle in tender than in tough meat. A number of factors affected the muscle fibre orientation at the time of shearing or biting. These include: the muscle fibre angle in the raw steak; the effects of vacuum packaging, freezing and/or cooking; the angle at which the core was removed from the steak; and the variation in fibre angle inherent in the steak. The angle of rotation of the meat sample at time of shearing also contributed to variation in shear values but this effect and that due to variation in fibre angle within steak are usually overcome by increasing the number of meat samples sheared or bitten (Dransfield & MacFie, 1980). If cores are obtained by coring perpendicular to the steak surface (Hedrick *et al.*, 1968), increasing the number of cores will not overcome the error incurred by cutting the steaks at an inconsistent fibre angle. This error will be manifest as an effect due to difference between animals and may seriously bias statistical analysis. This may tend only to increase the error mean square and thus decrease the precision of the experiment. However, very often adjacent steaks are used for the measurement of variables to be correlated. Adjacent steaks usually have very similar fibre angles, yet the average fibre angle may vary considerably from animal to animal. In this case if the two variables to be correlated (e.g. shear value and initial-bite tenderness) are both affected by fibre angle, spuriously high correlation coefficients may result. If only one variable is affected by fibre angle, the correlation coefficient may be inappropriately low.

Panel evaluation of overall tenderness has the advantage that it is relatively insensitive to variation in muscle fibre angle. Shear force values, although dramatically affected by fibre angle, are adequate estimators of the overall panel tenderness of the LD muscle if the muscle fibre orientation is controlled.

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(*Received 19 November 1982*)

J. Fd Technol. (1983) **18**, 619–627

Development of an improved soya protein-lipid film

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Summary

Soya protein-lipid film, an oriental food material, as traditionally prepared is limited in methionine and has inferior physical properties. A simple modification to the traditional method of preparation, involving addition of DL-methionine and glycerol to soya milk before film formation, yielded a product which was nutritionally improved and obviated the need to reconstitute it.

Introduction

Soya protein-lipid film (SPLF) and meat analogues made therefrom are traditional oriental foods. In the typical batch manufacturing process, the film which forms on the surface of heated soya bean milk is lifted manually with the aid of a slender stick and hung up to air-dry. Wu & Bates (1972a, b, 1973, 1975) have contributed significantly to knowledge concerning the mechanism and optimization of the film formation phenomenon.

Organoleptically, SPLF itself is rather bland. It is normally used as an ingredient in many food dishes, as a wrapping film for other materials such as meat, and as a starting material for the fabrication of a wide range of oriental simulated meat products or meat analogues with improved texture and flavour (Wu & Bates, 1975). Dehydrated SPLF has to be reconstituted into a pliable product for these purposes. The poor reconstitution properties and the susceptibility to breakage of the dried product as a result of its brittleness are areas which need to be improved upon. Besides these disadvantages, SPLF is also nutritionally limited in the sulphur-containing amino acids, as is generally true of foods derived from soya bean (Bates & Wu, 1975). The present study was thus aimed at eliminating these disadvantages by developing, through

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0022–1163/83/1000–0619 $02.00

incorporation of DL-methionine and glycerol, a nutritionally improved, intermediate moisture SPLF which is ready to eat without the need for rehydration, pliable enough for immediate use as a wrapping film or for fabrication into imitation meat products, and reasonably shelf-stable under ambient conditions.

Materials and methods

Preparation of SPLF

Dehulled soya beans of the Darien variety were soaked in water at 65°C for 1 hr, drained, and ground in a stone mill with ten times their weight of water. The soya milk was separated in a basket centrifuge and its soluble solids content was adjusted with water to 4° Brix using a refractometer. The milk filled to a depth of about 7 cm in a shallow stainless-steel container (45 L×30 W×10 H cm) fitted tightly over a steam bath, was heated to and maintained at a temperature of about 85°C. Films formed on the milk surface were picked up at 20 min intervals until the milk became too viscous to sustain further film formation. Seven sheets could be obtained per batch of milk, each sheet weighing an average of about 14 g after air-drying to about 5% moisture (on dry basis). The dried films from each batch were ground using a laboratory hammermill and thoroughly mixed. All chemical analyses and experiments were carried out using the ground samples.

Methionine fortification was achieved by dissolving the DL-amino acid in the soya milk before the film formation process. Various levels of DL-methionine were added to the milk, and the extent of its incorporation into the films was determined. An intermediate moisture product was produced by preparing films from soya milk containing added glycerol at a level of 40 g/l.

Chemical analysis

Moisture content was determined gravimetrically by drying ground samples of SPLF at 105°C for 6 hr. Nitrogen content was determined using the semi-micro Kjeldahl method (AOAC, 1975). The amino acid profile of unfortified, non-glycerated SPLF was determined on defatted samples after acid hydrolysis with 6 M HCl using a Technicon NC-2P amino acid analyser. Methionine was analysed separately using the non-chromatographic chemical method of Concon (1975) which involves preliminary extraction of protein from ground material with 0.075 M NaOH, alkaline hydrolysis of the extract with 2.5 M NaOH, simultaneous removal or suppression of interferences by histidine and tryptophan using HCHO-HCOOH mixture, and determination of the absorbance at 510 nm after colour development with sodium pentacyano-nitroferrate (also known as sodium nitroprusside). Glycerol content was determined using the periodate oxidation method (AOAC, 1975). In the determination of peroxide value (PV), lipids were first extracted from finely ground samples using a 3:1 (v/v) chloroform-methanol mixture. The AOCS

(1973) official method Cd 8–53 was then used to determine PV which was expressed as milliequivalents of peroxide/kg extracted fat. The method used for determination of thiobarbituric acid (TBA) value was essentially that described by Tarladgis *et al.* (1960). The TBA value was expressed as mg malonaldehyde/kg sample.

Determination of protein efficiency ratio (PER)

The PER was determined following AOAC (1975) procedures, except that five individually caged, male, weanling Sprague-Dawley rats were used for each dietary treatment instead of ten. Diets containing about 10% protein ($N \times 6.25$) were prepared and fed to six groups of five rats each for 28 days. Feed and water were supplied *ad libitum*. Several dietary treatments were compared using casein as the reference protein. Significant differences at the 5% probability level between PER treatment means were determined using Duncan's multiple range test.

Water sorption studies

Adsorption and desorption isotherms of finely ground SPLF, glycerated or non-glycerated, were obtained by equilibrating samples (previously dehydrated over phosphorus pentoxide or humidified over water) over different saturated salt solutions of known relative humidities (Rockland, 1960) in vacuum desiccators at 30°C. Moisture contents were determined after an equilibration period of 48 hr.

Stability studies

The stability to lipid oxidation (which appeared to be the main chemical deteriorative reaction limiting shelf life) of methionine-fortified, glycerated SPLF at 30°C was determined as a function of water activity (a_w) by following changes in PV and TBA value with time. Samples at different a_w were prepared by equilibration over various saturated salt solutions in vacuum desiccators.

Samples from each hydration level were then apportioned into airtight screw-capped bottles and stored in the dark at 30°C. Duplicate samples at each a_w level were withdrawn at suitable intervals of time and analysed for PV and TBA value.

Results and discussion

Nutritional improvement of SPLF

Soya protein-lipid film is a high-protein food with a protein content ($N \times 6.25$) of about 60% on a moisture-free basis. However, as shown in Table 1 which compares the amino acid composition of SPLF with the WHO/FAO (1973)

Table 1. Amino acid composition of SPLF

Amino acid (g/16 g N)	SPLF	WHO/FAO (1973) reference pattern
Isoleucine	4.6	4.0
Leucine	8.1	7.0
Lysine	7.7	5.5
Methionine	1.2	—
Methionine + cystine	2.0	3.5
Phenylalanine + tyrosine	9.3	6.0
Threonine	4.4	4.0
Tryptophan	—	1.0
Valine	4.8	5.0
Alanine	4.7	—
Arginine	8.5	—
Aspartic acid	12.0	—
Glutamic acid	20.4	—
Glycine	4.1	—
Histidine	2.9	—
Proline	4.9	—
Serine	5.4	—

reference pattern, the product suffers from a deficiency in the sulphur-containing amino acids. Bates & Wu (1975) have demonstrated that the nutritional value of SPLF could be improved by direct fortification with methionine. It was felt, however, that such a step, if left to the consumer (particularly the poor or thrifty housewife), would not always produce the desired result. The present study was thus directed towards finding a convenient way for manufacturers to effect methionine fortification of SPLF by adding the amino acid to the milk before film formation.

For the relationship between the degree of incorporation of methionine into SPLF and the level of its addition to the milk, films obtained for each level of addition were combined and the average methionine content was determined. The incorporation of methionine into SPLF appeared to increase proportionately with the level of addition of the nutrient to the milk, as shown in Fig. 1. Analysis of total methionine in individual films showed, however, that incorporation of the amino acid was non-uniform from film to film produced from any particular batch of milk. The data from a typical example (Table 2) show increases in methionine content with each film formed. Differences in methionine-incorporation efficiency from film to film could be due to the changing soluble solids and methionine concentrations in the milk as the film formation process progressed.

The results of animal feeding trials are summarized in Table 3. Statistical analysis of the data obtained shows that unfortified, non-glycerated SPLF (Treatment II) had a significantly lower PER value than casein and the methionine-fortified samples at the 5% level of probability. However, no

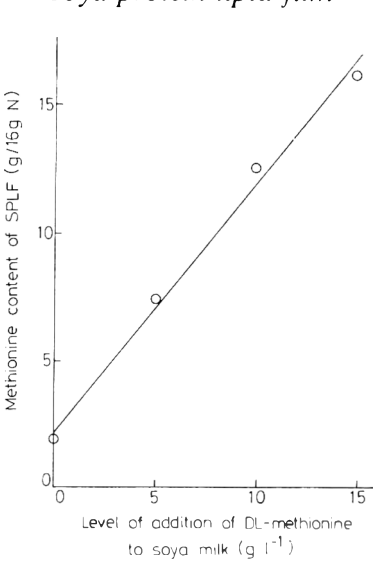

Figure 1. Relationship between the level of addition of DL-methionine to soya milk and the average methionine content of the resulting SPLF.

significant differences ($P<0.05$) were found to exist between casein and each of the methionine-fortified samples, as well as between any two of the fortified samples. An addition of methionine at a level of 5 g/l to the soya milk was thus more than adequate to ensure sufficient incorporation of methionine to raise the PER of the films beyond that of the casein reference. The presence of glycerol which was introduced into the films to produce an intermediate moisture product did not appear to have any significant effect on PER.

Intermediate moisture SPLF

An intermediate moisture product was developed in an attempt to improve on the physical properties of SPLF. Addition of glycerol at a concentration of 40 g/l and methionine at a level of 10 g/l to soya milk yielded satisfactory intermediate moisture films with average glycerol and methionine contents of

Table 2. Methionine contents of films from milk containing added methionine at a level of 5 g/l

Film number	Methionine content (g/16 g N)
1	3.1
2	4.6
3	5.2
4	5.6
5	7.5
6	11.9
7	20.2

Table 3. PER values of unfortified and methionine-fortified SPLF (means of five replications)

Dietary treatment	Weight gain (g)	Protein intake (g)	PER	
			Experimental	Adjusted*
I Casein (reference)	91.9	26.1	3.6	2.5a
II SPLF: unfortified, non-glycerated	49.5	20.6	2.3	1.7b
III SPLF: methionine-fortified (5 g/l)† glycerated (40 g/l)	89.2	21.2	4.2	3.0a
IV SPLF: methionine-fortified (10 g/l) glycerated (40 g/l)	94.3	24.5	3.9	2.7a
V SPLF: methionine-fortified (10 g/l) non-glycerated	100.4	24.8	4.1	2.9a
VI SPLF: methionine-fortified (15 g/l) glycerated (40 g/l)	84.1	20.3	4.3	3.0a

* Adjusted PER means followed by the same letter are not significantly different at the 5% probability level.

† Figures within parentheses indicate the levels of addition of DL-methionine and glycerol to the milk used to produce SPLF. Films obtained from each batch of milk were combined.

14.4 g/100 g of dry solids and 12.1 g/16 g *N* respectively. Addition of glycerol to the milk at levels exceeding 50 g/l is not recommended as the resulting high viscosity of the milk was found to retard the rate of film formation. A non-glycerated product, fortified to the same level with methionine, was also prepared as a control.

The water sorption isotherms of non-glycerated and glycerated SPLF are shown in Figures 2 and 3 respectively. The former system was observed to exhibit moderate moisture sorption hysteresis which was fairly evenly

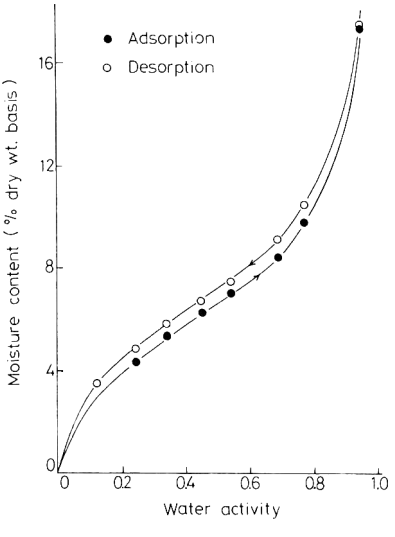

Figure 2. Water sorption isotherms of non-glycerated SPLF at 30°C.

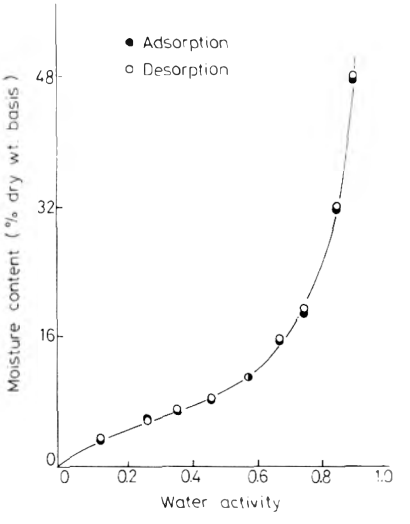

Figure 3. Water sorption isotherm of glycerated SPLF at 30°C.

distributed over practically the entire range of relative vapour pressures. Such a pattern may be designated as Type-C hysteresis according to the classification of Everett (1967) and is typical of adsorption of water on proteins or in high-protein foods (Wolf, Walker & Kapsalis, 1972). Hysteresis is known to affect many deteriorative reactions in foods, desorption samples generally being found to be less stable than the corresponding adsorption samples at any particular a_w (Labuza *et al.*, 1972; Wolf *et al.*, 1972; Labuza & Chou, 1974; Acott & Labuza, 1975; Kapsalis, 1981). The incorporation of

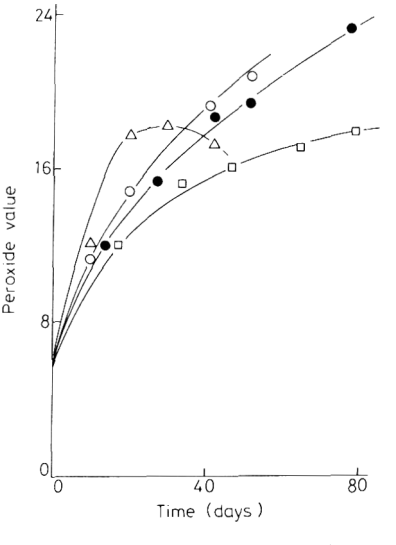

Figure 4. Changes in PV of intermediate moisture SPLF at different water activities (□ 0.67, • 0.75, ○ 0.86, △ 0.92) during storage at 30°C.

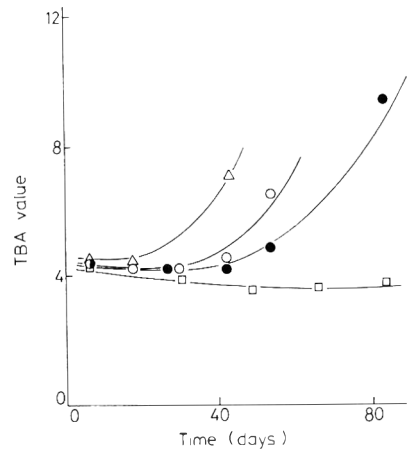

Figure 5. Changes in TBA value of intermediate moisture SPLF at different water activities (□ 0.67, ● 0.75, ○ 0.86, △ 0.92) during storage at 30°C.

glycerol into SPLF not only conferred a greater water-binding capacity, particularly over the intermediate and high a_w ranges, but apparently also eliminated the hysteresis phenomenon. The possibility of hysteresis affecting the storage stability of intermediate moisture SPLF, therefore, does not arise. The moisture content of the product over the range of a_w between 0.65 and 0.85 was high enough to confer the desired texture and flexibility to the films.

Changes in PV and TBA value of intermediate moisture SPLF at different a_w levels during storage at 30°C are given in Figures 4 and 5 respectively. The ascending slopes of the curves are indicative of the relative rates of lipid autoxidation. Where both oxidation parameters are concerned, it was observed that the rates of peroxide and malonaldehyde production increased as a_w was raised from 0.67 to 0.92. In addition, the induction period for formation of the secondary oxidation product was found to decrease with an increase in a_w (Fig. 4). Thus, water appeared to act as a 'pro-oxidant' as a_w was increaseed from 0.67 to 0.92. These results are consistent with those obtained by other workers in their studies on the influence of adsorbed water on lipid autoxidation in intermediate moisture foods and model systems. An increased rate of lipid autoxidation in the intermediate a_w region has been postulated to be due to increased solubilization and mobility of trace metal catalysts as the aqueous phase becomes less viscous, and to the exposure of new catalyst sites as the system swells (Heidelbaugh & Karel, 1970; Labuza *et al.*, 1971, 1972).

Rancidity (judged subjectively) in any particular sample was detectable at a PV of *circa.* 18 mEq./kg of lipid and a TBA value of *circa.* 4.3 mg malonaldehyde/kg of sample. A rancid odour was detected in the sample at 0.92 a_w after only about 20 days' storage. Rancidity was, however, not detected in the sample at 0.67 a_w even after a storage period of 80 days. Other aids to extension of shelf life such as low temperature storage, gas or vacuum

packaging, and the addition of suitable antioxidants and antimycotics may be employed in conjunction with the control of a_w and moisture content.

Conclusions

A methionine-fortified, intermediate moisture SPLF can be prepared by adding the necessary nutrient and humectant to soya bean milk before film formation. Such a technique could conceivably be extended to other suitable nutrients (for example, vitamins and minerals) and food additives. An intermediate moisture product with an a_w of about 0.7 would be reasonably shelf stable at 30°C and flexible enough to facilitate subsequent film preparation or fabrication operations without the need for reconstitution.

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J. Fd Technol. (1983) **18**, 629–638

The anthocyanins of sunflower

II. A study of the extraction process

PIER GIORGIO PIFFERI and ANGELO VACCARI

Summary

The extraction process of red pigments from the husks of a cultivar of sunflower has been studied. It has been shown that a fraction containing *circa* 80% of the starting pigments could be obtained by grinding. Best extractions were obtained with distilled water-organic solvent mixtures of intermediate polarity values. The extraction yields are strongly influenced by the pH and the solvent/powder ratio and reach the maximum after about 2 hr.

The experimental data showed that the greater part of the pigments, readily precipitable by acidification to pH 2.0, are bonded to a macromolecule, probably of protein nature, as confirmed by electrophoresis.

Introduction

One of the problems facing the food industry today is the replacement of the natural colouring of foodstuffs destroyed during processing. On the other hand, in the last few years there has been a gradual abandonment of the synthetic red colorants (Botnra, 1974; Italian Ministry of Health, 1976), because of their potential toxicity.

Interest in new sources of natural pigments like the anthocyanins is therefore justified, especially if the possibility exists of obtaining large quantities at low production cost.

In a previous paper we reported for the first time the presence of anthocyanins in the achenes of some varieties of sunflower, giving a preliminary identification (Vaccari, Pifferi & Zaccherini, 1981). This source is expected to become very interesting if new pigment-rich cultivars are introduced in farming, taking into account the increasing trend of dehulling achenes both to extract the oil with solvent and to obtain a proteinaceous meal of interest as a potential source of protein for human consumption (Lusas,

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Paper partially presented at 'Journées internationales du Groupe Polyphenols', Toulouse, France, 1982.

0022–1163/83/1000–0629 $02.00

1978; Lanzani & Petrini, 1979; Neumunz, 1980; Fetzer & Hostettler, 1980; Sosulski & Zadernowski, 1980; Sosulski, 1981; Tranchino *et al.*, 1981).

This paper deals with the development of the extraction method: we analysed in particular the influence of some parameters, as nature of the solvent, presence of water, polarity and acidity of the mixtures. For some solvents we also examined the effect on the extraction yield of the time, pH, temperature and solvent/powder ratio.

Lastly, we have reported some information on the nature of the macromolecule to which the greater part of the pigments are bonded.

Materials and methods

Materials

Laboratory grade reagents were used in all phases. For simplicity all successive operations were carried out on one cultivar of sunflower we isolated in the course of our research.

Methods

The husks were ground by a Janke-Kunkel electric-mill mod. A-10 at 20000 rev/min and crushed in three fractions (>0.56, $0.56<d>0.25$, and <0.25 mm).

For the preliminary study of the solvents 100 mg of the last fraction were treated in 100 ml amber glass centrifuge tubes with screw caps with 40 ml of solvent. The tubes were placed in a Dubnoff water bath thermostatically maintained at 25 ± 1°C under agitation. At different times after a centrifugation of 15 min at 5000 rev/min (3000 g) we took a 0.5 ml sample of the supernatant and added 2.0 ml of 0.1 N HCl, registering the absorbance in the 390–590 nm range, with a Perkin Elmer mod. 402 spectrophotometer and using an $\bar{\varepsilon}$ (mean-molar extinction coefficient) of 28000 in the calculations (Niketic-Aleksic & Hradzina, 1972). Where necessary, the sample was first diluted with 0.1 N HCl.

To study the influence of the solvent/powder ratio (S/P ratio), we proceeded as previously described using larger amounts of powder. To determine the total amount of the extracted substances, 20 ml of the supernatants after centrifugation of the extracts obtained after 4 hr were placed in a glass capsule and maintained at 40 ± 1°C until constant weight.

The influence of the pH of the solution was determined using: (a) HCl and NaOH; (b) 0.1 M buffer solutions prepared according to Gomori (1955), i.e. HCl—KCl, citric acid—sodium citrate, sodium phosphate monobasic-sodium phosphate dibasic; (c) methanol—buffer solution mixtures (6:4 v/v).

The amounts of protein were determined with a 0.06% (w/v) solution of Coomassie Brilliant Blue G-250 in 3% (w/v) perchloric acid, using egg albumin to prepare the standard solutions (Sedmark & Grossberg, 1977). The

Kjeldahl method was used to determine the nitrogen contents (Horwitz, 1960a).

The supernatant obtained by centrifugation at 5000 rev/min (3000 g) for 15 min of the water extract (4 hr, S/P ratio 10 ml/g) was acidified to pH 2.0, kept at 4°C for 24 hr and centrifuged again. The residue was analysed by electrophoresis on polyacrylamide gel according to Davis (1964) at pH 8.9, using Coomassie Brilliant Blue for staining the proteins and the 0.01 N HCl for the anthocyanins.

Results and discussion

In Table 1 we have reported some features of our cultivar (A) and, for the sake of comparison, those of a cultivar commonly found throughout the world (B): it may be noted that the results are very similar.

By grinding the husks, it is possible to increase the extraction rate 10-fold: this may be due to different factors as, for example, a cleavage of the wax and lipid rich layers of the husk (Cancalon, 1971), with a greater accessibility of the anthocyanins to the solvent.

From Table 2, it can be observed that it is possible to obtain a fraction equal to 40% of the starting powder containing 80% of the pigments: this fact is very important from the industrial point of view on account of the possible reduction of the extraction costs.

Furthermore, it should be borne in mind that the pigment content of this fraction is greater than that of the roselle (*Hybiscus sabdariffa* (Esselen & Sammy, 1975)) and is approximately the same as the commercial Italian solid oenocyanin (Pifferi, unpublished).

Table 1. Characteristic data of the examined sunflower cultivar (A) and of another cultivar commonly found throughout the world (B)

				Oil		Anthocyanins	
Sunflower cultivar	Moisture %	Seeds %	Husks %	Kernel %	Whole seed %	Husks %	Whole seed %
A	6.50	52.80	47.20	50.80	25.55	2.59	1.41
B	4.15	63.70	36.30	56.20	35.80	2.86	1.04

Table 2. Pigment distribution in the sunflower powder

Powder fraction mm	Weight %	Anthocyanin distribution %	Anthocyanin content (for the dry husks) %
>0.59	9 ± 2	2.25	0.014
$0.59 < d > 0.25$	48 ± 2	17.80	1.128
<0.25	40 ± 2	79.85	5.080
Losses	3 ± 2	—	—

Nature of the solvent

A glance at Table 3 shows that formic acid is the organic acid with the highest extraction capacity and rate due to its high polarity. The presence of −SH and −OH polar groups explains the higher extracting power of thioglycolic acid and lactic acid over acetic acid and propionic acid respectively. As for alcohols, extraction appears to be influenced by the concomitant effect of the increase in viscosity and the decrease in the dielectric constant. The latter also appears to be a dominant factor in extraction with bi- and trivalent alcohols.

As regards aqueous acid solutions, the extraction yield increases as acid strength and anion steric hindrance decrease. With organic acids, however, it is also necessary to take into account the changes in the surface tension of the solutions. Increasing the polar group number modifies the extraction yield unpredictably (see, for example, the oxyacids).

Finally, distilled water gives higher extraction yields than the acid solutions and this can be attributed to the action of native enzymes and/or to the fact that the greater part of the pigments are bonded to a molecule that is not very soluble in acid solution. In fact, the free anthocyanins have the highest solubility as flavylium salts at low pH both in aqueous and alcohol solution

Table 3. Relative extraction values and extraction half times for anhydrous and aqueous solvents (S/P ratio 400 ml/g)

Anhydrous solvents	$t_{50\%}$ (min)	Relative extraction value*	Aqueous solvents	$t_{50\%}$ (min)	Relative extraction value*
Formic acid	< 5	100.0	Distilled H_2O	9	52.0
Acetic acid	45	25.4	0.01 M Inorganic acid		
Propionic acid	50	19.0	HCl	8	21.5
Thioglycolic acid	< 5	58.0	H_2SO_3	60	28.8
Lactic acid	200	100.0	H_2SO_4	18	12.8
			H_3PO_4	12	18.9
0.01 N HCl in			0.01 M Organic acid		
Methanol	10	100.0	(a) formic	30	8.9
Ethanol	30	78.5	acetic	27	40.3
Proponal	50	36.0	propionic	30	26.4
Butanol	150	16.3	pivalic	18	36.0
			(b) oxalic	30	12.4
Ethylene glycol	30	100.0	malonic	12	8.6
Propylene glycol	90	50.5	succinic	18	30.5
Diethylene glycol	65	68.4	glutaric	12	10.7
Glycerol	28	78.8	(c) glycolic	12	23.6
			lactic	18	9.4
			glyceric	30	29.2
			(d) malic	10	18.1
			tartaric	13	13.7
			citric	16	11.0

* Referred to formic acid extraction value

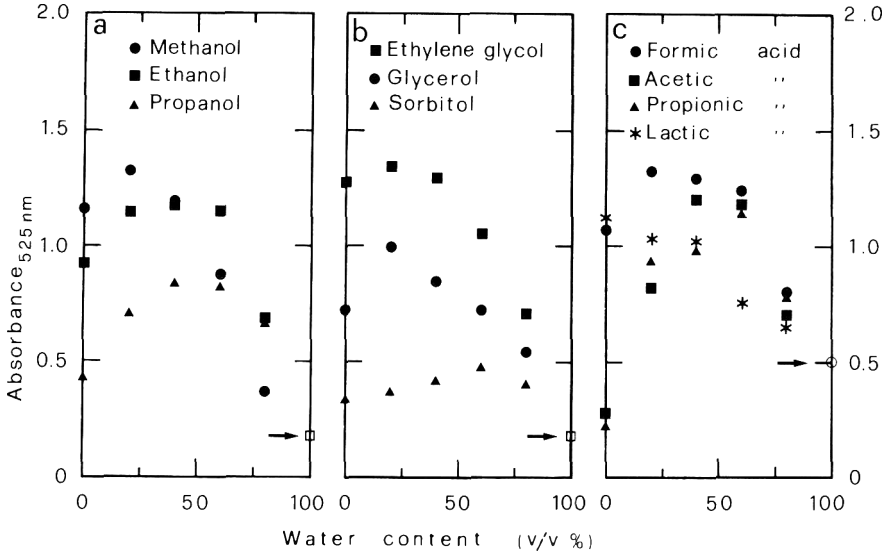

Figure 1. Extractions with organic solvent-H_2O mixtures, 0.01 N in HCl for the solvents of Fig. 1a and b (S/P ratio 1600 ml/g, extraction time 4 hr); □, 0.01 N HCl, ○, distilled water.

(Deibner & Bourzeix, 1966; Aubert & Poux, 1969; Sakellariades & Luh, 1974; Malgarini & Peri, 1978).

Figure 1 shows the extraction trends with organic solvent—H_2O mixtures (0.01 N in HCl for the solvents of Fig. 1a and 1b). It is possible to note that for each mixture there is an extraction maximum corresponding to a quantity of water as great as the polarity of the pure organic solvent is low. Pure lactic acid has a higher extraction power than its aqueous solutions (Fig. 1c). This can be explained by its high polarity.

The influence of the water percentage on the extraction capacity is greater for methanol and ethylene glycol than for the other solvents. Note that, considering the high S/P ratio, the solubility of free anthocyanins should not differ very much with varying mixture composition from the value in 0.01 N HCl: so these data also suggest that the greater part of the pigments are bonded.

Extraction time

From Figure 2, it can be observed that, with distilled water and 0.01 N HCl, the maximum extraction yield is reached after *circa* 1 hr. The alcohols, on the other hand, need a longer time, inversely proportional to the polarity of the alcohol.

The effect of the acidity (compare distilled water and 0.01 N HCl) and the somewhat low yields with the alcohols agree with the theory that the greater part of the sunflower pigments has a high molecular weight (Deibner &

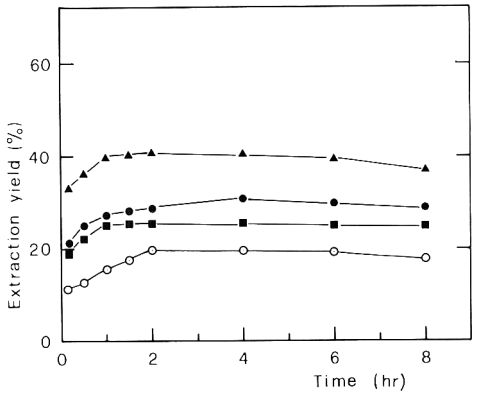

Figure 2. Extraction yields as a function of time (S/P ratio 10); ▲, distilled water, ■, 0.01 N HCl, ●, 0.01 N HCl—CH_3OH, ⊙, 0.01 N HCl—C_2H_5OH.

Bourzeix, 1966; Aubert & Poux, 1969; Sakellariades & Luh, 1974; Malgarini & Peri, 1978), with the anthocyanins bonded to a macromolecule of an acidic nature.

S/P ratio

Extraction yields vary considerably as a function of this ratio, on account of the buffer action exerted by the powder (Fig. 3a): note in particular the decrease in the yield for the 0.01 N HCl, which can be attributed to the marked

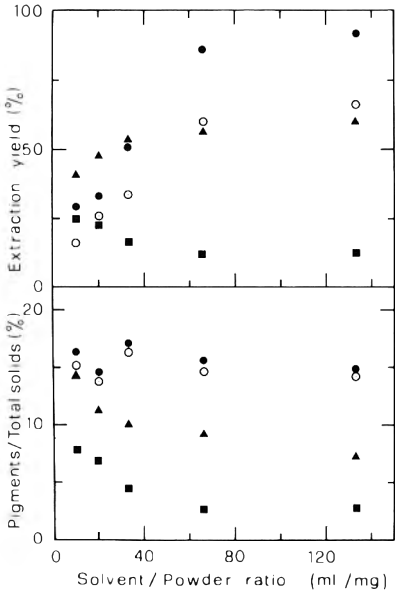

Figure 3. Extraction yields (a) and purity ratios (b) as a function of the S/P ratios (extraction time 4 hr). ▲, Distilled water, ■, 0.01 N HCl, ●, 0.01 N HCl—CH_3OH, ⊙, 0.01 N HCl—C_2H_5OH.

effect of the acidity of the solvent. Distilled water gives the best yields up to ratios ≤35 and it is the solvent which most rapidly reaches an asymptotic trend. The alcohol trends are practically linear up to ratio 65 and this confirms that the greater part of the pigments is soluble in these solvents to a limited extent strictly correlated to the polarity of the solvents.

Moreover, the alcohols have the highest values of the ratio of pigment quantity to total substances extracted (purity ratio). These values are influenced little by the nature of the alcohol and the S/P ratio (Fig. 3b). Vice versa, with the aqueous solvents, the purity ratio decreases rapidly on account of the solubilization of other substances. The 0.01 N HCl has the lowest values, caused by the depressive effect of the acidity on the extraction of the pigments.

pH and temperature

With the aqueous systems studied, extraction is lowest at about pH 2.0 and increases almost linearly as the pH increases up to approximately pH 8.0, after which degradation phenomena set in (Fig. 4).

In Figure 5, we have reported the data for the methanol-0.1 M buffer solution mixtures at different pH. A similar trend may be noted in the extraction yields of protein and anthocyanins. After acidification until pH 2.0 and centrifugation, the amount of the pigments in the solution was practically the same, independent of the starting pH value, and larger in aqueous solution.

These results suggest that the anthocyanins are bonded to a macromolecule in which acidic groups are prevalent, with an isoelectric pH value of around 2.0. This macromolecule is probably a protein, prolamine-like, taking into account the larger solubility in alcohol–water solution (Wall, 1964; Gheyasuddin, Cater & Mattil, 1970): this assumption is in good agreement with the data reported in Table 4.

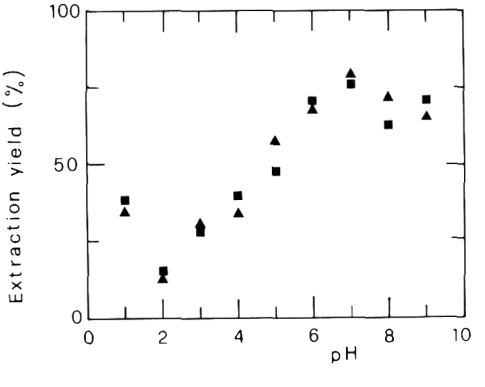

Figure 4. Extraction yields as a function of pH (extraction time 1 hr, S/P ratio 400 ml/g). ▲, HCl or NaOH solutions, ■, buffer solutions.

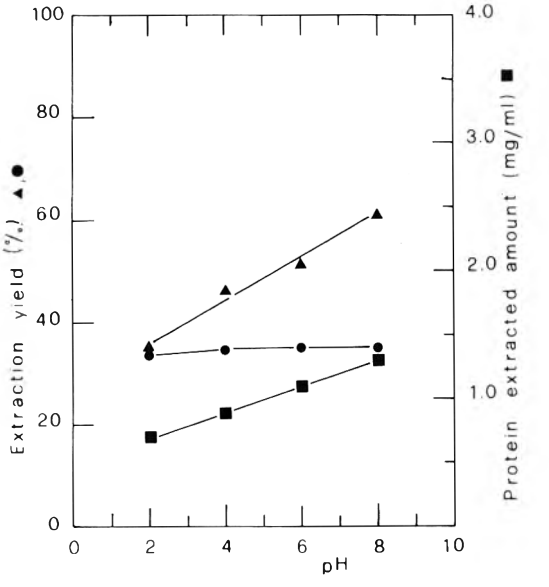

Figure 5. Extraction yields ▲ and protein contents ■ as a function of pH for the methanol-buffer solution mixtures (60:40 v/v) (S/P ratio 10 ml/g, extraction time 1 hr). ● extraction yields after acidification until pH 2.0, 24 hr at 4°C, and centrifugation for 15 min at 5000 rev/min (3000 g).

As regards temperature, the extraction yields increase up to about 40°C and then drop on account of the setting in of degradation phenomena; the effect, however, is quantitatively different for each solvent (e.g. of the alcohols, it is greater for ethanol than for methanol).

Serial extractions

Figure 6 shows the total yields for serial extractions, with distilled water, of the whole of the pigments and of the fraction remaining in solution after acidification to pH 2.0 for 24 hr at 4°C. It can be noted that the maximum yield is reached quickly, at the third extraction, and that the free anthocyanins represent about 20% of the pigments present.

Table 4. Nitrogen and protein contents in the sunflower powder and extracts (S/P ratio 10 ml/g). Solvent: (A) distilled water, (B) methanol–buffer solution mixture at pH 8.0

		Extract				
Powder		Before acidification		After acidification until pH 2.0		
N%	Protein %	N g/l	Protein g/l	N g/l	Protein g/l	
0.716	4.560*	0.188	1.200	0.017	0.106	(A)
		0.200	1.300	0.098	0.650	(B)

* Calculated according to Horwitz (1960b).

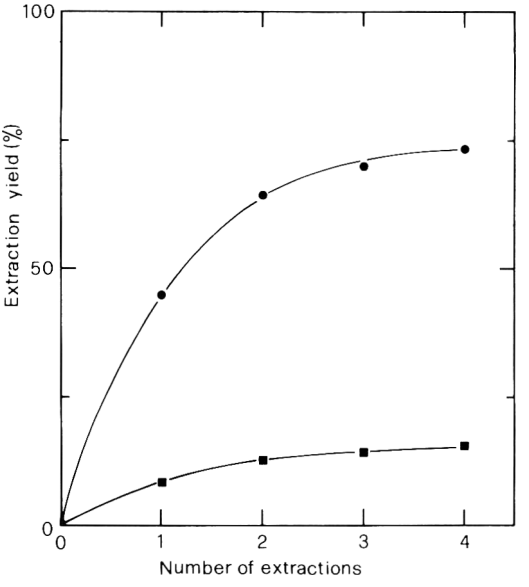

Figure 6. Extraction yields for serial extractions with distilled water (S/P ratio 10 ml/g, extraction time 4 hr). ●, before, and ■, after acidification until pH 2.0, 24 hr at 4°C, and centrifugation for 15 min at 5000 rev/min (3000 g).

Electrophoresis

When subjected to electrophoresis, the pigments extracted in distilled water and precipitated by acidification to pH 2.0 confirmed that the anthocyanins in them are bonded to a negatively charged molecule at pH 8.9. The reaction with the specific staining gives reason to believe that this molecule is a protein.

Conclusions

The husks of some cultivars of sunflower are a good low-cost source of anthocyanins. It has been shown how it is possible to reduce drastically by mechanical means the quantity of material to be extracted, with good pigment yields.

The most important extraction parameters are the polarity and the acidity of the solvent. The results relative to ethanol and distilled water appear to be of particular interest in view of the possible application in the food industry: with S/P ratios up to 35, distilled water gives extremely interesting extraction yields and purity ratios, offering also the possibility of increasing the yields by means of 2–3 serial extractions.

The greater part of the anthocyanins, readily precipitable by acidification to pH 2.0, are bonded to a macromolecule which, as the results of the electrophoresis and reactions with specific stainings suggest, is a prolamine-like protein, taking into account also the solubility trend in hydroalcoholic mixtures.

Acknowledgments

We wish to thank Dr S. Palmieri of the Experimental Institute for Industrial Cultivations (Bologna, Italy) and Dr D. Paganelli for their contributions to this research.

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(*Received 26 November 1982*)

J. Fd Technol. (1983) **18**, 639–649

Palatability of meat from electrically stimulated carcasses of yearling and older entire-male and female sheep

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D. M. DUGANZICH*

Summary

Pasture-fed male and female sheep ranging in age from yearling to more than 4 years old were slaughtered at various times throughout the year. Carcasses were electrically stimulated and frozen.

Flavour and odour of the cooked meat were evaluated with three different taste-testing procedures: an analytical laboratory taste panel, an in-house consumer taste panel and a mass consumer taste panel. Tenderness was assessed with both a tenderometer and the in-house consumer taste panel.

None of the panels found any differences between the meat from yearling rams or ewes. The laboratory panel detected foreign flavours more commonly in the meat from older rams than older ewes. These foreign flavours were not considered objectionable by the other two taste panels; in fact, meat from the older rams was often preferred to the meat from the older ewes. There were no differences in objective tenderness among sheep from any age group. Taste panel results, however, showed meat from the older sheep to be less tender than that from the yearling sheep, but found no sex-related differences in tenderness between the mutton of similar-aged animals.

Introduction

The scientific evidence for the existence of sex-related odours or flavours in meat from male sheep (rams) is inconclusive. Several researchers have found no unpleasant flavours or odours in the cooked meat from rams older than 15 months (Rhodes, 1969; Kirton & Paterson, 1972; Wenham *et al.*, 1973; Anon, 1973; Alvi,(1980), although such meat has been reported as less tender than ewe mutton when not conditioned. However, Charlet (1969) suggested that meat from rams older than 1 year has a strong 'wool' flavour which many consumers dislike and Crouse *et al.* (1978) reported inferior aroma scores from the heated fat of ram lambs compared to that of ewes. Much of the

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0022–1163/83/1000–0639 $02.00

taste-panel research to date has been based on analytical laboratory panels consisting of only a few panel members.

The New Zealand Meat industry regards the meat of rams over 1 year old as being sufficiently inferior to that of similar-aged wethers and ewes that slaughterhouses pay only 10–30% of the price per kilogram for ram carcasses compared with what they offer for all other classes of sheepmeat. Carcass cut-out information from previous trials (Fourie, Kirton & Jury, 1970; Kirton & Paterson, 1972) showed a higher proportion of muscular tissue in ram carcasses and only small differences in the distribution of cuts between ewe and ram carcasses. The higher proportion of muscular tissue in ram carcasses and the small differences in cut-out compared to ewes applied in older animals as well as younger ones. Thus, there is little justification for prejudice against ram animals based upon carcass characteristics. However, pelting older ram carcasses is more difficult because a greater force is required to separate the pelt and underlying fell layers of the skin (Roberts, personal communication).

The lower price for ram meat is usually justified on the grounds of the inferior shape of the ram carcass and the believed inferior palatability of the meat. Ram meat is believed to be most strongly flavoured during the mating season.

The present experiments compared the palatability of meat from rams and ewes 1 year and older by using a variety of taste-test procedures and relatively large numbers of panelists. The animals were slaughtered at various times throughout the year. Because some carcass classification systems separate yearling sheep carcasses (hogget) from older sheep carcasses (mutton) (e.g. Kirton & Colomer-Rocher, 1978), this age separation was used for the carcasses in this experiment, and animals were classed as yearling and 'old'.

Materials and methods

Animals

In trial 1, ten each of yearling rams, yearling ewes and rams over 2 years old, and nine ewes over 2 years old, all of the Romney breed, were selected from a research farm and transported to the research-station abattoir for slaughter on 11 February 1981. (The sheep mating season in New Zealand normally starts in March/April.)

For trial 2, nine Romney rams over 2 years old were removed from the ewe flock where they had been mating and together with ten similar-aged Romney ewes from the same research farm were slaughtered in the experimental abattoir on 24 March 1981. Ten Romney yearling rams straight from the mating paddock together with ten Romney yearling ewes were similarly selected and slaughtered on 8 May 1981.

In trial 3, ten rams (nine Romneys and one Perendale×Romney) and ten Romney ewes, all 4 years old or more, were selected from research farms different from these in trials 1 and 2 and were transported to the research

abattoir for slaughter on 8 July 1981. This batch of rams was therefore processed some time after the end of the mating season.

For trial 4, ten rams (nine Romneys and one Border Leicester×Romney) and ten Romney ewes over 2 years old were selected from the same research farm as the first two trials and slaughtered on 23 December 1981. Eleven each yearling Romney rams and ewes were selected from the same research farm for slaughter on 22 January 1982.

Slaughter and carcass-processing procedures

Animals were slaughtered and dressed using standard procedures. Immediately after dressing, all carcasses were electrically stimulated as described by Hagyard, Hand & Gilbert (1980) and held in a cooler at 5°C for at least 24 hr in trials 1–3 and at 3°C in trial 4 before being either frozen or sectioned. Carcasses were halved down the backbone. The hind legs, each containing one lumbar vertebra, were cut from the carcasses. An approximately 30 cm strip of the loin containing the *M. longissimus dorsi* was cut from the left side of each carcass. This loin was packaged in a polythene bag and stored frozen (−35°C) until required for laboratory taste panel and tenderometer analyses. Shoulder roasts were removed from eight of the rams and eight of the ewes slaughtered on 24 March 1981.

In-house consumer panel

Leg roasts from all animals were distributed to families experienced in participating in a consumer-type taste panel. Families were unaware of the identity of their roasts other than that they were sheepmeat. Depending on the size of the leg and the size of the participating family receiving each roast, it was possible to get one or two roasts per leg, and with two legs available per animal this allowed the distribution of two to four roasts per animal. In each trial, each participating family received a ram/ewe pair of roasts in random order over a 2 week period from either the yearling or older-sheep age group with the same families receiving the same age-group sheepmeat in each trial. Families were asked to roast the meat in an oven preheated to 163°C for 55–65 min/kg meat or until the meat was considered cooked (not all household oven controls are accurate). Each panel participant was asked to taste a slice of cooked meat before the addition of salt, gravy or sauces normally added to a meal and to record opinions, without discussion, on a standard taste panel form based on the nine-point hedonic scale of Peryam & Pilgrim (1957). They were asked to judge flavour (like extremely to dislike extremely), tenderness (extremely tender to extremely tough), juiciness (extremely juicy to extremely dry) and to give their overall opinion of the meat (like extremely to dislike extremely). The comments were then converted to numerical scores such that the higher the score the better the

meat was liked, with five representing the mid-point (neither like nor dislike, etc.) of the scale.

From one to three people tasted the meat from each family's roast. Family scores were averaged to give a roast score and then the scores from all the roasts from each animal were averaged to give the score for each animal.

Scores for each characteristic in each trial were tested by analysis of variance.

Mass consumer panel

Any flavour/odour problem with the cooked meat from older rams would be most likely to show up in meat from the group slaughtered just out of the mating paddock in trial 2. Frozen shoulder roasts from eight rams and eight ewes from trial 2 were thawed and then roasted in a convection oven at 163±5°C until the centre of the roast reached 80°C. Roasts were cooled at room temperature for 1−2 hr before being wrapped in aluminium foil and stored at 4°C until used.

Meat from each roast was chopped into pieces approximately 2×2×1.5 cm with pieces containing more than 30% estimated visual fat being discarded. The panel method used was based on that described by Moore, Jury & Bass (1978). The samples were served at room temperature.

Each ram was paired with every ewe giving sixty-four pairs per replicate, and each pairing was replicated fourteen times. The order of serving of each paired ram and ewe sample within each series of replicates was randomly assigned in the first replicate. In each succeeding replicate the order of serving the ram or ewe meat was reversed. Samples making up each pair were coded A and B. Participating tasters were asked to ignore all attributes of the meat except flavour and to indicate their flavour preference for sample A or B or whether they had no preference between the samples. The trial involved 896 tasters.

The percentage of tasters preferring the ram sample was analysed by fitting log-linear models (Baker & Nelder, 1978), testing for order of presentation and ram and ewe carcass effects plus their interactions. Tasters expressing no preference were omitted from this analysis.

Laboratory taste panel

Loins from all animals were thawed for 1 hr in a water bath at 40±2°C. The M. longissimus dorsi and its covering fat were excised. Minced lean muscle, containing 25% w/w added fat tissue was prepared and cooked according to the methods described by Winger & Pope (1981). An analytical panel of fifteen to twenty members, trained to detect rancid flavours in lamb, was asked to detect any non-sheepy, non-meaty foreign flavours present in the samples. Panelists were then to rate the intensity of these foreign flavours on a nine-point scale, where 1 indicated no noticeable foreign flavour and 9 indicated an extreme intensity.

All panelists received four samples per tasting session, including one sample from each of the animal treatments under investigation. Tasting order was randomized among panelists.

Results from all panelists were pooled and the means statistically analysed using analysis of variance.

Objective assessment of tenderness

Before the loin was thawed for the laboratory taste panel, a 2.5 cm chop was cut from the hind-leg end. This chop was cooked from the frozen state in a water bath at 80±1°C for 60 min, then cooled in an ice bath. A sample with a cross-sectional area of 1.0 cm^2 was cut from the *M. longissimus dorsi*. This sample was sheared across the muscle fibres using a tenderometer described by Macfarlane & Marer (1966).

Statistical comparison of scores from different panels

The mean scores from the three panels were ranked within each measured attribute. Spearman's rank correlation coefficients were calculated for each attribute in comparison to the mass consumer panel preference scores (Moroney, 1951).

Results and discussion

In-house consumer taste panel

The panel results in Table 1 are based on 766 taster observations for each palatability attribute. The outstanding feature of these results is the absence of any sex effects on the flavour, tenderness, juiciness or overall acceptability of the meat. There was no suggestion of any undesirable flavour or odour associated with the ram meat produced at different times of the year; not even in trial 2 when the rams were slaughtered straight from the mating paddock. Although the panel questionnaires had a space that allowed for comments on unpleasant cooking odours, the very few comments received were equally distributed between sexes.

Tenderometer shear force values indicated no sex effects on the tenderness of the *M. longissimus dorsi*. Mean shear force values in trial 1 were lower than in trial 4, probably because in trial 4 a more efficient and colder chiller was used to hold the carcasses immediately post-slaughter. Shear force values differed much more than panel scores between these two trials. Possible reasons include the fact that the muscle tested by tenderometer is known to be susceptible to cold shock while many leg muscles are less susceptible. In addition, the leg muscles are normally sliced thinly across the grain before being tasted whereas the tenderometer samples were thicker with the tenderometer shearing across the muscle fibre axis.

Table 1. In-house consumer panel scores and objective shear force values for sheep meat from groups of animals slaughtered at different times of the year. (The higher the score the higher the palatability)

Palatability factors	Yearling		Old*		Differences		
	Ram	Ewe	Ram	Ewe	Sex R–E	Age Y–O	s.e.
Trial 1							
No. animals	10	10	10	9			
Tenderness	7.1	7.1	5.9	5.7	0.04	1.33§	0.31
Flavour	7.2	7.1	6.8	6.8	0.05	0.33	0.27
Juiciness	6.5	6.4	6.0	5.9	0.09	0.50†	0.26
Overall acceptability	7.5	7.4	6.5	6.6	−0.08	0.92§	0.28
Shear force values	12.8	21.9	20.7	15.4	0.8	2.0	2.1
Trial 2							
No. animals	10	10	9	10			
Tenderness	6.4	6.4	5.4	5.9	−0.21	0.76‡	0.35
Flavour	7.1	6.5	5.8	6.2	0.15	0.80‡	0.35
Juiciness	6.7	6.4	5.3	5.8	0.09	1.01§	0.25
Overall acceptability	7.1	6.7	5.7	6.1	0.05	0.96§	0.34
Trial 3							
No. animals			10	10			
Tenderness			6.0	5.7	0.26		0.66
Flavour			6.8	6.5	0.28		0.64
Juiciness			6.3	5.9	0.39		0.49
Overall acceptability			6.7	6.4	0.25		0.52
Trial 4							
No. animals	11	11	10	10			
Tenderness	6.8	6.9	6.5	5.8	0.26	0.67‡	0.32
Flavour	7.2	7.1	7.1	6.5	0.35†	0.34†	0.20
Juiciness	6.1	6.4	6.3	6.0	0.01	0.12	0.23
Overall acceptability	7.1	7.2	6.9	6.2	0.22	0.58‡	0.25
Shear-force values	35.9	34.0	33.8	41.4	−2.6	−3.4	1.84

* Two or more years old;
† $P < 0.10$;
‡ $P < 0.05$;
§ $P < 0.01$.

The one effect that emerged from the in-house consumer trials is that the roasts from the yearling animals were more acceptable than those from the older sheep, mainly because the meat from the younger animals was judged to be more tender. However, the results also suggest that the flavour of the meat from the yearling animals was preferred.

Mass consumer panel

None of the roasts had undesirable cooking odours.

The number of preferences expressed for meat from the various rams and ewes is shown in Table 2. The 10% of tasters who expressed no preference

Table 2. Flavour preferences of mass consumer panel members for meat from old rams or ewes

Ram No.	Ram pref.*	Ewe pref.	No. pref.	Ewe No.	Ewe pref.†	Ram pref.	No. pref.	Total pairings
1	54	45	13	9	41	64	7	112
2	69	35	8	10	43	59	10	112
3	61	37	14	11	50	55	7	112
4	52	49	11	12	45	50	17	112
5	31	69	12	13	40	61	11	112
6	50	49	13	14	44	59	9	112
7	69	33	10	15	51	49	12	112
8	63	38	11	16	41	52	19	112
Total	449	355	92		355	449	92	896

* This half of the table gives the number of tasters preferring the listed ram in comparison with all eight ewes this ram was paired with over the fourteen replicates.

† This half of the table gives the number of tasters preferring the listed ewe in comparison with all eight rams this ewe was paired with over the fourteen replicates.

between the two samples tasted were ommitted from the analysis. Log-linear model analysis showed significant presentation order and ram carcass effects, but none due to ewe carcasses or any interaction. Averaging over order of presentation, the preferences expressed for the meat from the eight ram carcasses tasted were 68, 66, 62, 62, 55, 51, 51 and 31%. The preference expressed for each of the first four rams was significantly higher than the 50% value expected if, on average, the tasters had no real preference between the rams and the ewes. The value for the last ram was significantly less than the 50% value. This one disliked ram (31% preferences) represented a relatively fat-free animal that would not normally have been considered for table meat. The results indicate that ram mutton has no flavour problems compared to ewe mutton.

Laboratory taste panel

The mean panel scores for foreign flavour intensities are given in Table 3. The data from the first trial cannot be compared with those in the remaining three trials, because the scoring procedures were considerably different. The first trial attempted to evaluate sheepy and meaty flavours as well as off flavours. This proved too complex for the panelists to score satisfactorily. After the first trial, the intensities of the sheep and meat flavours were not scored: only the foreign flavours were scored. Young animals had no detectable foreign flavours irrespective of animal sex.

The minced meat from old rams in trials 2–4 usually exhibited a foreign flavour the panelists consistently described as 'musky, haylike, mousey, musty, stale'. Not all old-ram meat exhibited this flavour: fourteen of the twenty-nine animals tasted were considered to have no significant foreign flavours. The distribution of foreign flavour intensities for old rams and ewes

Table 3. Mean foreign flavour scores of trained laboratory panellists for rams and ewes of varying ages

Trial no.	Yearling ram (mean ± s.e.)	Yearling ewe (mean ± s.e.)	Old† ram (mean ± s.e.)	Old† ewe (mean ± s.e.)
1*	2.0 ± 0.3	1.4 ± 0.2	1.4 ± 0.1	1.6 ± 0.1
2	2.3 ± 0.2	2.3 ± 0.4	3.4 ± 0.3	2.4 ± 0.2
3	—	—	3.1 ± 0.3	1.8 ± 0.1
4	2.8 ± 0.1	2.3 ± 0.1	3.4 ± 0.2	3.0 ± 0.2
Overall	2.5 ± 0.1[a]	2.3 ± 0.2[a]	3.3 ± 0.1[b]	2.4 ± 0.1[a]

* The score format for this trial was different from the remaining three trials. Data from this trial cannot be compared directly with the other data and is not included in 'overall' means.

† Two years old or more.

[a, b] Means with different superscripts are significantly different at 95% level of confidence, as measured by Newman–Keuls sequential range test (Steel & Torrie, 1960).

is shown in Figure 1; higher intensities being more commonly found in the ram meat.

Minced meat from the old ewes was also variable in its taste (Fig. 1). The meat from six of the thirty old ewes tasted contained significant intensities of

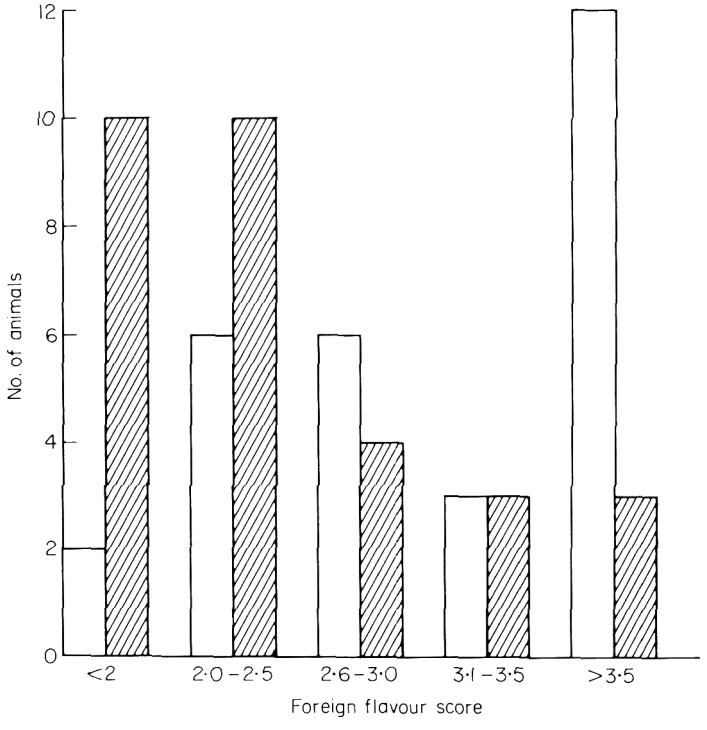

Figure 1. Numbers of old rams and old ewes in trials 2–4 with foreign-flavour scores in the different intensity ranges.

foreign flavours, which were described in an identical manner to the flavour found in rams. The remaining samples were considered to have no significant foreign flavour.

Panel comparison

A comparison of the results from the three panels is given in Table 4 for the older animals in trial 2. These were the only animals whose meat was common to the three panels. The three panels only had the flavour assessment in common. Amerine, Pangborn & Roessler (1965), however, have shown that consumers scoring for one palatability attribute can be influenced by their score for a different attribute. Therefore, the tenderness and overall acceptability scores from the in-house consumer panel have been included. It appears that the tenderness of the samples scored for flavour by consumers in the mass panel may have influenced their flavour assessment; this problem would not apply to the laboratory panel where minced samples were tasted.

For the rams, the mass consumer panel rankings for preference and the in-house consumer panel rankings for acceptability were significantly correlated ($P<0.001$). There was no significant correlation for the ewes. The

Table 4. A comparison between the scores from the different panels for the meat from older rams and ewes in trial 2

Animal No.	Mass panel preference (%)	In-house consumer panel scores*			Laboratory panel foreign flavour intensity†
		Overall acceptance	Tenderness	Flavour	
Rams					
7	68	6.5	7.0	5.5	3.0
2	66	6.2	5.5	6.0	4.3
3	62	6.8	5.2	7.3	3.2
8	62	6.3	5.5	6.8	4.6
1	55	5.9	6.4	5.3	4.7
4	51	5.3	4.8	5.2	1.7
6	51	5.8	6.3	6.0	2.4
5	31	1.8	2.0	3.0	3.7
Ewes					
15	51	7.3	7.8	7.2	1.6
11	48	7.3	6.0	6.8	2.1
12	47	5.5	4.2	6.8	3.4
16	44	6.7	5.7	5.7	2.8
14	43	5.0	5.8	5.0	1.9
10	42	5.4	4.5	6.5	1.7
13	40	3.8	4.5	3.0	3.5
9	39	6.8	7.0	6.8	2.1

* Nine-point hedonic scale (1 = worst; 9 = best);

† Nine-point structured scale (1 = no foreign flavour; 9 = extreme).

remaining correlations between the mass consumer panel rankings for preference and the other in-house (flavour, tenderness) and laboratory (flavour) panel scores were not significant for either the ewes or rams. Thus, factors other than flavour may have influenced the mass panel preference score. Both consumer panels agreed that the flavour of ram No. 5 was very poor. Given that the roasted meat for the in-house consumer panel was tasted hot, meat for the mass consumer panel was served cold and that for the laboratory panel was a warm, boiled mince sample, the lack of a common ranking for flavour in particular is not surprising. This applies particularly to the ewes where the flavour preferences were relatively uniform with none differing significantly from 50% preference level in the mass consumer panel. Amerine, Pangborn & Roessler (1965) have observed that laboratory panels often detect flavour differences that appear not to influence the preferences of consumer panels.

Overall assessment

None of the panels detected any palatability differences between the meat from the yearling rams and ewes. The in-house consumer and mass consumer taste panels detected no foreign flavours associated with the meat from older rams. The laboratory taste panel, however, found a higher incidence of foreign flavours in this meat, which may have to be considered if ram mutton is intended for consumers not accustomed to eating sheep meat. It should be noted that in the course of a normal meal, meat may have salt, spices, sauces or gravies added and will be mixed with other food items. Hence, any flavour features of old-ram meat would be much less conspicuous than in these trials.

The meat from pasture-fed yearling rams is clearly suitable as a table meat. These results indicate that the carcasses of older rams should be as suitable as those from older ewes for manufacturing purposes and they can be as satisfactory a source of table meat as older ewes in markets where the latter are normally eaten. Wenham *et al* (1973) have established that the meat from conditioned and aged older ewes and rams is satisfactory for roasting.

Acknowledgments

The technical assistance of Mr E. G. Woods and Miss C. G. Pope is gratefully acknowledged. This study was stimulated by a request for information by the New Zealand Meat Producers' Board.

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(*Received 7 December 1982*)

J. Fd Technol. (1983) **18**, 651–656

Measurement of fat and sucrose in dry cake mixes by near infrared reflectance spectroscopy

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Summary

A feasibility study was carried out to assess the potential of near infrared (NIR) reflectance spectroscopy for the measurement of fat and sucrose in dry cake mixes. The calibration of the NIR instrument was carried out in a research laboratory and then assessed under quality control conditions in the factory laboratory. It was possible to measure fat with an accuracy ($\pm 2\sigma$) of $\pm 3.4\%$ for products with a fat content of 8–25% compared with $\pm 0.76\%$ for the precision of the Soxhlet procedure. In the case of sucrose the accuracy was $\pm 5.4\%$ for products with a sucrose content of 10–40% compared with $\pm 2.0\%$ for a high pressure liquid chromatography (hplc) method. It must be concluded, therefore, that while NIR offers a quicker, simpler method of quality control, this is at the expense of accuracy.

Introduction

Near infrared (NIR) reflectance spectroscopy has been used to measure the fat content of oilseeds (Miller, 1979), meat (Ben-Gera & Norris, 1968) and pasta (Kaffka, Norris & Rosca-Kiss, 1982) and Giangiacomo *et al.* (1981) have demonstrated the measurement of sucrose in mixtures of pure sugars. Near infrared is quite widely used in quality control in the food industry particularly for cereals and cereal products such as flour (Osborne, Douglas & Fearn, 1982) and therefore it is a logical development to extent its use to ceral-based processed food products.

A study was undertaken to assess the potential of NIR for the control of major ingredient levels (other than flour) in dry cake mixes. The experiment was based on calibrations for real products not model systems and validation of these calibrations carried out in an actual factory quality-control environment.

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0022–1163/83/1000–0651 $02.00

Materials and methods

Samples

A total of 231 samples were used for calibration and prediction (Table 1). Fat was determined in duplicate on 211 samples by the Soxhlet method (4 hr using petroleum spirit 40–60°C boiling range) and sucrose in duplicate on 108 samples by hplc (using refractive index detector; aqueous extraction and acetonitrile solubilization of the sucrose).

Table 1. Calibration and prediction samples for NIR measurement of fat and sucrose in cake mixes

	Fat			Sucrose		
Product	Range (%)	Calibration (*n*)	Prediction (*n*)	Range (%)	Calibration (*n*)	Prediction (*n*)
Bread	2.8–3.4	20	14			
Scone	8–11	20		13–16	20	
Sponge	12–17	48	57	27–41	48	19
Shortbread				15–20	20	
Shortcrust	22–26	24	28			
Total	3–26	112	99	13–41	88	19

Near infrared reflectance measurement

The instrument used was an InfraAzer 400R (Technicon Instrument Co. Ltd, Basingstoke, Hants, U.K.) fitted with the following filters, 1445, 1460, 1680, 1734, 1940, 2082, 2100, 2139, 2180, 2280, 2230, 2310 nm. Samples were scanned without preparation using the procedure already described (Osborne, Douglas & Fearn, 1982). For the calibration samples only one scan per sample was taken but for the prediction samples each was scanned by three different operators and in three orientations (by rotating the sample cup by 120° between successive readings) making nine observations per sample.

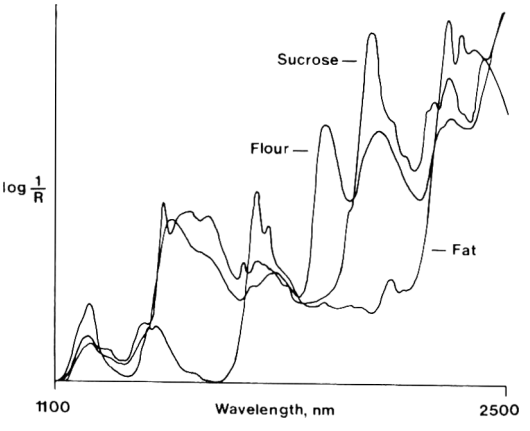

Figure 1. The NIR spectra of fat, flour and sucrose.

Statistical analysis

Calibrations for fat and sucrose were obtained by multiple linear regression of Soxhlet and hplc values respectively on all 12 log reflectance values then searching for minimal adequate subsets (Osborne, Douglas & Fearn, 1982). The calibrations were tested by comparing predicted NIR values with the appropriate reference values on further samples and computing the standard deviations of differences. Operator and orientation effects were examined and the replication of NIR prediction results were assessed by analysis of variance.

Results and discussion

The major components of cake mixes (Fig. 1) have sufficiently characteristic NIR spectra to enable their measurement in admixture to be a viable proposition. The major spectral characteristics of fat are due to the long chain fatty acid moiety which gives rise to a preponderance of CH_2 absorption bands: 1200 nm (CH_2 *str.* second overtone), 1734 nm (CH_2 *str.* first overtone, asym.), 1765 nm (CH_2 *str.* first overtone, sym.), 2310 and 2345 nm (CH_2 *str.*/CH_2 bend combinations). The main bands in sucrose, apart from those arising from C–H vibrations, are due to O–H overtones and combinations: 1445, 1500 and 1580 nm (first overtone of O–H *str.* in various stages of hydrogen bonding), 2082 nm (O–H *str.*/O–H bend combination). The calibration equation for fat involved log reflectance data at 1734, 1445, 1680, 2230 nm while that for sucrose involved 2100, 2310, 1940, 1734, 1445 nm. (The use of 2100 and 2082 nm gave rise to equivalent results; therefore, since the 2100 nm filter is standard on the 400R instrument and 2082 nm is not, 2100 nm was used.)

The calibration for fat (Fig. 2) was good within, as well as between, products, except that the variability in the bread mixes is too small to judge for

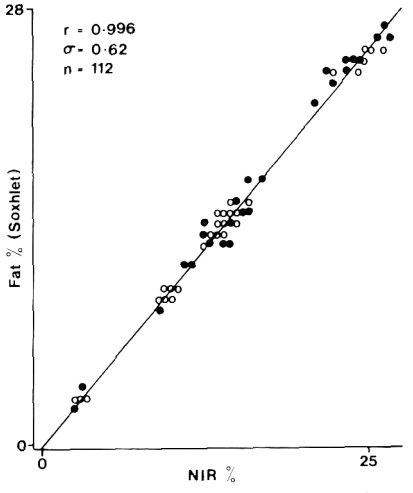

Figure 2. Calibration for fat in dry cake mixes by NIR.

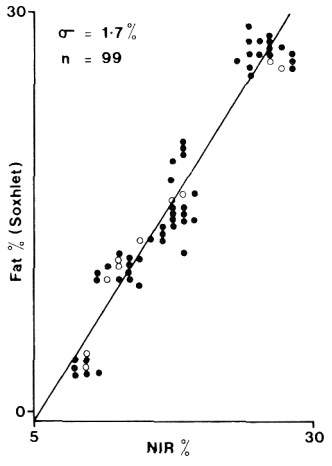

Figure 3. Prediction of fat in dry cake mixes by NIR; the 45° line includes the bias correction.

them. The pooled standard deviation of replicates (precision) for all 211 duplicates by the Soxhlet method was 0.38%, thus the accuracy of NIR in this case is approximately twice the precision of the reference method. For prediction experiments to assess the calibration, the constants had to be transferred to a second instrument in a different location and thus an adjustment to the bias (F_0) had to be made and the results presented are corrected for this bias. The standard deviation of differences between the NIR and Soxhlet results was 1.7% (Fig. 3) and this figure is rather worse than the calibration standard deviation and also worse than prediction standard deviations obtained with biscuits and biscuit doughs (Osborne *et al.*, 1983). This cannot be accounted for by the precision of the reference method or the precision of NIR (s.d. of replicates 0.29%) or because the prediction

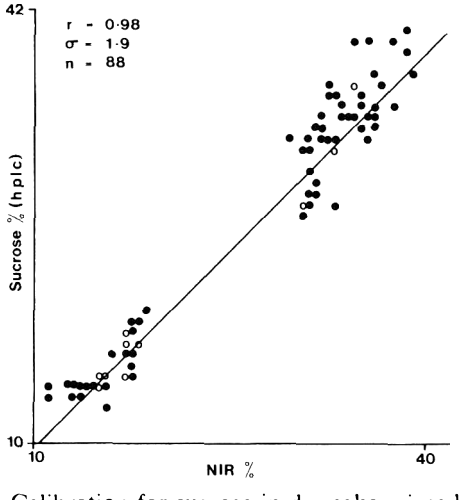

Figure 4. Calibration for sucrose in dry cake mixes by NIR.

experiments were carried out with operator and orientation variations. The only explanations for the poor prediction performance are that the nature of the fat had changed from the calibration products or the calibration equation is unsatisfactory. In the case of measurement of fat in biscuits by NIR the calibration involved only two wavelengths 1734 and 1550 nm (Osborne *et al.*, 1983) but 1550 nm was not available to the instrument used in the present study.

The calibration for sucrose is shown in Figure 4 but from the duplicates of the hplc method it is apparent that the standard deviation of a single determination is proportional to the mean, the average across the range being 1.04%. Therefore it is possible to achieve a better calibration for the samples with the lower sucrose content (scone and shortbread mixes) by treating them separately when a residual standard deviation of 0.89% was achieved; there was, however, no improvement to the sponge mixes by treating them separately. The combined calibration is still quite satisfactory when compared with other results (Giangiacomo *et al.*, 1981; Osborne *et al.*, 1983) and with the hplc method. The prediction of sucrose (Fig. 5) is as good as might be expected since only nineteen samples were available (the residual s.d. and s.d. of differences are not significantly different at the 5% level). However, the range of the prediction samples (34–43%) was much narrower than that of the calibration samples (10–40%) since only sponge mixes were available and in that context 2.7% is such a very large standard deviation compared with the range, that it is doubtful if sucrose is really being measured. The precision of NIR sucrose determination was 1.2%—about the same as that of the hplc method.

The NIR readings were taken at different orientations to check for effects due to lack of homogeneity particularly with regard to sucrose which is present in a granulated form. In fact, there was no orientation effect except for possibly chocolate sponge for sucrose and white bread for fat but the

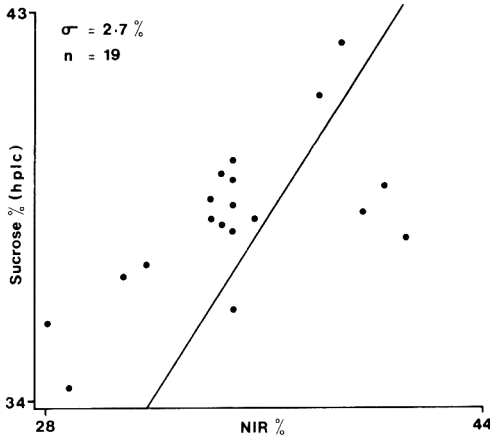

Figure 5. Prediction of sucrose in dry cake mixes by NIR; the 45° line includes the bias correction.

differences although statistically significant were very small. Similarly, in the case of operator effects, statistically significant differences were observable but these were very small except in two cases both involving chocolate sponge. The figures given for replication of NIR have been adjusted to compensate for the significant operator and orientator biases although the uncorrected figures were virtually the same. It is clear that replication of NIR is at least as good as that of the hplc and Soxhlet methods even when operator and orientation effects are taken into account.

Conclusions

Near infrared is equal in terms of precision of fat and sucrose determination in cake mixes to Soxhlet and hplc methods, respectively. However, because NIR is a predictive technique relying on an empirical calibration equation it is necessary to consider the accuracy as well as the precision when compared with the methods against which NIR was calibrated. In neither case is the accuracy satisfactory, but it must be borne in mind that this assessment was not carried out using model samples in a research environment but in the real world of a factory quality-control department.

Acknowledgment

We wish to thank the Analytical Department, Lord Rank Research Centre for providing the hplc and Soxhlet data, and the Directors of RHM Foods Ltd, for permission to publish.

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Book reviews

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Oxford: Pergamon Press, 1981. ISBN 0 08 025365 2. £175.00.

'Biotechnology is a multidisciplinary field.' So begins the Preface to these big and expensive volumes and a glance through the contents list is sufficient to make the point. There is something there for everyone with an interest in the area, but it is flung together in such a hotch-potch of material, style, quality and focus that it is difficult to visualize how the volumes will be used.

To be fair to the editors and publishers, it is not easy to see how a series of papers presented at the Sixth International Fermentation Symposium (Vols. 1–3) and the Fifth International Symposium on Yeasts (Vol. 4) in London, Ontario in 1980 could be otherwise. The great merit of this meeting seems to have been that it brought together a wide variety of experts in what one might now call 'classical biotechnology' at a time when 'new biotechnology' was becoming a household word. In Volume 1, for example, the number of papers in the trendy areas of microbial genetics (4), plant and animal cell culture (6) and immobilized enzymes or cells (9) are swamped by those on microbial culture (13), hydrocarbon oxidation (6), continuous culture (18), fermentation technology (37), and bioreactors (18). Papers in Volume 2 deal with lignocellulose degradation (8), ethanol (19) and other chemicals (7), biogas (10), SCP (16), fermented foods (16) and waste treatment (16) and Volume 3 concentrates on antibiotics (34), food additives (9), mycotoxins (5), biopolymers (8) and microbial enzymes and bioconversions (31). Here again the bulk of the papers represent comfortable conventional approaches but the discerning reader will extract a few papers that have potentially revolutionary impact.

Volume 4 is different and could be purchased by many separately from the other three volumes since it presents a comprehensive survey of both the science and the applications of yeasts. It does, of course, predate the big

explosion in yeast genetics of the last three years, but the book will be invaluable to both academic and industrial scientists as a summary of the ground from which this explosion was launched.

Who then will buy and who will read these volumes? Few scientific libraries will be able to resist pressure to stock it, if only for the five to ten papers relevant to the interests of their particular staff. For students the books will be hard grind, because few papers make much concession to the novice in their field and the layout and rudimentary index seem designed to frustrate the searcher after truth. But tackle it they will, because embedded in these forbidding and rather unattractive pages are facts and interpretation that they will find nowhere else. Fortunately the books are well bound and the paper is robust, because the pages are certain to be well-thumbed.

One warning to the potential reader is necessary. The title 'Advances in . . .' has come to suggest a style and format of scientific review that most of us have grown to value and depend upon. It is unfortunate that such a title has been stolen for these heterogeneous collections of papers. A true 'Advances in Biotechnology' series of reviews in depth of topics dealt with in these volumes is needed; these four books are merely source material for what must yet come.

Brian S. Hartley

Fermented Foods. (Vol. 7, Economic Microbiology). Ed. by A. H. Rose.
London: Academic Press, 1982. Pp. xiii+337. ISBN 0 12 596557 5. £29.20; US$60.00.

This is the seventh book of a series intended to introduce the reader to the economic impacts of microbiology. Fermented foods is a relevant subject in such a context since they are part of the cultural background of mankind. There is an increased concern towards the usually energy-efficient methods of fermentation to reduce the enormous food losses and to increase shelf life to commercially compatible periods in the developing countries as well as in the more developed world. Bearing this in mind the editor aimed to introduce the reader to the fundamentals of the 'microbiology involved in the production of the major fermented foods that are today manufactured world-wide'. Alcoholic beverages were described in Volume 1 of the series.

The introductory chapter seems to the reviewer a good and very concise exercise on what we know and what we don't know about the microbial activity and the control of food fermentation processes. 'The microbiology behind many of them, however, is remarkably poorly understood, despite their ancient origins.' This chapter is followed by another with some interesting but broad considerations about a wide range of fermented foods found mainly in the Oriental culture. Soy sauce and miso are described in a separate chapter. It is an interesting and readable introduction to processing

technology and microbiology with valuable statistical data of world production and trade (1976–78) of both products. The authors of the chapters on breadmaking and cheeses succeed in providing a good account of the fundamentals of microbial activity and its relevance to processing of such products. Fermented milks are dealt with briefly in a separate chapter. The chapter on fermented vegetables is an entertaining introduction for the student and for those in the food industry and involved with R & D on the needs for research and on the enormous possibilities of applications of modern knowledge of microbial physiology. Two separate chapters deal with coffee and cocoa. Whether coffee should be considered a food fermentation is an open question. Scientific investigation on cocoa is encouraged to 'bring about an improvement of the raw material in those parts of the world where it is not of premium quality'. The last chapter covers processing and economical aspects of yeast extracts.

The book seems to have few errors (e.g. p. 207 'Vescova' should be 'Vescovo'; on p. 239 'faculative' should be 'facultative'; on p. 274 'PICARDO, C. (1934). Anquivos Instituto Biologica Vegetables. 2, 67' should be 'PICADO, C. (1934). Archivos do Instituto de Biologia Vegetal. 1, 67'.

The student and those scientists, technologists, economists and other professionals concerned with food seeking for basic knowledge on food fermentation will find this book a useful tool. The subjects are well indexed and covered by recent and helpful references (with emphasis on literature published from 1976 to 1982) that are presented in an alphabetical order at the end of each chapter thus making it easy to use by the reader interested in further knowledge.

J. F. P. Martins

Nutrition and Killer Diseases: The Effects of Dietary Factors on Fatal Chronic Diseases. Ed. by John Rose.
Park Ridge, New Jersey: Noyes Publications, 1982. Pp. xvii+185. ISBN 0 8155 0902 2. U.S.$25.00.

This interesting volume, with its stirring and provocative title, reviews the evidence for the associations between nutrition and the chronic diseases which can lead to premature death. Several of the fourteen chapters are based on the proceedings of a National Conference on 'Killer Diseases' which was held at the Royal Society of Medicine in London in 1980 under the aegis of the Institution of Environmental Sciences. Additional material, including chapters on an analysis of the effects of heavy metals and vitamin deficiencies on the incidence of birth defects and diseases of middle-age, and the role of mycotoxins in certain idiopathic disorders and tumours in animals and man, rounds off the text to provide a useful contribution to the substantial literature on foods and their effect on health.

In a chapter on the 'diseases of affluence', Denis P. Burkett discusses briefly some of the diseases which are related to a deficiency of dietary fibre. Richard W. D. Turner reviews the dietary factors associated with coronary heart disease and Hugh M. Sinclair contributes an excellent and enjoyable chapter on the role of essential fatty acids and chronic degenerative diseases. Derek Bryce-Smith summarizes the role of heavy metals in degenerative disease, with particular reference to cadmium and lead. Dennis Shapcott discusses the need for research into the metabolism of trace elements, particularly in relation to their association with cardiovascular disease, and pays particular attention to the effects of deficiencies of dietary chromium and copper. Richard Jarrett, in a thoroughly readable chapter on diabetes mellitus, explores the relation between the degree of adiposity and the prevalence of the disease both within and between populations.

Several authors feel honour bound to make specific recommendations for various dietary changes which may produce a beneficial effect. Some go as far as to suggest practical steps to choose the 'right' foods, and at times there is the tendency to blur the distinction between evidence and opinion. However, Arnold Bender restores the balance of the book, and points out forcibly that diet is only one of the many factors that is involved in the aetiology of the degenerative diseases—albeit one that can be changed. Although there is convincing evidence presented throughout the book that nutrition does play an important role in disease, Professor Bender reminds the scientists that the media tend to sensationalize virtually every form of nutrition advice, and that care should be taken to avoid any further reduction in the credibility of public health dietary goals, especially when dealing with highly controversial issues.

The title of the book does little to allay my fears that the topic of nutrition and health will continue to be exploited. Nevertheless, the chapters are all well written and informative and the book should appeal to nutritionists and health professionals.

David P. Richardson

Food Science: A Chemical Approach, 4th ed. By Brian A. Fox and Allan G. Cameron.
London: Hodder & Stoughton, 1982. Pp. xii+370. ISBN 0 340 27863 3. £4.95 (paperback).

The fourth edition of this popular text contains several major changes with respect to earlier editions. In particular those topics related to diet and health have received more attention.

The first three chapters give a brief account of the nature of food systems and a concise description of digestion and absorption. The fourth chapter, some twenty-six pages, covers very basic chemistry and it is perhaps debatable whether this material is, in fact, necessary for the intended readership. The next seven chapters are devoted to the major food components with, perhaps

surprisingly, alcohols and acids providing the subject matter for the first of these. This is followed by a description of oils, fats and colloids and contains some very interesting illustrative material, such as margarine manufacture and the production of dairy products. Carbohydrates are discussed in two chapters with the division into sugars and polysaccharides; here again extensive use is made of illustrative examples, including flour milling and bread baking. The vital importance of amino acids and proteins is emphasized in chapter 9 with a description of protein structure and properties. Chapter 10 is concerned with the determination of inorganic components, namely water and 'mineral elements', the latter being defined as those elements that are left after the body is cremated! Chapter 11 covers the topic of vitamins with descriptions of their physiological functions and their more important food sources.

The final three chapters of the book, which are also the chapters most expanded from previous editions, deal with practical applications of the preceding material. These include effects of cooking, food spoilage and the controversial topic of chemicals in food. This material brings home the relevance of food science and emphasizes its importance to all of us who eat.

In conclusion this is an excellent book as an introduction to food chemistry with a wide range of applications to interest and motivate the reader. It will be eminently suitable for those students studying food science for use as a first text, or indeed for those students with only a peripheral interest in the subject as, for example, students of medicine, nursing or home economics.

R. Macrae

Copper in Biology and Medicine Series. By C. A. Owen.
Vol. 1. Copper Deficiency and Toxicity. Pp. xv+189. ISBN 0 8155 0868 9, £23.00.
Vol. 2. Wilson's Disease. Pp. xx+215. ISBN 0 8155 0879 4, £23.50.
Vol. 3. Biochemical Aspects of Copper. Pp. xvii+205. ISBN 0 8155 0891 3.
Vol. 4. Physiological Aspects of Copper. Pp. xvi+286. ISBN 0 8155 0904 9.
Vol. 5. Biological Aspects of Copper. Pp. xvi+156. ISBN 0 8155 0918 9.
New Jersey: Noyes Publications, 1981–82.

These five volumes by the same author cover a large part of the literature on copper in biology and medicine up to 1980, with the emphasis on animals and man. There are over 10000 references, all fully documented, with the titles of the papers and even the home towns of the authors. This multi-volume monograph is therefore a mine of information and it is particularly useful in the areas in which the author has had long experience, namely the distribution and metabolism of copper in animal tissues.

Volume 1 starts with a short preface and a note about the author who is a professor of medicine in Minnesota, then it plunges straight into acquired and inherited copper deficiencies in animals and man. This is followed by chapters on copper toxicity and therapeutic applications. Bacteria and plants are also mentioned but only ten pages out of 178 are devoted to these topics. Volume 2 deals exclusively with Wilson's disease in all its aspects. Much remains to be learnt about the aetiology of this rare hereditary condition and these problems are discussed in Chapter 2. Volume 3 contains a detailed survey of the copper-containing proteins, of which some thirty are now known. Probably the best known of these is the blue plasma protein, caeruloplasmin, which is accorded a chapter to itself. The title of Volume 4 is rather misleading since sixteen of its seventeen chapters are devoted to a survey of the copper concentrations in the body fluids and tissues and only one chapter is devoted to physiology proper, namely absorption, excretion and balance. The last volume opens with a short chapter on the occurrence of copper in water, soil, plants and food. This is followed by a chapter on analytical methods which, surprisingly, does not include the use of the radioisotopes of copper, a technique in which the author has extensive experience. Other important topics, including quality control, reference materials and the preparation of biological samples are also not mentioned. The last chapter in this volume is devoted to the interrelationships between copper and other elements, particularly cadmium, iron and zinc.

These volumes, therefore, will be useful to anyone interested in the biochemistry, physiology or pathology of copper in animals or man but they will probably be less helpful to those interested in plant physiology, nutrition or food technology.

It is perhaps inevitable that a work of this magnitude by a single author will contain other defects besides that of coverage, and one of these is the arrangement of the material. An introductory chapter stating the author's aims and the plan of the whole work is clearly needed in an enterprise of this size. Historical aspects deserve more space than they receive in the present brief preface. A discussion of some of the chemical properties of copper would also have been appropriate, particularly the role of copper in redox reactions and the ease with which it forms complexes with organic molecules, properties which determined the relatively late entry of copper in biological evolution. Regarding the arrangement of the work as a whole, it would have been better to start with the topics covered in Volume 5 and to end with those covered in Volume 1, rather than the reverse.

Another defect is the complete lack of illustrations in any of the volumes so that the text reads more like a catalogue than a review. Also, the reader receives little guidance as to the more important papers among the many that are cited. More summaries and critical conclusions would also have been welcome. Abbreviations are not always defined. For example the statement on p. 233 in Volume 4 that 'there is an ESR peak in the dried blood' could be misinterpreted by a medically oriented reader. It is a surprise also to find that

none of the volumes is numbered, although the author refers to 'vol IV' on p. 29 in Volume 5 and to 'vol V' on p. 105 in Volume 4.

There is a good index at the end of each volume but no general index; this is unfortunate since some topics are dealt with in more than one volume. The author's style is generally concise, factual and correct but there are occasional lapses. It is not enough, for example, to state that 'a pig's tongue contains 2.1 μg Cu/g wet weight' or 'copper in the rat's tongue is 1.3 μg/g wet weight (Vol. 4, p. 4); ranges should be given, or were these single measurements?

Despite these strictures, this is an important work of reference for all who are interested in the trace elements. There are few typographical errors and the work should be on the shelves of most university and technical libraries. However, the cost of the complete series is well over £100, which will deter most private purchasers.

A. Hodgkinson

Principles and Practice of Disinfection, Preservation and Sterilization. Ed. by A. D. Russell, W. B. Hugo and G. A. J. Ayliffe. Oxford: Blackwell Scientific Publications, 1982. Pp. x+653. ISBN 0 632 00547 5. £32.00.

Drs Russell and Hugo and Professor Ayliffe, with twenty-eight other contributors, have produced a useful book dealing with a very practical aspect of microbiology that has application in many fields.

There are three parts. Part 1, Disinfection, deals with: history; types of disinfectants; factors affecting efficacy; evaluation; modes of action; types of resistance; good manufacturing practice; application in hospitals. Part 2, Preservation, deals with: preservatives and their mode of action; preservation of pharmaceuticals and cosmetics, cutting oil emulsions, fuels and lubricants, paper and pulp, textile and leather, paint, building materials and wood. Part 3, Sterilization, deals with: sterilization by heat and applications in medicine and industry; ionizing and ultraviolet irradiation; sterilization by gases and filtration; control and monitoring of sterilization, processes.

The book is generally well presented with clear, large (almost too large) typeface, with clear diagrams and with very few typographical errors (e.g. equation 3.6, p. 120). There are few photographs and some of them are of a lower standard than the book deserves. The size and presumably cost of the book could with advantage have been reduced by having one list of references, rather than separate often repetitive lists at each chapter or part of chapter, and by reducing the space between subsections of the text (e.g. p. 184, 185, 247). The index is comprehensive. For a book of this sort, likely to be used frequently for reference in the laboratory, the binding might have been given a plastic coating.

Reading this book revealed or emphasized to me many interesting points. The difficulty of disinfectant evaluation leads to different countries and different industries using different tests and standards, a situation causing

difficulties for both the producer and the user. The apparent absence of statistical considerations is surprising but perhaps the complication is not at this stage thought appropriate in view of the low level of sensitivity obtained. Practical tests to simulate real-life situations for evaluation of disinfectants are said to have appeared recently—I had though that the Hoy & Clegg (1953) can test was an example of such a test. Chapter 8 on problems of disinfection in hospitals is of considerable interest to the food industry in indicating what errors in design and use can increase contamination. Apparently the main cause of disinfectant failure is inaccurate dilution by untrained staff. Also it seems that the uninitiated will spend too much effort on disinfecting floors, walls and ceilings which are rarely heavily contaminated while neglecting the insides of tubing and medical equipment, where contamination can rapidly increase and do maximum damage. The importance of preservation of items from microbial attack is emphasized in excellent sections on food, oils, wood, etc.

The Editors have brought together a wide range of interesting topics and have allowed individual styles to enrich the book. However, I would have appreciated more editorial control over certain aspects. The title of the book includes 'Principles' and so I expected to find chapters or sections clearly delimited to deal with those principles of wide relevance to the subjects of the book. For example, I had in mind consideration of exponential death and its implications, particularly *probability* of sterility. (In this context I was disappointed to read, in the preface, that sterilization is an 'infinitely more secure process' than disinfection and, on p. 115, the unsupported statement that 'it is obviously easier for an antimicrobial agent to be effective when there are few microorganisms against which it has to act'.) I also expected a unified treatment of the properties of the bacterial endospore in the context of killing by various methods. Certainly these subjects were dealt with in the book—but in various places, in specific contexts. As 'preservation' constituted a quarter of the book, I expected, but could not find, a definition of 'preservation' or guidance on the principles of the subject. In fact there seems to be disagreement about whether a preservative should kill microbes or merely stop them growing.

There are some minor aspects on which I differ with editorial judgement. I give some examples taken at random. I found it disappointing in the chapter on good manufacturing practice that details were given on packing of ovens but not on heating time; that autoclave temperature but not time was given, nor aspects of time and volume considerations; that *Pseudomonas aeruginosa* was considered not to be pathogenic; that there was minimal consideration of pyrogens; and that there was no mention of the standard U.K. manual on the bacteriological examination of water supplies.

I did not like the use of 'G−ves', 'G+ves' and 'slimicide' (can you kill a slime?) in some parts of the book, nor different units used for solubilities, even on the same page, e.g. p. 27. On page 207 and elsewhere the term bacterial 'spore' is used as synonymous with 'endospore'. While I acknowledge that endospores are the most important types of bacterial spores from

the point of view of resistance to heat and certain chemicals, I think that the occurrence of other types of spores should have been acknowledged. I cannot see how a virustatic agent could be assessed (Table 4.1), and was not helped by the statement on p. 154 that virucidal tests are still at the experimental stage.

Authors and editors seemed to me to have in mind readers with an extremely uneven knowledge of microbiology, expected not to know what ribosomes do (p. 170), but to know what plasmids are (p. 199), and to understand a mathematical explanation of a crucially important concept, the chemiosmotic theory. In this last context I imagine that a reference to an introductory treatment of the subject (e.g. in Lynch, J.M. & Poole, N.J. (1979) *Microbial Ecology A Conceptual Approach,* Blackwell Scientific Publications, Oxford), would have been appreciated. Alternatively a simple statement might have been made such as: Healthy bacteria maintain a lower concentration of hydrogen ions inside the cell than outside. Hydrogen ions 'pressing' to get back inside can be used to generate ATP, to cause flagella to rotate, and to allow transport of other substances against a concentration gradient.

The book will be an extremely useful reference work for many people in higher education, industry and medicine. I am confident its popularity will lead to a second edition, when the opportunity may be taken to increase its utility by attention to the minor points I have mentioned.

R. W. A. Park

JOURNAL OF FOOD TECHNOLOGY: NOTICE TO CONTRIBUTORS

The Journal of Food Technology publishes original contributions to knowledge of food science and technology and also review articles in the same field. Papers are accepted on the understanding that they have not been and will not be, published elsewhere in whole, or in part, without the Editor's permission. Papers accepted become the copyright of the Journal. This journal is covered by *Current Contents*.

Typescripts (two complete copies) should be sent to the Editor, Dr H. Liebmann, c/o Institute of Food Science and Technology (U.K.), 20 Queensberry Place, London SW7 2DR. Papers should be typewritten on one side of the paper only, with a $1\frac{1}{2}$ inch margin, and the lines should be doubled-spaced. In addition to the title of the paper there should be a 'running title' (for page headings) of not more than 45 letters (including spaces). The paper should bear the name of the author(s) and of the laboratory or research institute where the work has been carried out. The full postal address of the principal author should be given as a footnote. (The proofs will be sent to this author and address unless otherwise indicated.) The Editor reserves the right to make literary corrections.

Arrangement. Papers should normally be divided into: (a) Summary, brief, self-contained and embodying the main conclusions; (b) Introduction; (c) Materials and methods; (d) Results, as concise as possible (both tables and figures illustrating the same data will rarely be permitted); (e) Discussion and conclusions; (f) Acknowledgments; (g) References.

References. Only papers closely related to the author's work should be included; exhaustive lists should be avoided. References should be made by giving the author's surname, with the year of publication in parentheses. When reference is made to a work by three authors all names should be given when cited for the first time, and thereafter only the first name, adding *et al.*, e.g. Smith *et al.* (1958). The '*et al.*' form should always be used for works by four or more authors. If several papers by the same author and from the same year are cited, a, b, c, etc. should be put after the year of publication, e.g. Smith *et al.* (1958a). All references should be brought together at the end of the paper in alphabetical order. References to articles and papers should mention (a) name(s) of the author(s) (b) year of publication in parentheses; (c) title of journal, underlined, abbreviated according to the *World List of Scientific Publications*, 4th edn and supplements; (d) volume numbers; nnmber of first page of article. References to books and monographs should include (a) name(s) and initials of author(s) or editor(s); year of publication in parentheses; (b) title, underlined; (c) edition; (d) page referred to; (e) publisher; (f) place.

Standard usage. The *Concise Oxford English Dictionary* is used as a reference for all spelling and hyphenation. Statistics and measurements should always be given in figures, i.e. 10 min, 20 hr, 5 ml, except where the number begins the sentence. When the number does *not* refer to a unit of measurement, it is spelt out except where the number is one hundred or greater.

Abbreviations. Abbreviations for some commoner units are given below. The abbreviation for the plural of a unit is the same as that for the singular. Wherever possible the metric SI units should be used unless they conflict with generally accepted current practice. Conversion factors to SI units are shown where appropriate.

SI UNITS

gram	g	Joule	J
kilogram	kg = 10^3 g	Newton	N
milligram	mg = 10^{-3} g	Watt	W
metre	m	Centigrade	°C
millimetre	mm = 10^{-3} m	hour	hr
micrometre	μ = 10^{-6} m	minute	min
nanometre	nm = 10^{-9} m	second	sec
litre	l = 10^{-3} m^3		

NON SI UNITS

inch	in	= 25·4 mm
foot	ft	= 0·3048 m
square inch	in^2	= 645·16 mm^2
square foot	ft^2	= 0·092903 m^2
cubic inch	in^3	= 1·63871 × 10^4 mm^3
cubic foot	ft^3	= 0·028317 m^3
gallon	gal	= 4·54611
pound	lb	= 0·453592 kg
pound/cubic inch	lb in^{-3}	= 2·76799 × 10^4 kg m^{-3}
dyne		= 10^{-5} N
calorie (15°C)	cal	= 4·1855 J
British Thermal Unit	BTU	= 1055·06 J
Horsepower	HP	= 745·700 W
Fahrenheit	°F	= 9/5 T°C + 32

Figures. In the text these should be given Arabic numbers, e.g. Fig 3. They should be marked on the backs with the name(s) of the author(s) and the title of the paper. Where there is any possible doubt as to the orientation of a figure the top should be marked with an arrow. Each figure must bear a reference number corresponding to a similar number in the text. Photographs and photomicrographs should be unmounted glossy prints and should not be retouched. Line diagrams should be on separate sheets; they should be drawn with black Indian ink on white paper and should be about four times the area of the final reproduction. Lines and lettering should be of sufficient thickness and size to stand reduction to one-half or one-third. Whenever possible, the originals of line diagrams, prepared as described above, should be submitted and not photographs, The legends of all the figures should be typed together on a single sheet of paper headed 'Legends to figures'.

Tables. There should be as few tables as possible and these should include only essential data; the data should not be crowded together. The main heading should be in bold with an Arabic number, e.g. **Table 2.** Each table must have a caption in small letters. Vertical lines should not be used.

Offprints. Fifty offprints will be issued free with each paper but additional copies may be purchased if ordered on the printed card which will be sent to the senior author with the proofs.

Journal of Food Technology
Volume 18 Number 5 October 1983

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Printed by Adlard and Son Ltd, Bartholomew Press, Dorking