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Detection and partial characterization of soluble pig muscle proteins by immunoelectrophoresis in agarose gels

C. CASAS*, J. TORMO, P. E. HERNANDEZ AND B. SANZ

Summary

Immunoelectrophoresis in agarose gels has been used to detect and partially characterize specific protein precipitin bands of soluble proteins of pig muscle (PSP), free of cross-reactions with soluble muscle proteins of cow (CSP), horse (HSP) and chicken (CHSP). Out of six precipitin bands obtained by reacting PSPs against an anti-PSP antiserum produced by a rabbit, four bands were observed to appear by reacting HSPs against the same anti-PSP antiserum. Two more bands were detected by analysing CSPs against an anti-PSP antiserum and three bands were detected by analysing CHSPs against the same anti-PSP antiserum. Thus, only one band is specific to the soluble protein of pig muscle. This technique may have the potential to detect the presence of pork in unheated ground meat products.

Introduction

A typical adulteration of unheated ground meat products consists in the addition of vegetable (Parsons & Lawrie, 1972) or animal proteins (Deschreider & Meaux, 1974), not declared as such on the ingredients' list of these products. A simple, rapid test would make it possible to determine the addition of undeclared meat in a product and give the consumer greater protection.

Immunologically, it is possible to differentiate proteins from different animal species using antisera to muscle proteins or to blood serum (Warnecke & Saffle, 1968). In an effort to detect specific protein bands of soluble proteins of pig muscle, free of cross-reactions with soluble muscle proteins of cow, horse and chicken, the technique of immunoelectrophoresis in agarose gels has been used. These specific protein bands may indicate the presence of pig meat in fresh meat products. As far as we know, this is the first time that immunoelectrophoresis with anti (muscle soluble proteins) has been employed in trying to pursue the objective previously stated.

Materials and methods

Preparation of the antigenic extracts

Skeletal muscle tissue from pig (*Ms. intercostalis externi* and *Ms. trapezius*), horse (*Ms. glutaeus superficialis* and *Ms. biceps femoris*), cow (*Ms. rectus femoris*, *Ms. vastus lateralis*) and chicken (*Ms. pectoralis* and *Ms. supracoracoideus*) (total weight of 250 g)

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were finely triturated, minced and homogenized in 500 ml of a 0.85% saline solution. The soluble proteins were extracted by constant agitation of these homogenates for 1 hr at 1°C. The protein extracts were filtrated through a Whatman No. 1 filter paper, lyophilized and the dried extracts placed in an airtight container and stored at −20°C until used.

Preparation of the antiserum

Serum containing suitable PSP antibodies was obtained by injecting subcutaneously New Zealand male rabbits with single doses of lyophilized pig protein extracts (48 mg) in 2 ml of deionized and distilled water emulsified in 0.5 ml of Freund complete adjuvant (Difco). Fifteen booster doses were applied subcutaneously every 4 days for 62 days. The rabbits were periodically bled from the marginal ear vein and the blood was allowed to clot at room temperature and then cooled at 4°C for 18 hr. The antiserum was decanted and centrifuged at 1000 *g* for 10 min to remove any remaining blood cells present.

Agarose gel immunoelectrophoresis

The basic technique was that of Grabar & Williams (1953), modified by Scheidegger (1955). Consequently, only the details relevant to this application are given here. 1% agarose gel in veronal buffer, pH 8.6 was used. The gels were punched with a well cutter giving a hole of 1 mm diameter and a band of 0.2×5 cm. One μl (24 μg) of the antigenic extracts was deposited in the hole. The plate was then subjected to electrophoresis for 4 hr at 130 V. When the electrophoresis was concluded, the plate band was filled with 0.3 ml of the corresponding antiserum. Immunodiffusion was performed for 18–24 hr at 37°C. The protein precipitating bands were made visible with Amido black (Clausen, 1981). The immunochemical partial characterization of these bands comprised the detection of glycoprotein fractions according to NADI's method (Clausen, 1981), and lipoprotein fractions with Sudan black (Uriel, Avrameas & Grabar, 1963).

The degree of displacement of each band relative to bovine serum albumin (Fraction V Cohen, Sigma Chemical Co.,) used as standard was defined as follows:

$$\text{absolute mobility} = \frac{\text{band displacement (mm)}}{\text{V/mm/sec}};$$

$$\text{relative mobility} = \frac{\text{band displacement (mm)}}{\text{bovine serum albumin displacement (mm)}};$$

$$\% \text{ mobility} = \text{relative mobility} \times 100.$$

Results and discussion

To detect specific protein bands of soluble pig proteins (PSP), free of cross-reactions with muscle soluble proteins of horse (HSP), cow (CSP) and chicken (CHSP), lyophilized soluble protein extracts of each animal species were analyzed by immunoelectrophoresis in agarose gels against an anti-PSP antiserum produced by a rabbit.

The lyophilized pig muscle soluble protein extracts (PSP) analysed against an homologous antiserum (anti-PSP), allowed the identification of six protein precipitin bands of percent mobilities between 16±0.04 and 75±1.70 (Table 1 and Fig. 1). Three of the six observed bands were arbitrarily defined as major bands (bands 1, 4 and 6) by their strong staining with Amido black. The three remaining bands (bands 2, 3 and 5)

Table 1. Absolute, relative and percent mobilities of protein precipitin bands of soluble pig muscle proteins (PSP) against a rabbit homologous antiserum (anti-PSP)*

Precipitin band (No.)	Band displacement (mm)	Absolute mobility	Relative mobility	Percent mobility
1	6.0	0.69±0.06	0.16	16±0.04
2	6.5	0.75±0.06	0.17	17±1.03
3	12.5	1.44±0.22	0.33	33±1.32
4	14.0	1.62±0.17	0.37	37±1.09
5	18.0	2.08±0.20	0.48	48±0.66
6	28.0	3.24±0.38	0.75	75±1.70
B. albumin	37.0	4.28±0.34	1.0	100
Dextrane	0.0	0.0	0.0	0.0

*The degree of displacement of each band relative to bovine serum albumin used as standard, was defined as stated in Materials and methods.

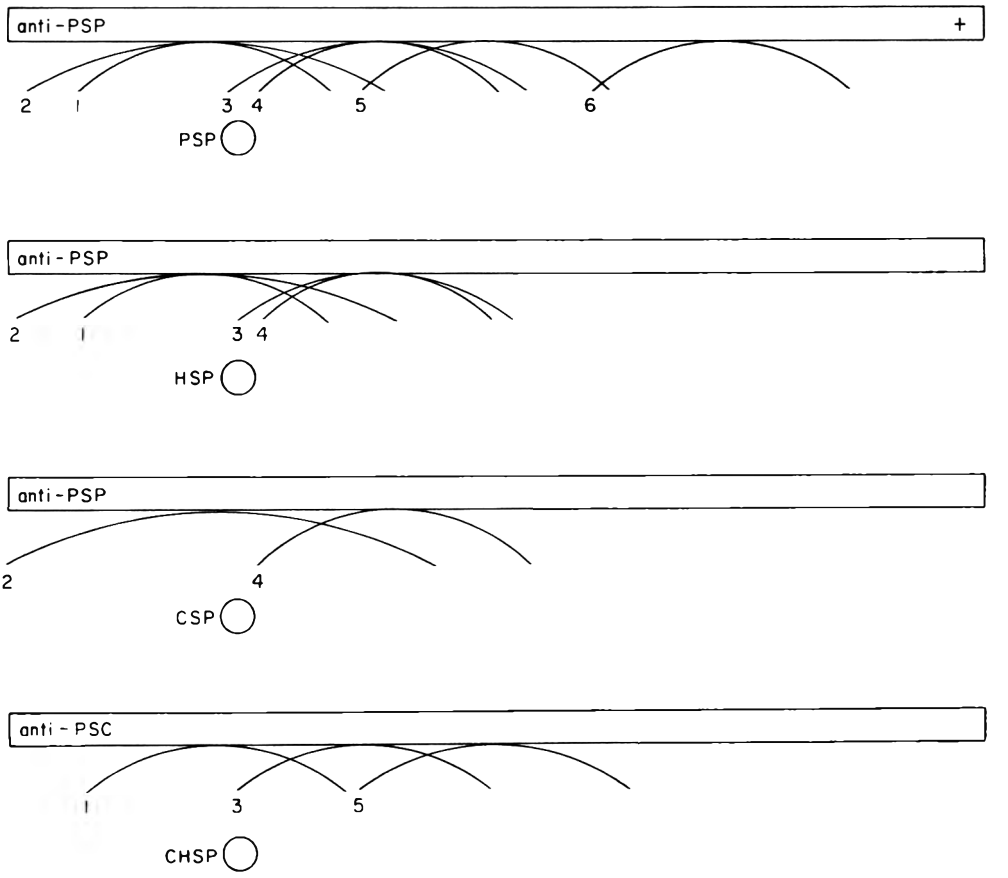

Figure 1. Patterns of protein precipitin bands of different animal soluble muscle proteins against a soluble pig muscle protein antiserum (anti-PSP). PSP, pig proteins; HSP, horse proteins; CSP, cow proteins; CHSP, chicken proteins.

Table 2. Detection and immunochemical characterization of protein precipitin bands of pig soluble muscle proteins (PSP) against a rabbit homologous antiserum (anti-PSP)

Immunization period		Precipitin bands (band No.)		Immunochemical characterization (band No.)	
Bleeding	Days	Major	Minor	Glycoproteins	Lipoproteins
B1	12	1	—	—	—
B2	28	1, 4, 6	—	—	—
B3	48	1, 4, 6	2, 3, 5	5	—
B4	62	1, 4, 6	2, 3, 5	5	—

were defined as minor bands by their weak staining with the same dye. The partial immunochemical characterization (Table 2) of these bands to detect the presence of glycoprotein and lipoprotein fractions in their structure, resulted in only one glycoprotein band (band 5).

When the lyophilized soluble muscle protein extracts of horse (HSP) were analysed against an anti-PSP antiserum, the presence of four protein precipitin bands of percent mobilities 16 ± 0.10, 17 ± 0.34, 33 ± 1.43 and 37 ± 0.99 were observed (Fig. 1). During the analysis of chicken protein extracts (CHSP) against the same anti-PSP antiserum, three protein bands of percent mobilities 16 ± 0.81, 33 ± 0.74 and 48 ± 1.02 were observed. During the analysis of cow protein extracts (CSP) against an anti-PSP antiserum, two protein bands of percent mobilities 17 ± 1.42 and 37 ± 0.87 were detected. Since all these bands have mobilities close to bands 1, 2, 3, 4 and 5 of the soluble pig proteins against an anti-PSP antiserum, they may be responsible for false positive reactions when trying to quantitatively analyse different soluble animal protein extracts by immunodiffusion (Gabucci & Flego, 1975). It is also important to emphasize that the major band No. 6 of the soluble pig muscle proteins against an anti-PSP antiserum, is a specific pig protein precipitin band which do not appear when the soluble muscle protein extracts of horse, cow and chicken are analysed against the same anti-PSP antiserum. This specific pig protein band may indicate the presence of pork in meat products, when soluble meat protein extracts are analysed against an anti-PSP antiserum. Theoretically, this protein should be present in any kind of soluble pig muscle protein homogenate, whatever the composition of the mixture may be.

Immunoelectrophoresis in agarose gels to differentiate proteins from different animal species has been used with antisera against serum protein as reference patterns (Karpas, Myers & Segre, 1970). We believe that the use of soluble muscle protein antisera instead of antisera against serum protein, may improve this assay, due to the fact that it is more rigorous to analyse soluble muscle proteins against anti soluble muscle proteins, than soluble muscle proteins against anti blood serum proteins. The real usefulness of this technique when applied to a mixture of meat components, as well as its possible interference with other components that could be present in the meat mixture (such as egg, milk, vegetable proteins), has not been verified yet. This area of work merits special attention and further work will be carried out.

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Functional aspects of blood plasma proteins. II. Gelling properties

NAZLIN K. HOWELL*† AND R. A. LAWRIE‡

Summary

A method for studying the gelation of small quantities of plasma proteins in a cake-type model system was devised and was found to produce reproducible gels. The results indicated that considerable increases in the strength of the gels of porcine and bovine plasma and porcine serum developed as time and temperature of heating were raised. These increases were much greater than those obtained for comparable egg albumen gels. Of the plasma proteins fractionated by ion exchange chromatography, fraction III (albumin) followed a similar pattern to that of whole plasma. However, the gels of all three fractions were weaker than those of whole plasma, indicating interaction between the different proteins in the latter.

Introduction

There is considerable potential for the utilization of blood plasma proteins in meat, bakery and other food products in which their inclusion would alter such properties as gelation, viscosity and solubility. In particular, the partial replacement of egg albumen or egg white in products such as cakes would be economically attractive since, at present, the cost of dried egg albumen is about £5000/ton whereas that of dried plasma is about £2400/ton. In order to utilize plasma in cakes it is clearly important to assess its suitability and compatibility with other cake ingredients.

One of the key functions of egg albumen in cakes is the formation of the cake structure by gelation. At high temperatures, in the final stages of baking, the egg albumen proteins bind ingredients such as flour proteins, starch, sugar and water, forming a matrix which supports the air cells (Howell, 1978). Earlier work indicated that whole plasma could be used in partial replacement for egg proteins in bakery products, such as cakes, bread and biscuits (Johnson *et al.*, 1979). However, the nature of the functional properties of whole plasma, and of its component proteins, in cakes has not been reported hitherto. One of the most effective ways of studying functional properties is by the use of model systems. A cake-type model system was adopted in the present investigation to study the gelation of whole plasma, serum, and the three plasma fractions (I, II and III) obtained by ion exchange chromatography (Howell & Lawrie, 1983) and egg albumen. A high ratio sponge cake normally contains approximately 6% (w/w) egg albumen proteins and 45% (w/w) sucrose, as well as ingredients such as flour, fat, emulsifiers and small quantities of other proteins. The

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model system adopted thus consisted of 6% (w/w) protein in 45% (w/w) sucrose solution in distilled water, adjusted to pH 8.0.

Due to the complex nature of protein gelation (Lapanje, 1979) a range of time/temperature heat treatments was selected to cover those experienced during the baking process. It was necessary to devise a valid quantitative method to test the gelling properties of small quantities of proteins since standard methods for studying functional properties, including gelation, are lacking. Furthermore the terminology for measuring gel texture parameters is not well defined. This has led to a variety of approaches being used by different workers, involving many compression cycles and peak area measurements, as reported by Szczesniak (1975) and Bourne (1978). In the present study, gel strength and breaking strength were measured on the Instron Universal Testing Machine.

Methods

Materials

The following proteinaceous powders were used (protein contents are given in parentheses): freeze-dried porcine plasma (70%); freeze-dried porcine serum (84%); ion exchange porcine plasma fractions I (78%), II (70%), III (76%); commercial dried porcine Regalbumin (70%) and commercial dried bovine plasma Regalbumin (70%); and commercial spray-dried egg albumen (80%).

General procedure

The dry protein powder (6% w/w) was mixed with sucrose AR (45% w/w) in a 100 ml beaker. Distilled water (49% w/w) was added gradually and the sample was mixed until the powder had dissolved (about 1 h). The pH was adjusted to 8.0.

Gels were made in stainless steel tubes 50 mm long and 16 mm internal diameter. One end of the tube was stoppered with a rubber bung (size 17) and secured tightly with waterproof insulating tape. The gel solution (7 ml) was poured into the tube and covered with a short rubber bung taped loosely in place. For each sample eight to ten gels were prepared. The tubes were placed in a test tube rack and heated in a water bath at a specific temperature (±0.5°C) for a specific time (±30 sec). It was noted that accurate control of water bath temperatures and heating time was necessary for reproducible gels. The rate of coming up to temperature was not studied. The water level was maintained at 5 mm below the top edge of the tube. A thermocouple passed through a hole in the rubber bung and centred in one of the tubes was used to monitor the temperature inside the gel.

After heating, the tubes were cooled in a water bath for 10 min to 20°C and left overnight (16 h) to age. The gels were easily removed from the tubes by gentle tapping or syringing a small quantity of distilled water between the gel and the tube wall. Excess water was wiped with a tissue. Each gel was cut to a length of 15 mm with a scalpel and subjected to a compression test on the Instron Universal Testing Machine.

Gel testing on the Instron

The Instron Universal Testing Machine was equipped with a compression load cell having a maximum load capacity of either 2000 g or 20000 g. A cylindrical brass plunger, to which a disc with a diameter of 43 mm was attached, was screwed on to the crosshead. The crosshead was driven by a gear at 10 mm/min. Calibration of the chart

was carried out by means of weights (0–100 g on sensitivity scale ×1, for the light cell; 0–2000 g on scale ×2 for the heavy cell). The chart speed was set at 100 mm/min.

The gel, with the cut end facing upwards, was placed on the centre of the table. Following the manual adjustment of the plunger to just touch the surface, the plunger automatically pressed the gel to a distance of 12 mm at 1 cm/min. The load was removed at a fast speed. The force required to press the gel was recorded against time on the chart recorder and the following parameters were calculated:

(i) rigidity, brittleness or gel strength (GS), i.e. the load (g) required to depress the gel surface a specified distance (6 mm in these experiments); and
(ii) breaking strength (BS); i.e. the load (g) required to rupture the gel surface.

Effect of time and temperature of heating on plasma and egg albumen proteins

Solutions of the proteins listed were made up as described. Heat treatment was performed at each of the temperatures 80, 85, 90 and 95°C for 15, 30 or 60 min for egg albumen commercial porcine plasma, and commercial bovine plasma; at 85, 90 and 95°C for 15, 30 and 60 min for porcine serum, and plasma fractions I and III; at 85 and 95°C for 15, 30 and 60 min for plasma fraction II; and 95°C for 15, 30 and 60 min for porcine plasma. After gelation the gels were compressed and characterized on the Instron.

Results

The effects of time and temperature of heating on the gel strength (GS) and breaking strength (BS) of gels prepared from proteins of commercial porcine or bovine plasma, freeze-dried porcine plasma, porcine serum and plasma fractions I, II and III are illustrated in Figs 1–6.

The GS and BS of both commercial porcine (Fig. 1) and bovine (Fig. 2) plasma increased with both temperature and time of heating. The BS of porcine plasma was similar to that of bovine when heated at 95°C ($P > 0.1$) but lower at 80°C ($P < 0.005$). For both porcine and bovine plasma increases in BS were related to the time of heating

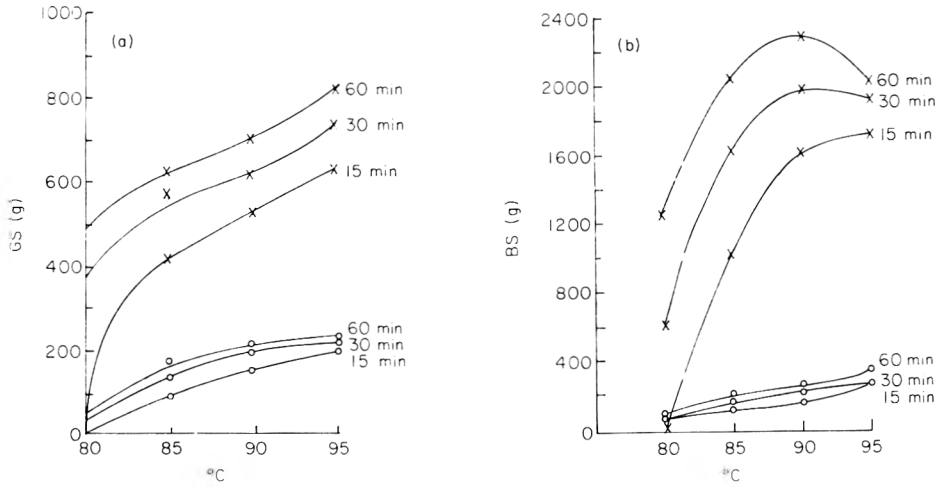

Figure 1. Variation of (a) gel strength (GS) and (b) breaking strength (BS) of commercial porcine plasma (x—x) and egg albumen (o—o) proteins with temperature and time of heating (min).

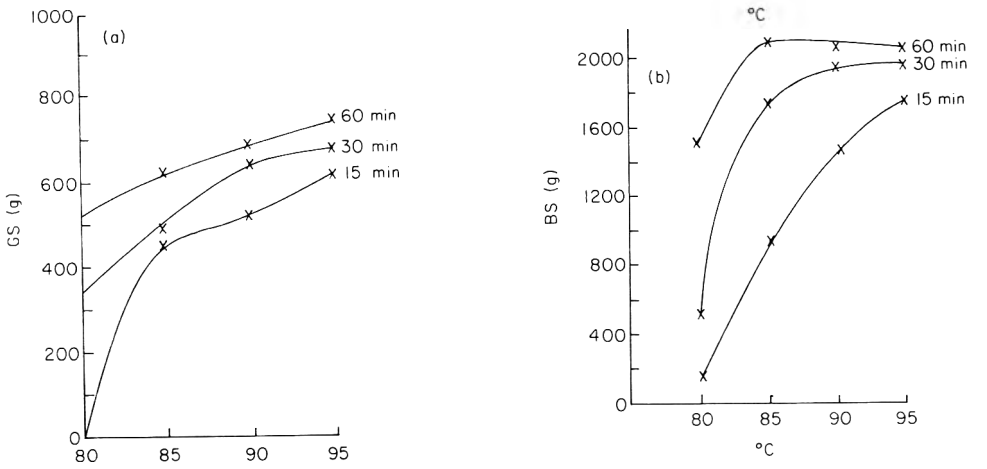

Figure 2. Variation of (a) gel strength (GS) and (b) breaking strength (BS) of commercial bovine plasma with temperature and time of heating (min).

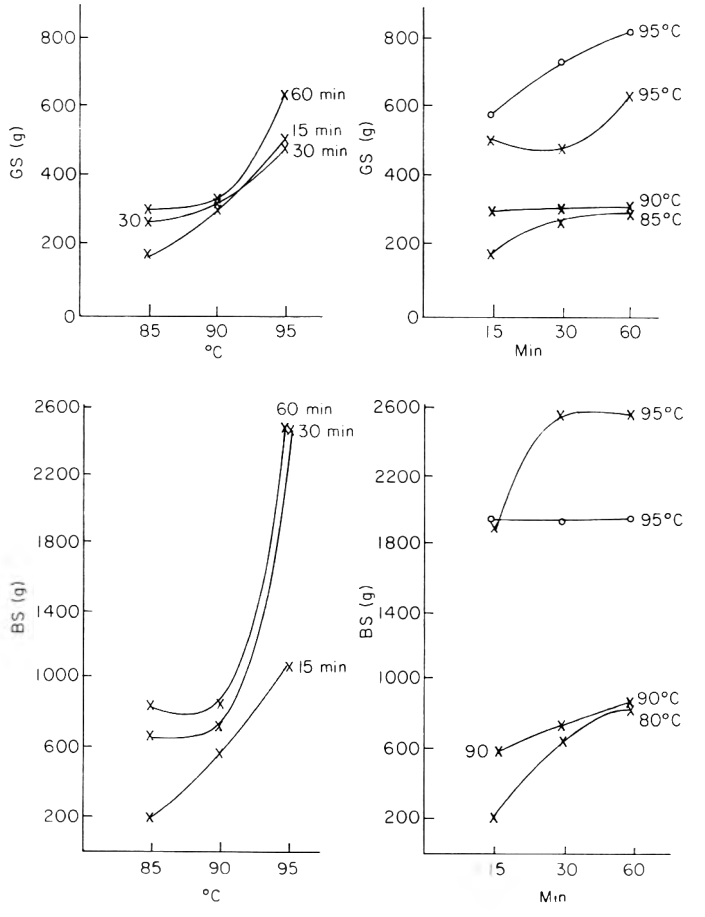

Figure 3. Variation of (a) gel strength (GS) and (b) breaking strength (BS) of porcine serum (x—x) and freeze-dried (FD) porcine plasma (o—o) gels with temperature (°C) and time of heating (min).

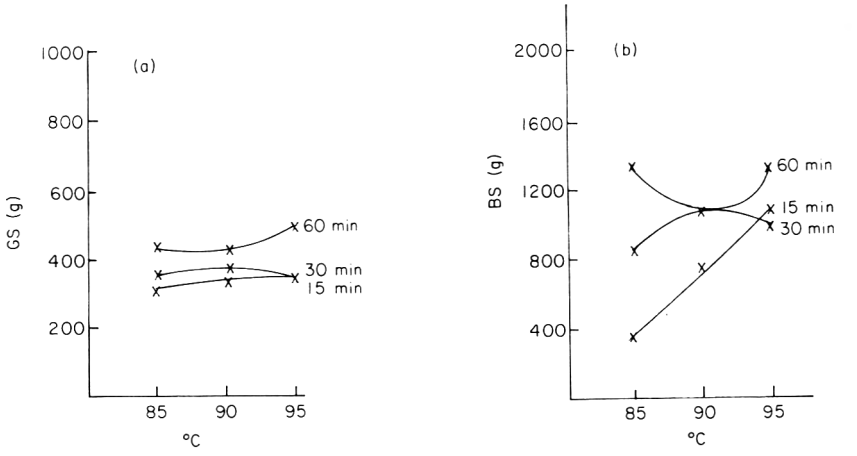

Figure 4. Variation of (a) gel strength (GS) and (b) breaking strength (BS) of porcine plasma fraction I gels with temperature (°C) and time of heating (min).

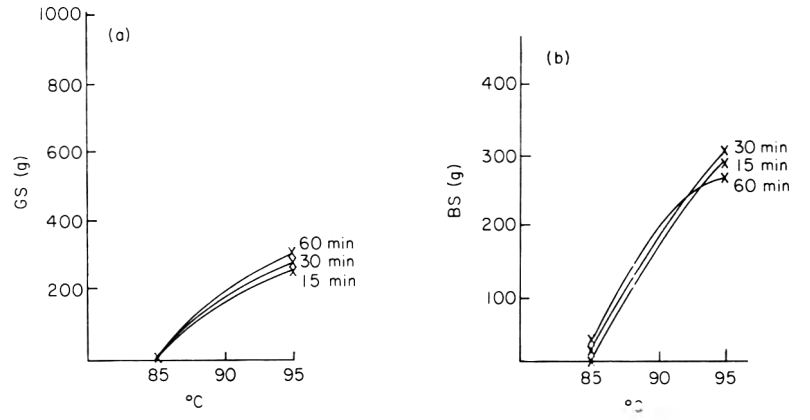

Figure 5. Variation of (a) gel strength (GS) (g) and (b) breaking strength (BS) (g) of porcine plasma fraction II gels with temperature (°C) and time of heating (min).

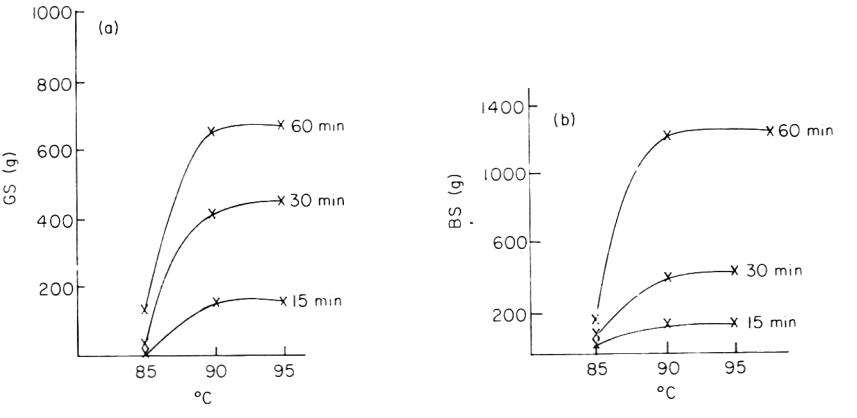

Figure 6. Variation of (a) gel strength (GS) (g) and (b) breaking strength (BS) porcine plasma fraction III gels with temperature (°C) and time of heating (min).

at 80 and 85°C. At 90 and 95°C however, there was little difference in the BS for different times of heating (Figs 1 and 2).

The GS and BS of freeze-dried porcine plasma gels were measured at 95°C only, as values were found to be similar to those of commercial porcine plasma (Fig. 3). Increases in time and temperature increased both the GS and BS values for freeze-dried porcine plasma gels. As in the case of porcine plasma, the GS of porcine serum protein gels also increased with time and temperature of heating but the values were lower than those of porcine plasma (Fig. 3). Also the BS was lower than that of porcine plasma gels at 85 and 90°C but higher at 95°C ($P < 0.005$) (Fig. 3). The gel strength of porcine plasma fraction I gels exhibited a low response to time and temperature of heating after reaching a fairly high level at 85°C and 15 min (Fig. 4). In contrast, although the BS increased with temperature at 15 min and with time at 85°C it varied less at 90 and 95°C for heating times of 30 and 60 min.

Plasma fraction II gels had lower GS and BS values than those of plasma fraction I ($P > 0.005$). Both the GS and BS values (Fig. 5) increased with temperature increase; but did not vary much with time.

Plasma fraction III gels showed a marked increase in GS and BS values with both time and temperature as shown in Fig. 6. Although both GS and BS values were lower than those of fraction I at 85°C the values were much higher at 90 and 95°C at 30 and 60 min (Fig. 6) ($P > 0.005$).

Discussion

Using this model system, satisfactory and reproducible gels were obtained within stainless steel tubes. It was noted that accurate water bath temperatures for gel formation were critically important for gel reproducibility. In addition, the measurements of gel strength and breaking strength on the Instron were found to be reproducible, practical and convenient parameters. These parameters had a coefficient of variation of less than 10% for most samples tested using eight replicates.

Both porcine and bovine plasma proteins increased considerably in gel strength and breaking strength with both time and temperature of heating. Freeze-dried plasma behaved in a similar manner to the commercially dried plasma proteins—the separation of plasma and drying procedures evidently had not affected the gelling properties. However, it was noted that the presence of small amounts of red cell fraction in plasma lowered the gel strength and breaking strength.

The contribution of fibrinogen to the gelling properties of whole porcine plasma was indicated by the higher gel strength and breaking strength values of whole plasma compared with serum. Fibrinogen was also present in plasma fraction I which formed gels; but these were less strong than those involving whole plasma or serum. Unlike whole plasma gels, longer heating times and higher temperature did not increase the gel strength or breaking strength of fraction I gels substantially.

In contrast to fraction I, which formed heat irreversible gels at 79°C, fraction II gelled at a markedly higher temperature (90°C) and a shorter time of heating. Fraction III (albumin) formed irreversible gels at an intermediate temperature (85°C). The gelling behaviour of fraction III was similar to that of whole plasma in that the gel strength and breaking strength values increased considerably with both time and temperature of heating. This behaviour indicated that gelling of whole plasma and serum is largely governed by the properties of albumin (which constitutes 50% of the plasma proteins). However, the values for each of the three fractions of plasma were lower than those of

whole plasma. Clearly the proteins of fractions I, II and III contribute to the strength of the plasma gels partly by interaction with each other.

Considerable differences in the gelation of whole porcine or bovine plasma and egg albumen were noted. Unlike whole plasma, with which an increase in the temperature and time of heating produced large increases in gel strength and breaking strength, heating led to only small increases in these parameters with egg albumen.

It would thus appear that whole plasma, serum or plasma fractions might be used to partially replace the gelling function of egg albumen in cakes. Furthermore, the strong gelling properties of plasma might be exploited in other food products such as meat and desserts. A study of the compatibility of plasma proteins with other ingredients on the gelling function of whole plasma as an egg replacer in cakes will be reported in subsequent papers.

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Functional aspects of blood plasma proteins. III. Interaction with other proteins and stabilizers

NAZLIN K. HOWELL*† AND R. A. LAWRIE‡

Summary

The proteins of whole blood plasma, serum and plasma fractions interacted with egg albumen proteins producing stronger gels than those of the plasma protein preparations in isolation. An index was devised to measure this interaction, which varied with time and temperature of heating and plasma protein concentration. Although whey proteins exhibited positive (i.e. increased gel strength) interaction with plasma proteins, the admixture of proteins of soya isolate or sodium caseinate, and the incorporation of selected stabilizers, resulted in very weak gels.

Introduction

The potential for using plasma proteins in foods such as bakery products, meat products and health drinks is high due to both economic and functional reasons. Howell & Lawrie (1984) pointed out that the gelling behaviour of the plasma proteins could be used to replace that contributed by egg albumen proteins in cakes. Indeed whole plasma, serum and albumin proteins produced strong gels, the strength of which increased with time and temperature of heating. In contrast, egg albumen proteins produced weak gels regardless of time and temperature of heating thus implying that a relatively small proportion of plasma proteins might replace egg albumen proteins to produce a similar gelling effect.

Because of the complexity of food systems, however, it is necessary to assess the compatibility and interactions of plasma proteins with egg albumen and other proteins and stabilizers likely to occur in foods. In the present paper results from a study of the interaction of the proteins of whole plasma, serum and three plasma fractions with egg albumen proteins, using the gelling method described previously (Howell & Lawrie, 1984), are reported.

Times and temperature were selected for the heat treatment to cover the range of those experienced during the baking process. At the outset synergistic interaction between plasma and egg albumen proteins was noted. This led to the calculation of an interaction index which could be used to study the interaction quantitatively.

The study was extended to include whey, sodium caseinate and soya isolate proteins. For this purpose one time–temperature combination (95°C and 20 min) was chosen to resemble the baking process.

Most food products also contain stabilizers which impart viscosity and water-

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binding properties to the product. Therefore the effect of stabilizers on the gelation of plasma proteins at both high (8.0) and low (4.0) pH values was examined.

Materials and methods

Materials

The following powders were used, the protein contents are given in parentheses: freeze-dried porcine blood plasma (70%); freeze-dried porcine blood serum (84%); porcine blood plasma fractions I (78%), II (70%) and III (76%); commercial porcine plasma Regalbumin (70%); commercial bovine plasma Regalbumin (70%); spray dried egg albumen (80%); soya isolate RP 500E (90%); sodium caseinate (90%); MMB whey protein Lactein 75 (75%). The blood plasma fractions I, II and III were prepared as described by Howell & Lawrie (1983).

Methods

Interaction of plasma proteins with egg albumen proteins. The interaction of the plasma proteins with spray dried egg albumen proteins was examined using the following combinations: 4% (w/w) plasma protein and 2% (w/w) egg albumen proteins; 3% (w/w) plasma protein and 3% (w/w) egg albumen proteins; 2% (w/w) plasma protein and 4% (w/w) egg albumen proteins. Sucrose (45% w/w) was added and the samples made up with distilled water (49% w/w). Heat treatment and gel tests were performed as described by Howell & Lawrie (1984).

Interaction indices I_{GS} and I_{BS} were calculated using either the gel strength (GS) or breaking strength (BS) values as follows:

$$\frac{\text{Actual value} - \text{additive value}^*}{\text{Additive value}} \times 100,$$

*i.e. the value derived from summing the contributions of the component proteins as measured in isolation, in proportion to the concentration of each in the mixture.

Interaction of plasma proteins with the proteins of whey, casein and soya. The interaction of 4, 3 and 2% (w/w) commercial porcine plasma proteins with 2, 3 and 4% (w/w) respectively of each of the proteins from whey, casein and soya was examined. A single heat treatment of 95°C for 20 min was employed. The gelling behaviour of 6% (w/w) of the proteins of whey, casein and soya was also tested. All the gels were examined as described previously (Howell & Lawrie, 1984).

Interaction of plasma proteins with stabilizers. To a protein solution (6% w/w) of either commercial porcine plasma, porcine serum or porcine plasma fractions, in either distilled water or 45% sucrose solution, was added 1% (w/w) of either sodium alginate (Keltone, Kelco Co., Ltd, U.S.A.), κ-carrageenan (Copenhagen Pectin Fabrik Ltd) or LM pectin (Copenhagen Pectin Fabrik Ltd).

The effect of calcium was examined by adding 0.8% (w/w) calcium lactate (which is only partially soluble and releases Ca^{++} slowly in the solution) in the presence of 0.4% (w/w) sodium citrate. Gels were produced by heating at 95°C for 20 min. Low pH gels were also prepared by adding 0.4% citric acid to a solution of 6% (w/w) protein, 45% (w/w) sucrose, 1% (w/w) stabilizer, 0.16% sodium hexametaphosphate and 0.16% dicalcium phosphate warmed to 60°C. The gels were allowed to set overnight and tested on the Instron tester the following morning.

Results

Effect of time and temperature of heating on the interaction of plasma proteins with egg albumen proteins

The gel strength (GS) and breaking strength (BS) values for mixtures of plasma and egg albumen were higher than the calculated values obtained by adding the contributions of the individual plasma and egg protein components, as measured in isolation, in proportion to the concentration of each in the mixture ($P < 0.005$). Examples of the results are shown for the GS and BS values for porcine and bovine plasma proteins heated with egg albumen proteins at 85 and 95°C for 15, 30 and 60 min (Tables 1–4). The interaction indices for the GS of the mixtures are shown in Fig. 1. The interactions between porcine or bovine plasma and egg albumen proteins as a function of temperature, time and percent of plasma in the plasma–egg albumen protein mixture is illustrated. For porcine plasma, interaction was highest at temperatures of 80 and 85°C with heating times of 15 or 30 min and plasma concentration about 2% w/w. On the other hand, intraction between bovine plasma and egg albumen was low at 80 and 85°C but higher at higher temperatures (90 and 95°C). As with porcine plasma, however, interaction was higher at 2% (w/w) plasma concentration and at heating times of 15 and 30 min.

Table 1. Effect of time of heating (min) and of protein concentration on the gel strength (g) of either commercial porcine plasma (P) or egg albumen (E) or a mixture of both (P + E), all in 45% sucrose solution at 85°C

	% protein (w/w)				
Time of heating	P 6	P+E 4+2	P+E) 3+3	P+E) 2−4	E 6
15 min (n)	8	8	8	6	8
Mean gel strength	428	437	411	318	101
s.d. ±	18.0	8.8	21.3	31.6	24.8
CV%	4.2	2.0	5.1	9.9	24.4
Additive value	428	319	264	210	101
Interaction index		+ 37	+ 55	+ 51	
30 min (n)	8	9	9	8	8
Mean gel strength	582	510	433	338	137
s.d. ±	21.2	23.9	18.0	20.3	11.4
CV%	3.6	4.7	4.1	5.9	8.2
Additive value	582	434	360	285	137
Interaction index		+ 17	+ 20	+ 18	
60 min (n)	8	8	7	7	8
Mean gel strength	633	534	467	367	172
s.d. ±	34.6	52.8	46.4	17.9	29.8
CV%	5.4	9.8	9.9	4.9	17.3
Additive value	633	422	403	326	172
Interaction index		+26	+ 15	+ 12	

Definition of terms for tables 1–4: n, number of replicates; s.d. standard deviation; CV, coefficient of variation; additive value, the value derived from summing the contributions of the component proteins, as measured in isolation, in proportion to the concentration of each in the mixture; interaction index, $\frac{\text{actual value} - \text{additive value}}{\text{additive value}} \times 100$.

Table 2. Effect of time of heating (min) and of protein concentration on the gel strength (g) of either commercial bovine plasma (P) or egg albumen (E) or a mixture of both (P+E), all in 45% sucrose solution at 84°C

	% protein (w/w)				
Time of heating	P 6	P+E 4+2	P+E 3+3	P+E 2+4	E 6
15 min (*n*)	10	10	10	8	8
Mean gel strength	458	408	333	285	115
s.d. ±	30.4	28.5	15.6	38.5	16.9
CV%	6.5	7.0	4.7	13.5	14.6
Additive value	458	350	291	232	115
Interaction index		+ 16	+ 14	+ 22	
30 min (*n*)	8	10	10	10	8
Mean gel strength	481	450	405	330	154
s.d. ±	30.4	45.7	25.0	32.6	9.8
CV%	6.3	10.1	5.6	9.9	6.3
Additive value	481	372	317	263	154
Interaction index		+ 20	+ 27	+ 25	
60 min (*n*)	9	10	10	9	9
Mean gel strength	625	545	442	350	117
s.d. ±	27.8	15.1	7.8	26.4	12.0
CV%	4.4	2.7	1.7	7.5	6.7
Additive value	625	476	401	326	177
Interaction index		+ 12	+ 10	+ 7	

Table 3. Effect of heating (min) and of protein concentration on the breaking strength (g) of either commercial porcine plasma (P) or egg albumen (E) or a mixture of both (P+E), all in 45% sucrose solution at 95°C

	% protein (w/w)				
Time of heating	P 6	P+E 4+2	P+E 3+3	P+E 2+4	E 6
15 min (*n*)	21	23	22	22	18
Mean breaking strength (g)	1724	1761	1475	841	233
s.d. ±	185.2	155.6	168.5	96.4	42.8
CV%	10.7	8.8	11.4	11.4	18.3
Additive value	1724	1225	978	729	233
Interaction index		+ 43	+ 50	+ 15	
30 min (*n*)	17	21	23	22	17
Mean breaking strength (g)	1835	1721	1431	862	241
s.d. ±	151.9	176.4	112.6	140.6	39.5
CV%	8.2	10.2	7.8	16.3	16.3
Additive value	1835	1303	1038	772	241
Interaction index		+ 32	+ 37	+ 11	
60 min (*n*)	31	32	35	33	20
Mean breaking strength (g)	1966	1749	1486	852	273
s.d. ±	206.5	144.0	150.5	111.8	23.4
CV%	10.5	8.2	10.1	13.1	8.5
Additive value	1966	1401	1119	837	273
Interaction index		+ 24	+ 32	+ 1	

Table 4. Effect of time of heating (min) and of protein concentration on the breaking strength (g) of either commercial bovine plasma (P) or egg albumen (E) or a mixture of both (P + E), all in 45% sucrose solution at 95°C

	% protein (w/w)				
Time of heating	P 6	P+E 4+2	P+E 3+3	P+E 2+4	E 6
15 min (*n*)	12	14	14	16	8
Mean breaking strength (g)	1760	1825	1509	928	241
s.d.±	157.9	242.2	132.5	117.8	35.7
CV%	8.97	13.2	8.7	12.7	14.8
Additive value	1760	1253	1000	747	241
Interaction index		+45	+50	+24	
30 min (*n*)	10	15	14	14	13
Mean breaking strength (g)	1931	1790	1657	983	239
s.d.±	176.9	128.2	112.6	87.0	21.0
CV%	9.1	7.1	6.7	8.8	8.7
Additive value	1931	1366	1085	802	239
Interaction index		+30	+52	+22	
60 min (*n*)	11	13	12	13	14
Mean breaking strength (g)	2065	1883	1589	1026	250
s.d.±	258.1	99.7	101.4	74.2	32.4
CV%	12.4	5.3	6.3	9.2	12.9
Additive value	2065	1459	1157	855	250
Interaction index		+29	+37	+20	

Interaction indices in terms of BS (Fig. 2) showed a high level of interaction for porcine plasma (3 and 4% w/w) at 80°C with a heating time of 15 min. Thus plasma protein concentration of 3 and 4% (w/w) had higher I_{BS} indices than had a concentration of 2%, but lower I_{GS} interaction indices. At 85°C, high interaction index values were observed for all porcine plasma concentrations. With bovine plasma, interaction with egg albumen was highest at 80°C for a heating time of 15 min, and was also high at 95°C at 15, 30 and 60 min. The interaction index was lower with lower bovine plasma protein concentration (except for 2% (w/w) bovine plasma proteins heated for 15 min at 80°C).

The GS and BS interactions of porcine serum with egg albumen followed a pattern similar to that for the porcine plasma–egg albumen interactions, being greatest at 85°C with a heating time of 15 min (Fig. 3), and least at 95°C. The concentration of serum in the mixture did not contribute greatly to the interaction index. The interaction values for serum were about 3–4 times as much as those for porcine plasma at 85 and 90°C but were similar at 95°C.

For plasma fraction I, the interaction index in terms of GS was highest at 4% (w/w) Fr I protein concentration when heated at 95°C for 15 min. At 85°C, 3 and 2% (w/w) Fr I proteins showed high interaction values with 15 min heating. Generally, the interaction decreased with increased time of heating (Fig. 4). The highest level of BS interaction index was obtained on heating 3 or 4% Fr I with egg albumen at 85°C for 15 min. Interaction decreased with increased time, increased temperature and decreased Fr I concentration (Fig. 4).

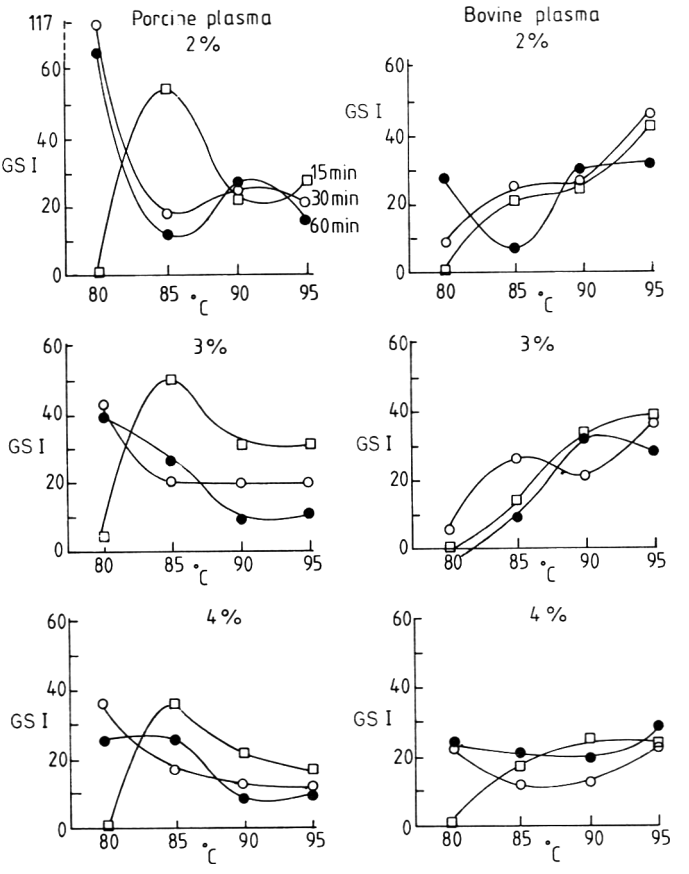

Figure 1. Variation of interaction index (I), in terms of gel strength, with temperature for 2, 3 and 4% commercial porcine and bovine plasma protein levels. In all cases the plasma proteins were made up to 6% (w/w) total protein in 45% (w/w) sucrose solution with egg albumen proteins.

Interaction indices, in terms of both GS and BS, for Fr II are illustrated in Figs 5 and 6. The interaction index was high with decreased time, decreased temperature and increased concentration. As the time–temperature treatments for Fr II were only carried out at 85 and 95°C, a plot of the interaction index against temperature for different protein concentrations has not been drawn.

With Fr III the interaction index I_{GS} was high with 15 min heating at 85°C but, lower with nigher time and temperature of heating and with lower Fr III protein concentration (Fig. 4). The interaction index I_{BS} (Fig. 4a) was highest with 4% (w/w) Fr III proteins heated at 95°C for 15 min. The interaction value decreased rapidly with increased time and with decreased Fr III protein concentration. At 85 and 90°C the concentration of Fr III had a small effect on the interaction index which decreased with increased time (Fig. 4b).

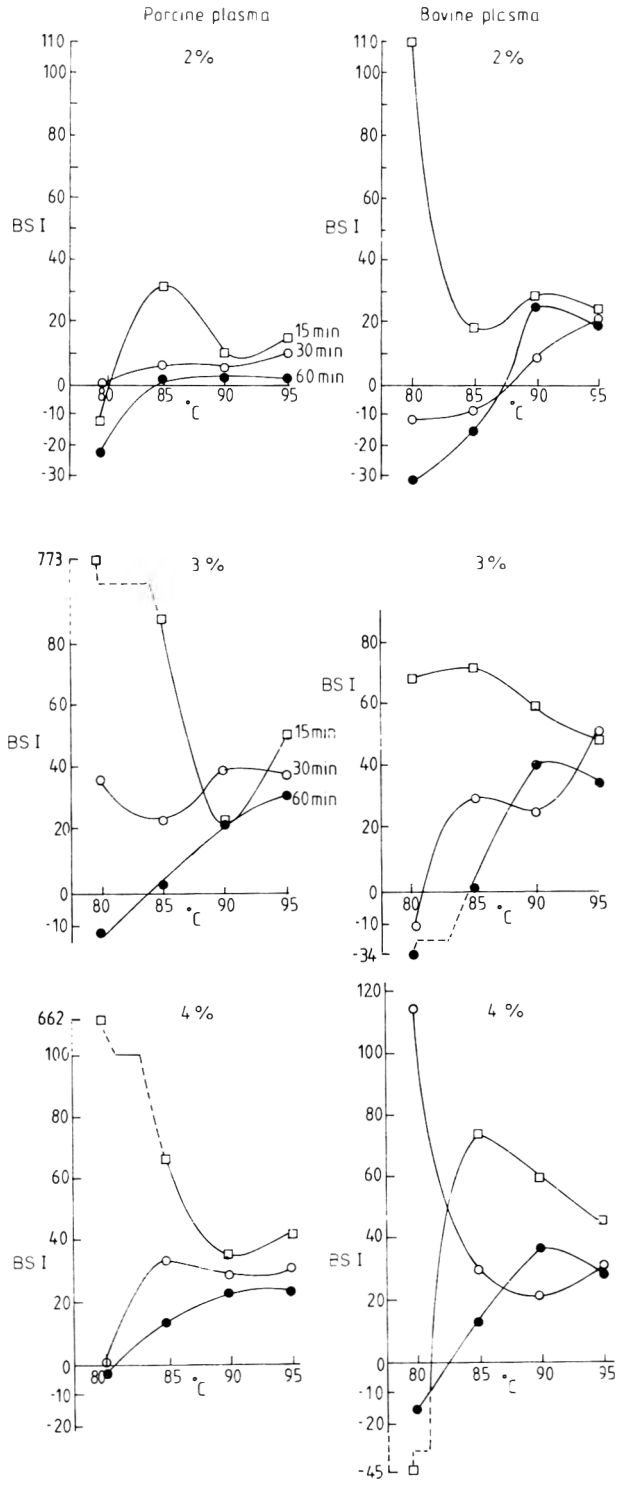

Figure 2. Variation of interaction index (I), in terms of breaking strength, with temperature, for 2, 3 and 4% commercial porcine and bovine plasma protein levels. In all cases the plasma proteins were made up to 6% (w/w) total protein in 45% (w/w) sucrose solution with egg albumen proteins.

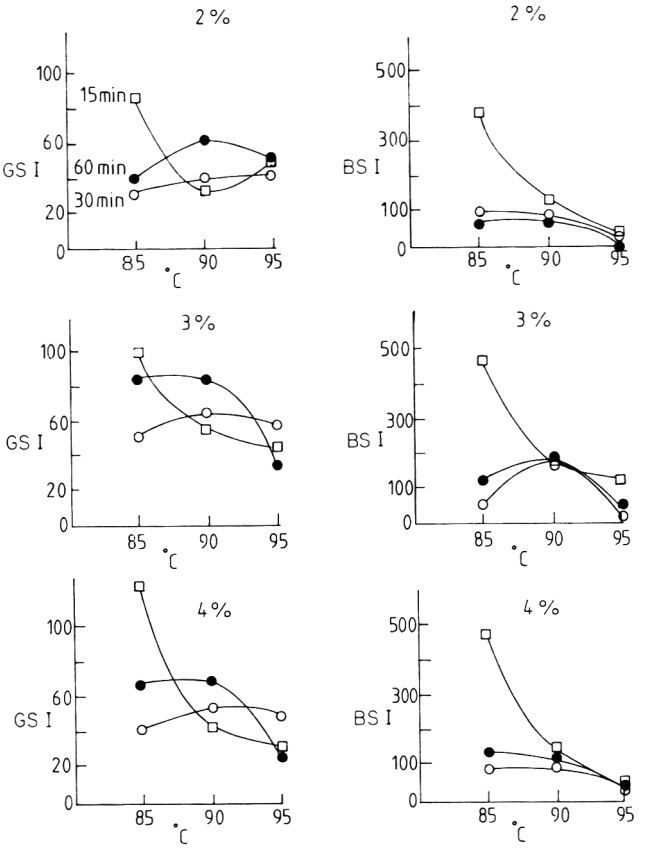

Figure 3. Variation of interaction index (I), in terms of gel strength (GS) and breaking strength (BS), with temperature, for 2, 3 and 4% (w/w) porcine serum protein levels. In all cases the serum proteins were made up to 6% (w/w) total protein in 45% (w/w) sucrose solution with egg albumen proteins with which the interaction occurs.

Interaction of porcine plasma proteins with the proteins of whey, sodium caseinate and soya isolate

Although 6% (w/w) whey proteins in 45% (w/w) sucrose solution produced very weak gels, reasonably strong gels were obtained when 4% (w/w) whey proteins were mixed with 2% (w/w) plasma protein and heated at 95°C for 20 min. However, when heated at 95°C for 60 min, the same mixture indicated negative interaction values for BS and GS (Tables 5 and 6).

All concentrations (2–4% w/w) of porcine plasma fraction I proteins interacted positively with whey proteins (2–4 w/w) when heated at 95°C for 20 min. However, in terms of BS the interaction index was positive only at 2% (w/w) Fr I and 4% (w/w) whey proteins (Tables 5 and 6).

Positive interactions I_{GS} and I_{BS} were found for mixtures of 3% (w/w) Fr II or III and 3% (w/w) whey proteins. Interaction indices for GS ad BS were positive for mixtures of 2% (w/w) porcine plasma, 2% (w/w) whey and 2% (w/w) egg albumen proteins (Table 8).

Sodium caseinate and soya isolate RP 500E did not get at 6% (w/w) concentrations. Negative interactions (i.e. experimental values lower than expected values) were

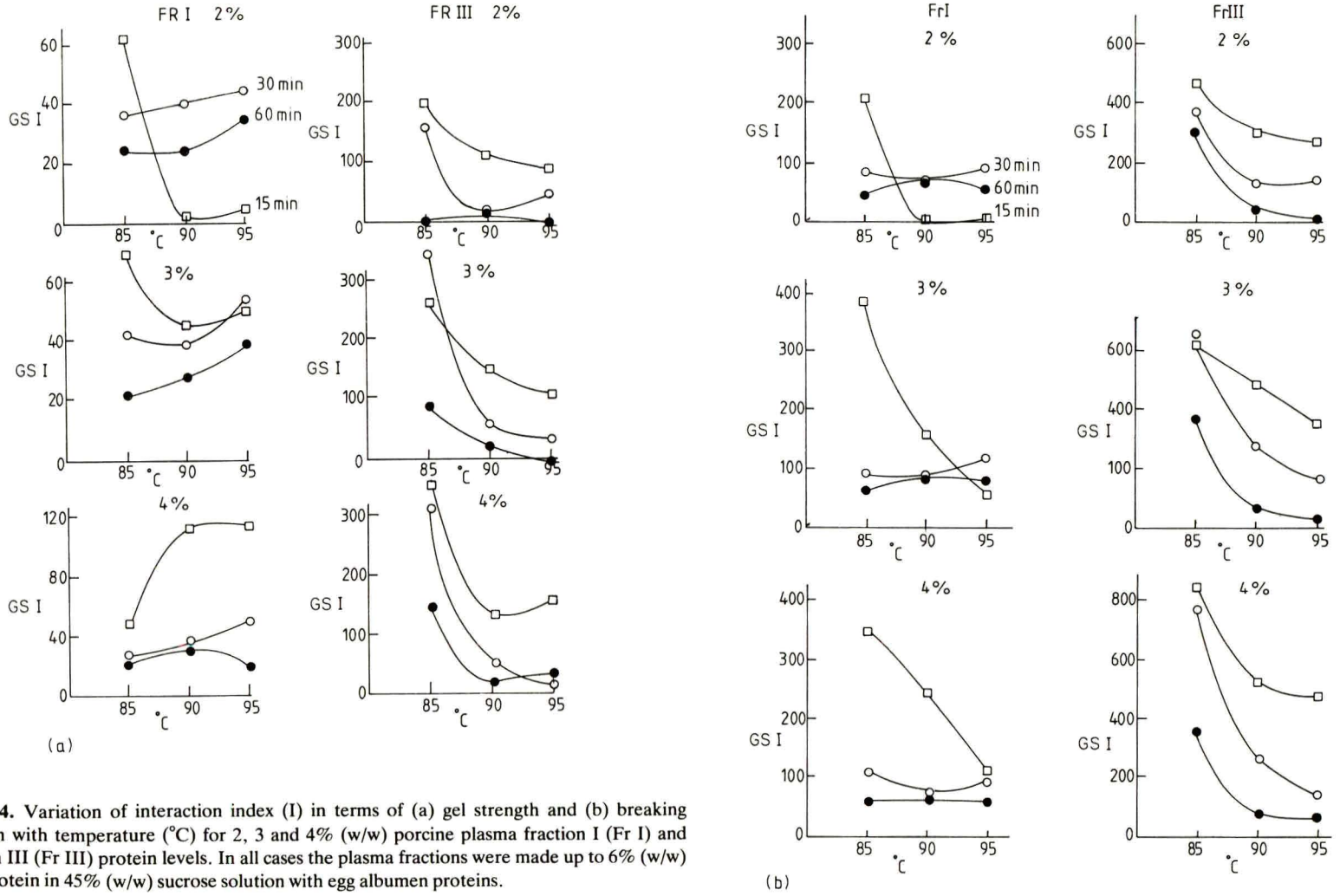

Figure 4. Variation of interaction index (I) in terms of (a) gel strength and (b) breaking strength with temperature (°C) for 2, 3 and 4% (w/w) porcine plasma fraction I (Fr I) and fraction III (Fr III) protein levels. In all cases the plasma fractions were made up to 6% (w/w) total protein in 45% (w/w) sucrose solution with egg albumen proteins.

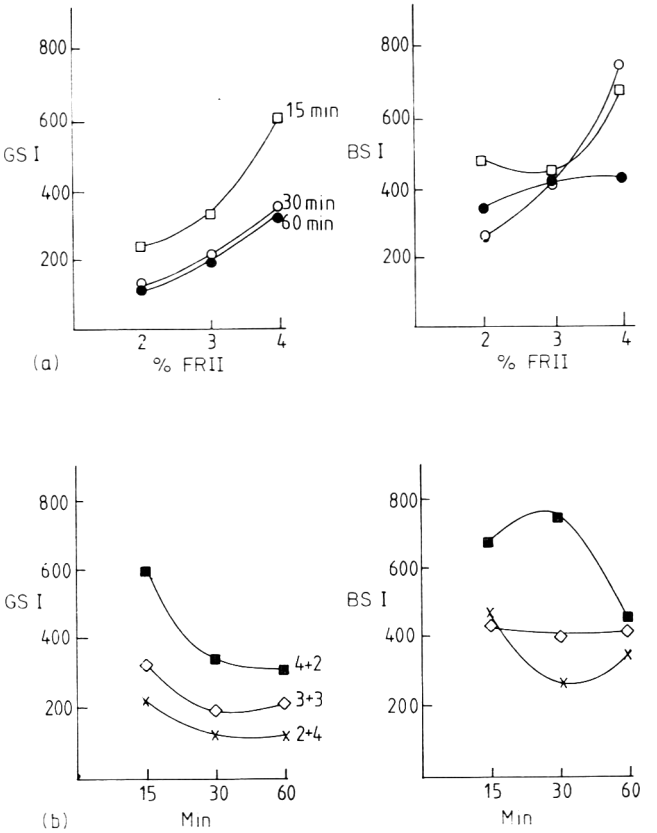

Figure 5. Variation of interaction index (I) in terms of gel strength (GS) and breaking strength (BS), with (a) percent protein, and (b) time of heating for plasma protein fraction II (Fr II) heated at 85°C. In all cases the Fr II proteins were made up to 6% (w/w) total protein in 45% (w/w) sucrose solution with egg albumen proteins.

observed with mixtures of commercial porcine or bovine plasma and either sodium caseinate or soya isolate (Table 7).

A mixture of 2% (w/w) plasma, 2% (w/w) sodium caseinate and 2% (w/w) egg albumin proteins showed positive I_{GS} but negative I_{BS} interaction indices. However a mixture of 2% (w/w) plasma, 2% (w/w) soya isolate and 2% (w/w) egg albumen proteins resulted in positive I_{GS} and I_{BS} interaction indices (Table 8).

Interactions of plasma proteins with stabilizers

The addition of either 1% (w/w) sodium alginate or 1% (w/w) κ-carrageenan, both in the presence of 0.8% (w/w) calcium lactate and 0.4% (w/w) sodium citrate, considerably reduces GS and BS of commercial porcine plasma (6% w/w) in either distilled water or 45% (w/w) sucrose solution. On the other hand, the addition of 1% (w/w) low methoxyl pectin in the presence of 0.8% (w/w) calcium lactate and 0.4% (w/w) sodium citrate did not affect the GS or BS of the porcine plasma gel test but were squeezed flat with considerable syneresis (Table 9). The preparation of gels at low pH (4 or 5) using citric acid also resulted in weak gels with syneresis (Table 10).

It was thought that the addition of calcium, which is necessary for the gelation of these stabilizers, might cause weak gels with plasma due to the precipitation of

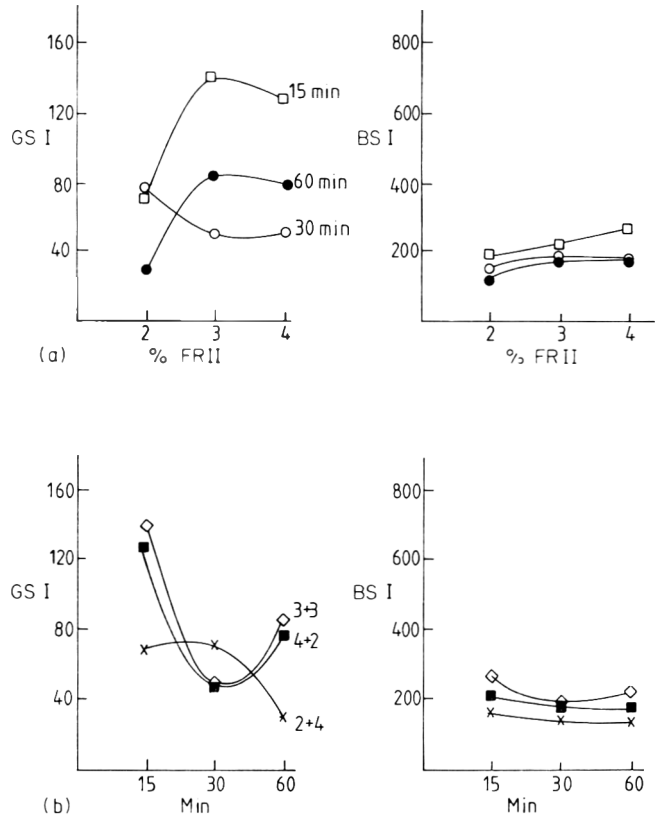

Figure 6. Variation of interaction index (I) in terms of gel strength (GS) and breaking strength (BS) with (a) percent protein, and (b) time of heating for plasma protein fraction II (Fr II) heated at 95°C. In all cases the Fr II proteins were made up to 6% (w/w) total protein in 45% (w/w) sucrose solution with egg albumen proteins.

fibrinogen. Therefore the interaction of porcine serum protein and stabilizers was examined. The breaking strength of 6% (w/w) serum protein gels increased in the presence of calcium lactate but the addition of either 1% (w/w) LM pectin, 1% (w/w) κ-carrageenan or 1% (w/w) sodium alginate reduced the BS (Table 11). The gels were white and crumbly and underwent syneresis. Plasma fraction I produced very weak gels with 1% (w/w) of either sodium alginate, κ-carageenan or LM pectin both in the presence and absence of calcium lactate and sodium citrate. The interaction of plasma fractions II and III with stabilizers was not studied.

Discussion

The interaction index—i.e. the value derived from summing the contributions of the constituent proteins in proportion to what would result using the same concentration of each in isolation—was found to be convenient for studying protein interactions.

An important finding in the gelation of the plasma and egg albumen proteins was the observed interaction between them. Whole plasma, serum, and plasma fractions I, II and III all interacted with the egg albumen proteins. The interaction varied with time and temperature of heating and the concentration of each protein present in the

Table 5. Gel strength and interaction index for gels prepared by heating the proteins of the specified plasma protein (P), whey (W) and mixtures of the two (P+W) at 95°C for 20 min. In all cases the plasma proteins were made up to 6% (w/w) total protein in 45% (w/w) sucrose solution with whey proteins with which the interaction occurs

Protein	% protein (w/w)				
	P 6	P+W 4+2	P+W 3+3	P+W 2+4	W 6
Commercial porcine plasma	686 (±32)*	475 (±33)	407 (±22)	420 (±34)	58 (±7)
Interaction index		0	+9	+57	
Commercial bovine plasma	592 (±40)	462 (±35)	291 (±15)	263 (±14)	58 (±9)
Interaction index		+42	0	+33	
Porcine plasma fraction I	455 (±13)	355 (±7)	385 (±29)	301 (±8)	30 (±6)
Interaction index		+13	+59	+76	
Porcine plasma fraction II	285 (±23)		290 (±5)		39 (±5)
Interaction index			+81		
Porcine plasma fraction III	160 (±15)		290 (±11)		40 (±3)
Interaction index			+190		40 (±3)
60 min at 95°C					
Commercial porcine plasma	813 (±48)	510 (±38)	430 (±30)	300 (±18)	72 (±4)
Interaction index		−9	−3	−5	

*Gel strength (g); (figures in brackets refer to s.d. based on eight replicates).

Table 6. Breaking strength and interaction index for gels prepared by heating the proteins of the specified plasma protein (P), whey (W) and mixtures of the two (P+W) at 94°C for 20 min. In all cases the plasma proteins were made up to 6% (w/w) total protein in 45% (w/w) sucrose solution with whey proteins with which the interaction occurs

Protein	% protein (w/w)				
	P 6	P+W 4+2	P+W 3+3	P+W 2+4	W 6
Commercial porcine plasma	1646 (±30)*	820 (±46)	680 (±54)	473 (±61)	58 (±5)
Interaction index		−26	−19	−19	
Commercial bovine plasma	1580 (±150)	975 (±16)	597 (±17)	542	38 (±9)
Interaction index		−8	−26	−2	
Porcine plasma fraction I	1467 (±61)	758 (±47)	753 (±98)	450 (±62)	38 (±6)
Interaction index		−23	0	12	
Porcine plasma fraction II	310 (±10)		557 (±48)		42 (±6)
Interaction index			+218		
Porcine plasma fraction III	175 (±12)		558 (±52)		40 (±7)
Interaction index			+421		

*Breaking strength (g); (figures in brackets refer to standard deviations based on eight replicates).

mixture. This synergistic interaction might be used to advantage in product formulation to achieve stronger gels with the use of lower amounts of proteins.

Results obtained by plotting the interaction index against time, temperature or concentration enabed comparisons to be made of the interaction of the different plasma proteins with the egg albumen proteins quantitatively (Fig. 1).

Table 7. Breaking strength and interaction for gels prepared by heating the proteins of the specified plasma protein (P) and either (i) sodium caseinate (C) or (ii) soya isolate RP 500E (S) and mixtures of the two (P + C) or (P + S) at 95°C for 20 min

	% protein (w/w)				
(i) Sodium caseinate (C)	P 6	P+C 4+2	P+C 3+3	P+C 2+4	C 6
Commercial porcine plasma	1650 (±88)*	139 (±7)	24 (±5)	0	0
Interaction index		−88	−96		
Commercial bovine plasma	1888 (±13)*	141 (±31)	29 (±13)	0	0
Interaction index		−50	−96		
(ii) Soya isolate (S)	P 6	P+S 4+2	P+S 3+3	P+S 2+4	S 6
Commercial porcine plasma	1753 (±61)	380 (±40)	55 (±5)	50 (±5)	0
Interaction index		−68	−93	−91	
Commercial bovine plasma	1608 (±124)	373 (±56)	58 (±9)	0	0
Interaction index		−65	−93		

*Breaking strength (g).

Table 8. Gel strength, breaking strength and interaction index of gels which were prepared by heating 2% (w/w) porcine (PP) or bovine (BP) plasma, 2% egg albumen (E) and 2% (w/w) (i) whey, (ii) sodium caseinate or (iii) soya isolate RP 500E proteins with which interaction occurred, at 95°C for 20 min

% protein (w/w) in 45% sucrose solution	Gel strength (g)	Breaking strength (g)
(i) PP + W + E (2+2+2)	393 (±25)	863 (±71)
Interaction index	+18	+36
BP + W + E (2+2+2)	406 (±34)	950 (±75)
Interaction index	+40	+54
(ii) PP + C + E (2+2+2)	363 (±22)	410 (±31)
Interaction index	+18	−33
BP + C + E (2+2+2)	363 (±25)	413 (±28)
Interaction index	+18	−33
(iii) PP + S + E (2+2+2)	455 (±32)	562 (±35)
Interaction index	+40	+8
BP + S + E (2+2+2)	428 (±35)	560 (±29)
Interaction index	+38	+8

W: whey; C: sodium caseinate; S: soya isolate.

Table 9. Gel strength and breaking strength of gels which were prepared from commercial porcine plasma and the specified stabilizer by heating at 95°C for 20 min. Each stabilizer (1% w/w) and plasma proteins (6% w/w) were dispersed either in distilled water or 45% (w/w) sucrose solution in the presence or absence of 0.8% calcium lactate and 0.4% sodium citrate

	Gel strength (g)	Breaking strength (g)
Porcine plasma in distilled water	300 (±23)	331 (±29)
Porcine plasma in 45% (w/w) sucrose solution	564 (±39)	1644 (±62)
In distilled water		
Plasma + sodium alginate	135 (±11)	No clear break. Squeezed to a pellet with syneresis
Plasma + sodium alginate + calcium lactate + sodium citrate	Very weak	
Sodium alginate + calcium lactate + sodium citrate	107 (±8)	107 (±8)
Sodium alginate	No gel	No gel
In 45% (w/w) sucrose solution		
Plasma + sodium alginate	100 (±9)	100 (±9)
Plasma + sodium alginate + calcium lactate + sodium citrate	117 (±6)	117 (±6)
Sodium alginate + calcium lactate + sodium citrate	230 (±16)	233 (±15
Sodium alginate	No gel	No gel
In distilled water		
Plasma + κ-carrageenan	194 (±14)	194 (±12)
Plasma + κ-carrageenan	144 (±10)	144 (±10)
κ-carrageenan + calcium lactate + sodium citrate	Weak gel	Weak gel
κ-carrageenan	30 (±2)	30 (±2)
In 45% (w/w) sucrose solution		
Plasma + κ-carrageenan	230 (±10)	230 (±10)
Plasma + κ-carrageenan + calcium lactate + sodium citrate	190 (±8)	195 (±5)
κ-carrageenan + calcium lactate + sodium citrate	Weak	Weak
κcarrageenan	Weak	Weak
In distilled water		
Plasma + low methoxyl pectin	247 (±16)	No clear break. Squeezed to a pellet with syneresis
Plasma + low methoxyl pectin + calcium lactate + sodium citrate	124 (±12)	
Low methoxyl pectin + calcium lactate + sodium citrate	36 (±5)	36 (±5)
Low methoxyl pectin	No gel	No gel
In 45% (w/w) sucrose solution		
Plasma + low methoxyl pectin	552 (±38)	1700 (±96)
Plasma + low methoxyl pectin + calcium lactate + sodium citrate	120 (±7)	Squeezed to a pellet with syneresis
Low methoxyl pectin + calcium lactate + sodium citrate	230 (±13)	230 (±13)
Low methyoxyl pectin	No gel	No gel

The figures refer to mean values based on eight replicates.

Interaction between plasma and egg albumen proteins varied with time and temperature of heating, plasma concentration and species. Porcine plasma–egg albumen protein interaction was high at temperatures of 80 and 85°C and with a relatively short heating time of 15 min (Fig. 1). If the general theory of protein denaturation and unfolding of molecules to expose reactive groups is applied then it appears that, at low time and temperatures of heating, the egg albumen proteins were

Table 10. Gel strength and breaking strength of low pH gels which were prepared by heating commercial porcine plasma and the specified stabilizers at 95°C for 20 min. Each stabilizer (1% w/w) and the plasma protein (6% w/w) were dispersed in 45% (w/w) sucrose solution in the presence of 0.4% (w/w) citric acid, 0.16% (w/w) sodium hexametaphosphate and 0.16% (w/w) dicalcium phosphate

Stabilizer	Gel strength (g)	Breaking strength (g)	pH
1% sodium alginate		80 (±6)	3.5
Plasma + sodium alginate	100 (±8)	No clear break	5.2
κ-carrageenan	Did not gel		3.1
Plasma + κ-carrageenan	200 (±12)	No clear break	3.0
Low methoxyl pectin		90 (±6)	2.8
Plasma + low methoxyl pectin	40 (±5)	No clear break	4.1

The figures refer to mean values based on eight replicates.

Table 11. Breaking strength of gels which were prepared from porcine serum and the specified stabilizer by heating at 95°C for 20 min. Each stabilizer (1% w/w) and the serum protein (6% w/w) were dispersed in distilled water in the presence of 0.8% calcium lactate and 0.4% sodium citrate

6% (w/w) serum protein + 1% (w/w) stabilizer	Breaking strength (g)	Description of gel
Serum	70 (±5)	
Serum + calcium lactate	130 (±8)	Milky syneresis
Low methoxyl pectin + calcium lactate + sodium citrate	20 (±2)	Clear syneresis
Serum + low methoxyl pectin + calcium lactate + sodium citrate	33 (±2)	White, crumbly
κ-carrageenan + calcium lactate + sodium citrate	45 (±3)	Clear
Serum + κ-carrageenan + calcium lactate + sodium citrate	130 (±9)	Milky, spongy
Sodium alginate + calcium lactate + sodium citrate	75 (±8)	Clear
Serum + sodium alginate + calcium lactate + sodium citrate	55 (±5)	Milky

The figures refer to mean values based on eight replicates.

almost completely unfolded. In this state they might be suitable for interaction with the partially unfolded plasma protein. At higher temperatures and times of heating plasma–plasma protein interaction was dominant.

The behaviour of whole porcine plasma, serum and plasma fractions (described below) indicated which proteins were responsible for the interaction with egg albumen proteins. Interaction between porcine serum and egg albumen proteins were 3–4-fold greater than those of porcine plasma at 85 and 90°C; but both were similar at 94°C (Fig. 3). The overall gel strength and breaking strength of the serum protein gels were lower than those of whole plasma—this was attributed to the absence of fibrinogen in a previous paper (Howell & Lowrie, 1984). It appears, therefore, that fibrinogen did not contribute greatly to plasma-egg albumen protein interaction. However, all three plasma fractions I, II and III interacted with the egg albumen proteins. In the main, fraction I proteins, which included fibrinogen, β-globulins and immunoglobulins exhibited little interaction (Fig. 4) with the egg albumen proteins. Fraction II (α-globulins) which formed weak gels only at high temperatures showed high gel strength interaction values when heated at 85–95°C with egg albumen proteins (Figs 5

and 6), and fraction III (albumin) exhibited both high gel strength and breaking strength interaction values (Fig. 4). In addition, the interaction pattern of fraction III (albumin) with egg albumen proteins was similar to that of whole porcine plasma—i.e. greatest interaction at low temperature (80 and 85°C) over 15 min. At higher temperatures (90 and 95°C) and over 60 min, the gel strength and breaking strength values increased rapidly suggesting that albumin–albumin interaction may have been dominant (Fig. 4). Moreover, as albumin constitutes 50% (w/w) of the plasma proteins it may be concluded that albumin is mainly responsible for the gelation and interaction of whole plasma with the egg abumen proteins.

Unlike porcine plasma proteins, bovine plasma proteins exhibited greatest positive interaction with egg albumen at the higher heating temperatures of 90°C (Fig. 2) and negative interaction at 85°C (Fig. 2).

The difference in the behaviour of porcine and bovine plasma proteins might be due to, for example, the difference in the N terminal and C terminal amino acids of bovine and porcine albumin (Low, 1970; Brown, 1975). However, it is not clear how the conformational changes resulting from the differences in primary structure affect gelation. For both porcine and bovine plasma proteins, interaction with egg albumen proteins, as judged by the gel strength and breaking strength values, was highest at low plasma concentration namely 2% (w/w) and 3% (w/w) respectively (Figs 1 and 2). Higher plasma protein concentration probably resulted in plasma–plasma protein interaction.

In practice the type of protein species might be chosen depending on the food product and processing conditions involved. For example, bovine plasma proteins might be used to interact with egg albumen proteins at high temperature whereas porcine plasma proteins might be employed at lower temperatures.

The effect of other proteins on the plasma proteins highlighted the importance of the compatibility of different ingredients in a product. As with egg albumen proteins, a small degree of heating of the plasma proteins encouraged interaction with the whey proteins. This combination might be used in cake or meat products to enhance gelation.

But mixtures of whole porcine plasma and sodium caseinate produced only weak gels (Table 7).

Neither soya isolate RP 500E (6% w/w) or a mixture of porcine plasma and the soya isolate formed gels on heating. This soya isolate usually gels at high protein concentrations of 12% (w/w) (unpublished data). Unlike plasma, whey and egg albumen protein gels, which are elastic, this soya isolate gel was pasty. The differences in the gelling mechanism of the plasma proteins and the soya isolate could have prevented the interaction of the two proteins but this aspect requires further study.

On adding the stabilizers to a solution of whole plasma or serum, the gelling ability of these proteins was greatly reduced (Table 11). It is not clear what factors were responsible for this behaviour. Two possible factors might be the binding of the anionic stabilizer to the basic groups on the protein molecules; and steric hindrance by the high molecular weight polysaccharide polymers. Both aspects require further sudy.

Conclusion

The interaction of the plasma proteins and the proteins of egg albumen, whey, casein and soya and also stabilizers is a potentially useful and curious phenomenon which cannot be explained by our current knowledge of these proteins and of gelation theory.

Further work is being carried out on the relationship of the structure of food proteins and their functional properties to increase our understanding in this area.

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Analysis of the interface conditions during drying of rice

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Summary

The influence of the relative air humidity on the surface conditions of rice grain during drying was investigated. It was found that the equilibrium condition given by the desorption isotherm of the product is not valid over the whole range of relative humidity analysed. While this condition was verified with air of high relative humidity (between 43 and 59% for drying temperatures ranging from 40 to 70°C), the experimental results indicated that the surface moisture approaches the monolayer value given by the Brunauer, Emmett and Teller (BET) desorption isotherm when drying takes place with air of low relative humidity (7 and 14% for the same temperature range). An explanation of these facts is suggested taking into account the high value of the heat of desorption associated with the monolayer moisture content of the product under investigation. Finally, a method based on the Fick's second law solution when the time of drying becomes large enough is proposed to evaluate the surface moisture content of the grain independently of the conditions of the air drying.

Introduction

It is possible to find in the literature many mathematical models for the description of the drying process (Sherwood, 1929; Harmathy, 1969; Berger & Pei, 1973). The application of these models to a particular food product generally requires some simplifications applicable only under very specific circumstances. In grain drying, the assumption that the heat transfer effects in the grain can be neglected (and the process treated as a purely mass transfer one) has been used with satisfactory results by many investigators (Becker & Sallans, 1955; Suárez, Viollaz & Chirife, 1980; Fortes, Okos & Barret, 1981).

Becker & Sallans (1955) developed a mathematical model based on Fick's law of diffusion to analyse the experimental drying curves of wheat kernel. In that work the authors used the standard solution of the non-stationary-state diffusion equation for diffusion out of spheres, assuming moisture diffusivity independent of grain moisture content. As was previously observed by Jones (1951), the authors determined that the effective surface moisture content of the material reaches a constant value, virtually independent of the temperature level. They explained this by suggesting that this value represents 'the upper level of more firmly held water' and consequently a different drying behaviour above and below this value must be expected. Subsequently, Becker (1958) tried to correlate the value of the effective surface moisure with the sorption

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properties of the product. With this purpose, the equilibrium moisture content data of wheat where analysed within the framework of BET theory, in order to establish a relation between the interface conditions existing during drying and the monolayer value predicted by the BET equation. This idea was finally rejected because of the marked variation of the monolayer value with temperature, which was in disagreement with the facts previously observed during drying. However, the author found an exact correlation between Langmuir parameters m_L and m_1 (related with the number of localized sites and occupied at saturation, respectively) and the surface moisture content value $m_{s\infty}$ and m_{so} where $m_{s\infty}$ is the value toward which the surface moisture tends with time and m_{so} corresponds to the initial drying conditions.

Although the methodology developed by Becker and co-workers gives a reasonable interpretation of the experimental facts observed during wheat drying, the same cannot be applied outside the limits of the experimental conditions used by these authors (in particular, the approximate solution of Fick's second law used to evaluate the surface moisture cannot be used when the grain moisture content is below 12%). In this study, a different analysis of the interface conditions during rice dehydration is given. The methodology employed proposes to be general enough and is not limited to a predetermined value in the grain moisture content. On the other hand, the experimental fact found in this study that the surface moisture of the grain depends on the drying temperature is in disagreement with the results reported by Becker and co-workers and therefore does not permit the application of the methodology employed by these authors to the analysis of rice drying conducted in this investigation.

Four drying runs were undertaken in this work at 40, 50, 60 and 70°C, with the relative humidity of the air varying from 7 to 14%. This set of experiments—which will be indicated for convenience as drying runs at low relative humidity (LRH) to differentiate from another set of runs conducted at higher relative humidities and reported in a recent paper Aguerre, Suárez & Viollaz (1982)—are analysed in order to investigate the behaviour of the surface of the rice grain during drying in air flow for a wide range of operating conditions. An analysis of the drying process operating with air previously humidified by blending the air with saturated steam has been performed by Aguerre *et al.* (1982). In that investigation the relative humidity (RH) of the air was varied from 43 to 59% for the same drying temperatures used in the present work (this set of experiments is indicated as drying of high relative humidity, HRH). The results obtained in that work can be summarized as follows: (i) the migration of moisture in the grain can be interpreted in terms of Fick's law of diffusion; (ii) the diffusion coefficients at the different drying temperatures were calculated and found not to depend on the moisture content of the grain in the range of moistures investigated; and (iii) the surface of the rice grain is in equilibrium with the RH of the air and its value given by the desorption isotherm of the drying material. However, certain experimental facts would indicate that the same criteria cannot be used when drying occurs with air of LRH, as usually happens in drying operations. In other words, it can be said that this criterion depends particularly on the conditions of the drying. Accordingly, the purpose of this investigation is to establish the limitations of this criterion as well as to present a general method to evaluate the interface conditions of the rice grain during dehydration.

Materials and methods

Materials

A local variety of medium rough rice grain was used in the experiments. The grains

were harvested in March 1983 and field-dried to a moisture content of 13% (dry basis). The grains were screened to obtain samples of more uniform size, and the size distribution used in the range 2.8–3.3 mm. For a better characterization the major and minor axes of the grain were measured by means of a dial micrometer. The average values obtained over sixty grains were 8.81 and 2.19 mm respectively. The equivalent radius of the grain was also determined from specific volume measurements made by displacement of chlorobenzene.

The grains were humified before dehydration experiments by placing them in thin layers on trays in vacuum desiccators over pure water at 4°C until they attained the equilibrium moisture content (22%, dry basis). The dry matter content of the grains was determined by placing the samples in a vacuum oven at 70°C, over magnesium perchlorate, for 96 hr.

Drying equipment

The dryer consisted of a centrifugal fan to supply the necessary air flow, an electric heater, and an air filter and an electronic proportional temperature controller. The air was blown by the fan through the electric heater to a rectangular duct. At the outlet of the duct was placed a basket with a screened bottom and lid. For each experiment 20 g of grain were placed into the basket, forming a thin layer. The drying process was monitored by weighing the sample periodically on a precision balance (±0.0001 g).

The drying experiments were carried out at four temperatures (40, 50, 60 and 70°C). In each run the air temperature was controlled by means of a proportional controller (±0.1°C). The dry-bulb and wet-bulb temperatures were measured and the relative humidity of the air calculated from the wet-bulb depression; the relative humidity varied between 7 and 14%. An air velocity of 18 m/sec was used during the experiment; it was measured with a velocimeter connected at the outlet of the duct. The equilibrium moisture content (corresponding to the desorption isotherm) at the different drying temperatures, were determined by equilibrium against saturated salt solutions which provided known constant relative humidities. These values are reported in Table 1.

Results

The drying runs conducted at four temperatures are plotted in Fig. 1 in terms of $\bar{m}/m_o$ *versus* time (Θ). the variables $\bar{m}$ and m_o are the moisture of the grain respectively,

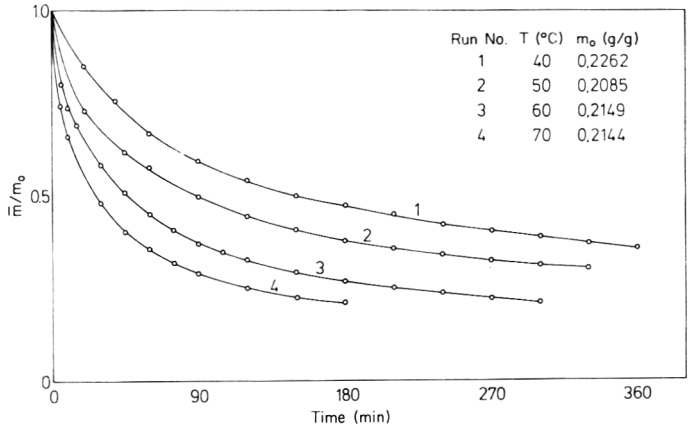

Figure 1. Moisture content variation with time. Air velocity: 18 m/sec.

expressed on a dry basis. The air velocity employed in all drying experiments was 18 m/sec. As reported by Aguerre *et al.* (1982), operating with air velocities greater than 12 m/sec, the drying rate is not modified. Hence, it can be concluded that the mechanism of rice drying under the specified conditions is totally controlled by moisture migration in the grain.

The analysis of rice drying was conducted under the following assumptions:

(i) The diffusion coefficient, at a given drying temperature, does not depend on the grain moisture content. This assumption was made by different investigators (Becker & Sallans, 1855; Steffe & Singh, 1980; Suárez *et al.*,, 1980), during the drying of different cereal grains, with satisfactory results.

(ii) The grain temperature remained constant and equal to the air dry bulb temperature practically during the whole drying process. Due to the relatively low initial moisture content of the grain and the small value of the Biot number for heat transfer usually found in food drying (Vaccarezza, Lombardi & Chirife, 1974; Alzamora and Chirife, 1980) it is possible to neglect the internal temperature gradients of the grain.

(iii) The surface moisture content remains constant during the dehydration process. Its determination is discussed.

(iv) The effect of shrinkage of the grain was not considered, given the small size differences observed at the beginning and the end of the drying experiments (less than 4%).

Based on these considerations, rice drying can be considered as a process controlled by the migration of moisture in the grain where the heat transfer effect can be neglected (on the other hand, the results reported by Fortes *et al.* (1981) during wheat drying simulation seem to confirm this assumption). Consequently, Fick's solution for diffusion out of spheres, with constant diffusion coefficient and boundary conditions of the first kind, is employed in our analysis. This leads to the following equation, expressed in terms of moisture content results (Luikov, 1968):

$$m^* = (m - m_s)/(m_o - m_s) = 6/\pi^2 \sum_{\kappa=1}^{\infty} \frac{1}{\kappa^2} \exp(-\kappa^2 \pi^2 D\theta/r^2), \quad (1)$$

where D is the diffusion coefficient, r the equivalent radius of the sphere and m_s the surface moisture content (dry basis). The experimental values of m^* were processed by means of Eqn (1) for each drying experiment. As a first approximation for the estimation of the values of m_s, the surface moisture content was calculated from the

Table 1. Comparison between m_s values predicted by isotherm and Eqn (3) for drying runs at LRH

T (°C)	RH (%)	m_s (g/g) (isotherm)	m_s (g/g) (Eqn 3)
40	14	0.0511	0.0651
50	9	0.0321	0.0532
60	5	0.0173	0.0447
70	7	0.0113	0.0404

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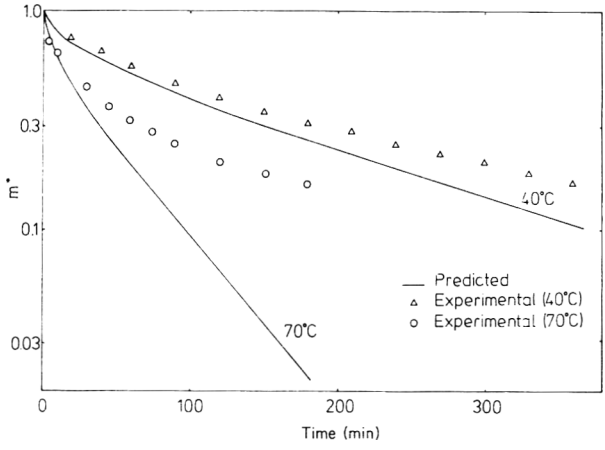

Figure 2. Comparison of experimental and predicted (Eqn 1) drying curves. Surface moisture content calculated from desorption isotherms.

desorption isotherms of the product. These values were calculated from the equilibrium data, corresponding to the desorption branch of the sorption isotherm of rough rice grain; they are given in Table 1 for the drying conditions of each run.

To simulate drying, Eqn (1) was programmed on a digital computer and the values of m^* fitted by means of a non-linear regression method (the parameter D was allowed to vary). The results comparing the predicted and experimental drying curves, for two drying experiments, are shown in Fig. 2. From these curves it can be seen that the experimental results deviate considerably from the predicted ones. To explain the reason for these deviations the results of this analysis are now compared with those recently reported by Aguerre *et al.* (1982) with reference to rice drying with air of HRH. In that work the authors found that the analysis of the experimental drying data by means of Eqn (1) provided a good description of the drying phenomenom. Consequently the diffusivities corresponding to the migration of moisture in the grain, were calculated at 40, 50, 60 and 70°C (it must be pointed out that the value of m_s for each drying run is calculated from the desorption isotherm).

Considering the results reported by Aguerre *et al.* (1982) together with those obtained in this work, it seems reasonable to try to explain the apparent non-'Fickian' behaviour shown in Fig. 2, considering that the cause of this deviation must originate in certain anomalous conditions existing at the grain surface and not in a possible change of the mechanism of drying, suggested by Becker & Sallans (1955). In other words, the results obtained operating with air of LRH would indicate that the surface moisture content of the grain, during drying, tends to a value which is not necessarily that predicted by the desorption isotherm.

To estimate the value of the surface moisture content the following alternative method was used. When the time becomes large the limiting form of Eqn (1) results:

$$m^* = (\bar{m} - m_s)/(m_o - m_s) = 6/\pi^2 \exp(-\pi^2 D\,\theta/r^2). \quad (2)$$

From this equation it can be also shown (Shivhare, Maharaj & Singh, 1982) that for any set of three moisture contents taken at equally spaced time intervals of duration j, the following expression can be obtained:

$$m_s = (\bar{m}_i \cdot \bar{m}_{i+2j} - \bar{m}_{i+j}^2)/(m_i + \bar{m}_{i+2j} - 2\,m_{i+j})$$

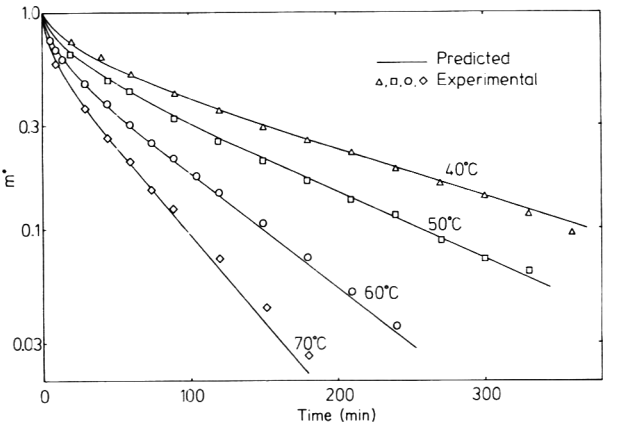

Figure 3. Comparison of experimental and predicted (Eqn 1) drying curves. Surface moisture content calculated from Eqn (3).

$\bar{m}_i$ and $\bar{m}_j$ being the average moisture contents of the grain at time i and j. Equation (3) was used to calculate the surface moisture contents of the rice grain for the different drying temperatures; these values are given in Table 1. The equilibrium moisture contents were also included in this table in order to observe the discrepancy between the corresponding pair of values. It can be seen that this discrepancy becomes more important while the air relative humidity decreases. The new estimations of m_s were used to calculate the dimensionless variable m^+ and the resulting values processed by means of Eqn (1) using the diffusion coefficients obtained from the drying runs performed at HRH (Aguerre *et al.*, 1982). The experimental and predicted curves are compared in Fig. 3, the agreement being, in this case, totally satisfactory.

Equation (3) was also tested to predict the surface moisture for the drying runs conducted at HRH. The results are given in Table 2 and show that the surface moisture contents obtained by means of Eqn (3) and from desorption isotherms are practically the same for conditions of HRH. This results suggests that Eqn (3) can be used to determine the surface moisture content of the product under investigation, for both HRH and LRH drying conditions.

Discussion

The analysis of the drying kinetic data carried out in the present study suggests that the calculation of the surface moisture content of the rice grain from the desorption

Table 2. Comparison between m_s values predicted by isotherm and Eqn (3) for drying runs at HRH.

T (°C)	RH (%)	m_s (g/g) (isotherm)	m_s (g/g) (Eqn 3)
40	44	0.090	0.091
50	59	0.107	0.106
60	43	0.066	0.066
70	57	0.070	0.069

Table 3. Comparison of the surface moisture contents (effective or dynamic) with the monolayer values (m_m) obtained from BET theory

T (°C)	m_m (g/g)	m_s (g/g) (runs at HRH)	m_s (g/g) (runs at LRH)
40	0.063	0.090	0.0651
50	0.052	0.107	0.0532
60	0.044	0.066	0.0447
70	0.039	0.070	0.0404

isotherm data is not always an appropriate method. It has been shown that the differences between the 'static' and 'dynamic' moisture sorption (terms introduced by Becker and co-workers to differentiate between the surface moisture values obtained from isotherm and drying experiments, respectively) increase, while the air relative humidity decreases. This fact can be quantified when the values of the surface moisture content obtained from equilibrium considerations for the drying runs conducted at LRH and HRH, are compared with the corresponding values obtained by means of Eqn (3) as given in Tables 1 and 2. While the differences between the static and dynamic surface moistures were not significant for the drying runs at HRH (which justified the use of the static equilibrium moisture given by the desorption isotherm instead of the dynamic one), they became more important for the drying experiments conducted by LRH.

In order to give an explanation of the observed behaviour, the monolayer moisture contents of rough rice reported by Suárez *et al.* (1983) in the range 40–70°C is considered and compared with the dynamic moistures sorption obtained for both groups of runs. All these values are included in Table 3; it is observed that for the runs conducted at HRH the surface moisture contents obtained through Eqn (3) are considerably higher than the monolayer values but are practically equal at LRH. This fact suggests that when drying takes place with air with continuously decreased humidity content, the surface moisture of rice grain tends to a limit value given by the monolayer value of the desorption isotherm of the product.

The results obtained in this investigation do not agree with those reported by Becker (1958) in his analysis of the surface conditions during wheat drying. While the author rejected the BET theory, given the marked dependence of the monolayer moisture of wheat grain on temperature as found experimentally, the results obtained here, reveal that the same can be interpreted within the framework of BET theory. Furthermore, the analysis of the data suggests that, within the limits of the temperatures investigated, the variation of the dynamic and monolayer moisture sorption with temperature is practically the same, and, therefore, it can be concluded that the surface moisture of the rice grain tends towards the monolayer moisture of the grain when the humidity content of the air is sufficiently low.

This fact could be related to the difficulty to remove the water associated with the monolayer moisture (Labuza, 1968). It is well known that the heat of desorption in foods of very low moisture contents are very high, amounting to 2–2.5 times the heat of vaporization of pure water (Iglesias & Chirife, 1976). Particularly, in the case of rice grain the mean heat of desorption is about 9 kcal/mol (Suárez & Viollaz, 1983) for moisture contents corresponding to the monolayer values and decreases very rapidly for higher moisture contents. Based on these considerations, it seems reason-

able to assume that when drying is conducted under conditions where the surface moisture of the grain is above the monolayer value—i.e. for the drying runs at HRH—the equilibrium assumption is valid. Therefore, in these conditions it can be said that the difference between static and dynamic moisture sorption, if it exists, cannot be observed. On the other hand, considering that the heat of desorption corresponding to the drying conditions previously mentioned are markedly higher than the heat of vaporization of pure water (Suárez *et al.*, 1983), it is reasonable to consider that the surface of the grain does not offer any resistance to the water evaporation, and hence the drying process is totally controlled by the migration of moisture in the grain. On the contrary, when drying takes place with air of relative humidity less than that corresponding to the equilibrium value with the monolayer moisture, the equilibrium condition is no longer valid. As was previously mentioned, the high values of the heat of desorption associated with the monolayer values could be an important reason for the difference between the static and dynamic moisture sorption experimentally oberved. From the point of view of a drying mechanism, this fact would justify the hypothesis that the surface of the grain is an additional resistance to the water evaporation process during drying.

Conclusions

It is possible to conclude from the analysis of drying data of rough rice grain that the equilibrium moisture predicted by the desorption isotherm is not always an appropriate value to estimate the surface conditions of the product during drying. The alternative method given in this paper, based on a Fick's law solution, is not only a very simple method but also an accurate one.

The information that the desorption isotherm provides is however very valuable in the sense that permits the explanation of the influence of the air humidity upon drying kinetics. The apparent deviation of the 'Fickian' behaviour observed when drying takes place with air of LRH has been explained as a consequence of the departure of the interface conditions from the equilibrium. Conversely, the monolayer values predicted by the BET equation from data of equilibrium moisture content reported previously (Suárez *et al.*, 1983) are the limits toards the surface moisture approaches when the air relative humidity is decreased. The high values of the heat of desorption related to the monolayer moisture contents seem to be a reasonable explanation of the effect observed.

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Calculation of the variation of the heat of desorption with moisture content on the basis of the BET theory

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Summary

Data of water desorption on different food products were analysed from the point of view of the BET theory. A plot of log C against 1/T as used for the independent evaluation of E_1-E_L and the pre-exponential a_1b_2/b_1a_2 in the expression for the constant C of the BET theory. It was found that a_1b_2/b_1a_2 may range from 10^{-3} to 10^{-6} and the E_1-E_L values are correspondingly much higher than usual. An equation has been obtained within the framework of the BET theory for the dependence of the net heat of desorption on moisture content. In the present analysis the variation of the monolayer value with temperature is considered by means of a simple empirical equation. The results are compared with the heats of desorption calculated directly by the Clausius–Clapeyron equation.

Introduction

The object of this paper is to analyse the sorption isotherms of various food products available in the literature from the point of view of the BET theory. An important characteristic of the BET model is that it provides a simple method to estimate the heat of sorption (ad-desorption) and the monolayer value of coverage, both parameters of importance in food processing and storage.

According to this theory, Eqns (17) and (22) of the paper of Brunauer, Emmett & Teller (1938) can be combined to give:

$$C = \frac{a_1b_2}{b_1a_2}\exp(E_1-E_L)/\mathrm{RT}, \tag{1}$$

E_1 being the heat of sorption of the first layer and E_L the heat of liquefaction. In the absence of any information on the ratio a_1b_2/b_1a_2, the original postulate of the BET theory was that $b_1/a_1 = b_2/a_2$, which is the equivalent of considering the pre-exponential of Eqn (1) equal to unity. With this assumption the relation between E_1-E_L and C is given by the simplified expression:

$$E_1-E_L = \mathrm{RT}\ln C. \tag{2}$$

Equation (2) was used in the literature to estimate the heat of sorption of a wide variety of substances and food products (Masuzawa & Sterling, 1968; Bettelheim & Volman, 1957; Bushuk and Winkler, 1957; Volman *et al.*, 1960; Iglesias and Chirife, 1976; Mohlin and Gray, 1974; Roman, Urbicain & Rofstein, 1982). However, it is a fact

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Table 1. Values of C and u_m for different food products

	5		20		25		30		35		45		47		50		65	
Material	C	u_m (%)	C	u_m (%)	C	u_m (%)	C	u_m (%)	C	u_m (%)	C	u_m (%)	C	u_m (%)	C	u_m (%)	C	u_m (%
Thyme	108	6.52	—	—	41	5.27	—	—	—	—	19.8	3.75	—	—	—	—	—	—
Aniseed	76.2	5.0	—	—	58.4	4.44	—	—	—	—	13.2	4.42	—	—	—	—	—	—
Marjoram	75.1	7.48	—	—	43.7	4.96	—	—	—	—	7.86	3.64	—	—	—	—	—	—
Fennel	23.7	4.24	—	—	12.1	3.54	—	—	—	—	5.24	2.79	—	—	—	—	—	—
Wheat kernel	—	—	—	—	—	—	33.8	7.3	26.6	7.09	—	—	—	—	14.9	5.97	—	3.8
Sugar beet root	—	—	12.02	5.55	—	—	—	—	10.0	4.95	—	—	7.0	4.99	—	—	4.39	—

commonly accepted among many investigators that the values of $E_1 - E_L$ determined by Eqn (2), the so-called BET heats of sorption, are in general much lower than the calorimetric or isosteric heats and consequently of little practical interest.

A considerable discussion exists in the literature about the simplifying assumption of the BET theory and consequently of the validity of Eqn (2) to predict heat of sorption. Cassie (1945) based on statistical considerations determined that the ratio of the constants of Eqn (1) should be much lower than unity. The author suggested that alteration in this direction would give rise to better agreement between BET and other heats of sorption (experimental or isoteric). Kemball & Schreiner (1950) found that the ratio $a_1 b_2 / a_2 b_1$ may vary from 10^{-5} to 10, dependng on the system sorbate-sorbant. The authors determined a relationship between these coefficients and the entropy change accompanying the sorption process, concluding that the single isotherm method can only be used in the cases where this change of entropy is known.

Based on these considerations, the water sorption equilibrium data of various food products available in the literature were analysed within the framework of the BET theory with two purposes. First, to demonstrate that in all the cases investigated the pre-exponential factor of Eqn (1) differs markedly from unity and that this equation gives heats of sorption which are more in agreement with the isosteric ones. Second, to apply the Clausius–Clapeyron equation to the BET equation in order to predict the dependence of the heat of sorption on moisture content. In this part of the investigation, an empirical equation was assumed to take into account the variation of the monolayer moisture content with temperature.

Results and discussion

Estimation of the BET heat of desorption

Six food products, of which desorption isotherms at various temperatures (three or more) are available in the literature, were used for our analysis. These were thyme, aniseed, marjoram and fennel (Wolf, Spiess & Jung, 1973), wheat kernel (Day & Nelson, 1965) and sugar beet root (Iglesias *et al.*, 1975). The equilibrium data of the first five products were analysed according to the BET equation:

$$\frac{a_w}{u(1-a_w)} = \frac{1}{Cu_m} + \frac{C-1}{Cu_m} a_w, \tag{3}$$

in the range $0.05 < a_w < 0.5$; a_w being the water activity and u and u_m the equilibrium and monolayer moisture contents respectively (dry basis). The values of C and u_m for each product at different temperatures (obtained by linear regression of the data $a_w/u(1-a_w)$ *versus* a_w in the range of the a_w specified) are given in Table 1. In the case of sugar beet root, the values of C and u_m given in this table are those reported by Iglesias *et al.* (1975). It can be observed for each product that a regular decay of the values of C and u_m with increasing temperature exists.

The BET heat of desorption (E_1-E_L) in each case was calculated through Eqn (1) by plotting the values of ln C against 1/T. These are given in Table 2 together with the values of $\kappa = a_1b_2/b_1a_2$. It can be seen that the κ values differ markedly from unity in all materials investigated and that therefore, the calculation of E_1-E_L from the simplified equation gives in all cases net heats of desorption lower than those calculated from Eqn (1), as can be observed in Table 2.

Calculation of the variation of the heat of desorption with moisture content

The following method was employed in this investigation to obtain the dependence of the net heat desorption on the moisture content by application of the Clausius-Clapeyron equation to the BET model. An expression relating the isosteric heat of sorption to E_1-E_L and the amount of gas adsorbed was developed by Berezin & Kiselev (1966). The equation derived by the authors was tested with experimental data assuming for C and u_m a constant value (obtained from equilibrium data at only one temperature). The results of this comparison indicated that the analytical expression gives heats of sorption which are too low compared with experimental data. Such discrepancy was attributed to actual occurence of sorbate–sorbate interactions which are not considered in the original postulates of the BET theory.

A modification of the solution given by Berezin & Kiselev (1966) was proposed by Suárez *et al.* (1983) to estimate the dependence of the heat of desorption on moisture content in rice grain, with satisfactory results. In that paper the authors included the variation of the heat of desorption with moisture content and the dependence on temperature of the parameters C and u_m of the BET equation. A similar treatment will be used here taking into account the results reported in Table 1.

Table 2. Comparison of the heats of desorption calculated from Eqns (1) and (2)

Material	E_1-E_L Eqn (1), kcal/g-mole	E_1-E_L Eqn (2) kcal/g-mole	κ
Thyme	7.46	2.2	$1.46\ 10^{-4}$
Aniseed	7.57	2.1	$1.05\ 10^{-4}$
Marjoram	9.79	2.0	$1.87\ 10^{-6}$
Fennel	6.61	1.4	$1.58\ 10^{-4}$
Wheat kernel	7.87	1.9	$7.09\ 10^{-5}$
Sugar beet root	4.50	1.5	$5.72\ 10^{-3}$

To determine the dependence on T of the variable a_w we start from a modified expression of Eqn (3). This equation can be rearranged in the following way:

$$a_w = 1-\frac{1-(1-2r/(1+u/u_m))^{1/2}}{r}, \qquad (4)$$

where

$$r = \frac{2u}{u_m}\left(\frac{1-1/C}{1+u/u_m}\right). \tag{5}$$

According to the BET theory, the influence of the temperature on the sorption isotherm is only given by the dependence of C on T. In this paper the more general expression (given by Eqn 1) instead of the simplified equation (Eqn 2) proposed in the BET theory, is used to relate this parameter to the temperature. This criterion was adopted taking into account that the pre-exponential factor of Eqn (1) was in all cases very different from unity.

It was also found that the value of u_m decreases markedly with increasing temperature, as is common in systems of this type (Iglesias *et al.*, 1975). Although the exact form of the variation cannot be determined precisely, it was found that for the range of temperatures analysed, u_m may be approximately represented by the relation:

$$u_m = a+bT, \tag{6}$$

where a and b are constants. The same was calculated by linear regression from the plot of the u_m values given in Table 1 against T. The values of a and b calculated for the different products are given in Table 3.

Table 3. The values of the constants a and b of Eqn (6) for the different food materials

Material	a	b
Thyme	25.8268	−0.06925
Aniseed	8.9432	−0.0145
Marjoram	33.6709	−0.095
Fennel	14.3313	−0.03625
Wheat kernel	28.8803	−0.07088
Sugar beet root	15.9662	−0.03535

The heat of desorption corresponding to a given amount of adsorbed water can now be calculated from the Clausius–Clapeyron equation:

$$\left(\frac{\mathrm{d}\ln a_w}{\mathrm{d}(1/T)}\right)_u = \frac{Q_{st}}{R}, \tag{7}$$

where Q_{st} is the isosteric heat. The left term of Eqn (7) was calculated numerically through Eqns (1), (4), (5) and (6), approximating the derivative by a finite difference (the same was evaluated within the linear range of the BET equation, $a_w = 0.3$). The results of this procedure for the different materials are given in Figs 1–3, which show the variation of the net heat of desorption with moisture content.

For the purpose of comparison, the isosteric heat curves were also determined in the usual manner by application of the Clausius–Clapeyron equation to the equilibrium data obtained from the literature. It can be observed from the above mentioned figures, that the procedure developed in this investigation gives, in general, a reasonable modelling of the heat of desorption variation with moisture content. These results permit to conclude that a better estimation of the isosteric heat curve, in comparison with the relation derived by Berezin & Kisselev (1966), within the framework of the BET theory can be attained including the dependence of C and u_m on temperature in

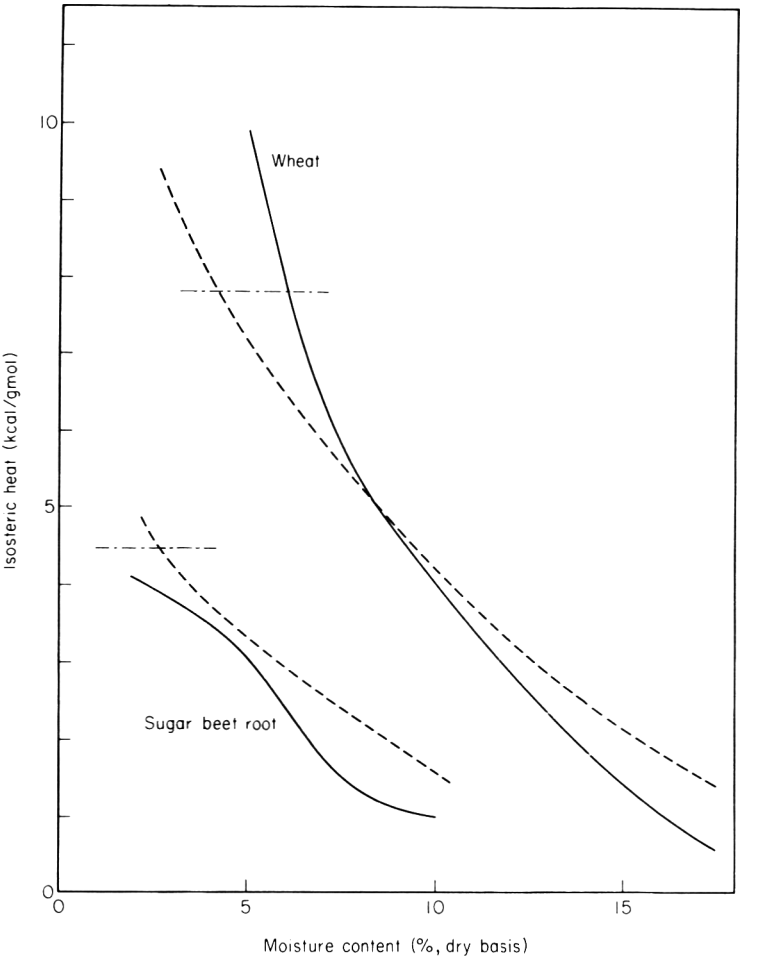

Figure 1. Isosteric heat variation with moisture content in sugar beet root and wheat (——— Calculated from Eqn (7). – – – – – from Eqns (1 and 4–7). and —·—·— $E_1 - E_l$).

the calculation of the heat of desorption. The marked variation of these parameters with temperature generally observed during the sorption of water in food products seems to justify the methodology adopted in this paper.

It is interesting to note that the $E_1 - E_L$ values calculated by means of Eqn (1) can be taken as an estimation of the isosteric heat at low moisture contents. Although the comparison between both forms of heats is rather difficult, given the strong dependence of the isosteric heat on the moisture content generally observed in this kind of products, the same was widely used in literature as a way of determining the limitations of the BET model (Volman *et al.*, 1960; Masuzawa & Sterling, 1968; Iglesias & Chirife, 1976). It can be observed in Figs 1–3 that the BET heats calculated from Eqn (1) can be taken as an estimation of the isosteric heats within the range of the monolayer values. Based on these results, it can be concluded that the BET heats derived from Eqn (1) instead of those calculated by means of the simplified expression, seem to be more representative of a given desorption process, particularly for that kind of products which shows a marked dependence of the heat of sorption on moisture content.

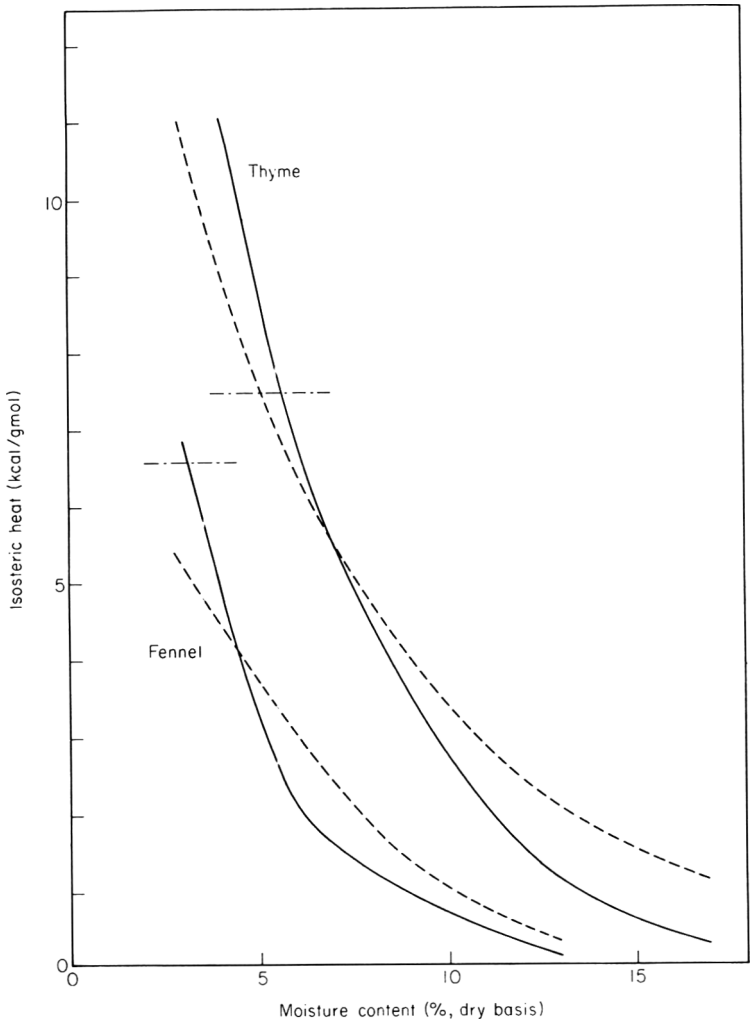

Figure 2. Isosteric heat variation with moisture content in fennel and thyme (for key, see legend to Fig. 1).

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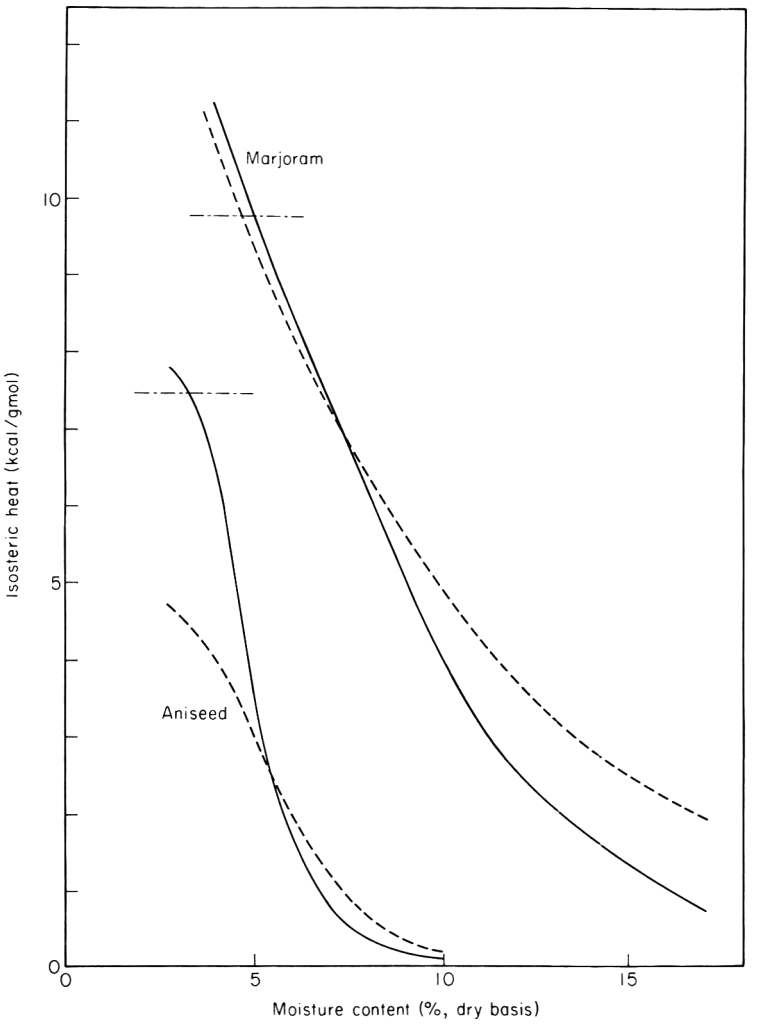

Figure 3. Isosteric heat variation with moisture content in marjoram and aniseed (for key, see legend to Fig. 1).

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Quality changes of Nigerian traditionally processed freshwater fish species. I. Nutritive and organoleptic changes

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Summary

Traditionally smoked (TS), traditionally solar dried (TD), oven dried (OD), Ife solar dried (ISD), and fresh (FF) *Clarias lazera, Sarotherodon niloticus, Sarotherodon galileus, Tilapia zilli,* and *Hemichromis fasciatus* were assessed for proximate composition, nutritive value and organoleptic changes.

Proximate composition of the fish treatment groups were similar, and this increased with decreasing moisture content.

Biological value (BV), true digestibility (TD), net protein utilization (NPU), protein efficiency ratio (PER), were greater than casein ($P < 0.01$), although they are not statistically different from each other.

From consumer-type taste panel, no statistically significant differences between fish species and organoleptic attributes scores could be discerned. However, a comparison between fish treatment and organoleptic attributes showed obvious differences. The fresh fish always scored highest while TD scored lowest.

The findings are discussed.

Introduction

Freshwater fish sources constitute 69.6% (FOS, 1980) of the total fish supply available to Nigeria. This represents a major source of animal protein supply in Nigeria, which has a low per capita protein consumption. Since the purchase and maintenance of freezing and/or chilling equipment is beyond the means of most small fishermen, the unsold catch is preserved by traditional solar drying (TD) or smoking (TS). It is estimated by the authors that 40–45% of freshwater fish (FF) available is processed this way. The disadvantage of this method is high insect infestation or windborne sand, in solar dried fish, and deposition of potentially toxic contaminants generated during smoking (Lo & Sandi, 1978).

Despite the high consumption of fish processed in this manner (3 kg per capita in 1980 and 9.9–11.1 kg per capita projected for 1985; Ladipo, Fabiyi & Fatunla, 1981), very little is known about the quality changes which occur during processing. The aim of this study is to evaluate nutritional and organoleptic changes which occur during traditional smoking or solar drying of *Sarotherodon niloticus, Sarotherodon galileus, Tilapia zillii, Clarias lazera,* and *Hemichromis fasciatus* which together constitute about

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40% of Nigerian freshwater fish supply. These changes were compared with fish processed using a Fisher drying oven and a University of Ife-deigned and -built solar dryer (A. M. A. Salau, unpublished). This paper is also one of the series of work at Ife which attempts to provide much needed information on the effect of traditional processing techniques in Africa where smoked or solar dried fish are an important part of the staple diet (Hoffman *et al.*, 1977).

Materials and methods

Sample treatment

Fish used in the traditional preservation portion of this study were caught in the wild in various locations in Nigeria, ungutted, soaked in saturated brine for 3 min, and smoked (TS) or solar dried (TD) on site and were transported to Ife. Oven dried (OD) or Ife solar dried (ISD) fish were collected from fresh waters in Oyo State of Nigeria, soaked in saturated brine for 3 min and dried immediately, using a drying oven or the Ife solar dryer. Samples from fresh fish (FF) were obtained from freshly killed fish which were transported live from various locations in Nigeria to Ife.

Traditional smoking techniques vary from ethnic group to ethnic group in Africa. The most popular method, which is placing the fish on wire gauze in a wood fired kiln made from a steel drum, was used in this study. Smoking time used was 2 hr (an average time used by local fishermen) and carcass temperature recorded for the fish at the end of smoking was 110–120°C. Traditional solar drying time was 4–6 days in the open air as practiced by Nigerian fishermen. This resulted in carcass temperatures of 50–60°C. Oven dried fish were subjected to 70°C for 48 hr. Fish prepared in the Ife solar dryer were dried for 36 hr (6 hr/day at temperatures of 60–90°C). Fish of each species and treatment were placed together, ground finely and stored at −30°C for no longer than 2 weeks before use in this study.

Proximate analysis

Moisture content in ten 1 g samples from each treatment group and 4 g from fresh fish was determined according to AOAC (1980). Similarly, oil content in ten 5 g samples from each treatment group and 10 g from fresh fish was determined by the method of Bligh & Dyer (1959). Crude nitrogen contents were determined by the Kjedahl procedure as described by Eastoe & Courts (1963).

Nutritive evaluation

The nutritive evaluation was determined by animal assay technique using weanling litter mate rats of the Wistar strain from our animal colony. The technique used was the same as that described previously (Afolabi & Oke, 1981) in which treated and fresh fish were used as the main sources of protein. All fish samples were dried in the oven at 70°C for 24 hr before use in the feeding trials.

The net protein utilization (NPU) was determined by the carcass nitrogen technique of Miller & Bender (1955). The true digestibility (TD) of the dietary nitrogen was obtained in the same experiment by the 'balance sheet' method of Mitchell (1923—24). The biological value was calculated by dividing NPU by TD (Bender & Haizeldon, 1957). Protein efficiency ratio (PER) was determined as described by Rippon (1959).

The rats were collected at 23–24 days of age, numbered and housed individually in wire-screen-bottom cages. They were weaned to the stock diet in the experimental

cages for a week so that on commencement of feeding trials the rats were always 30–31 days old and weighed 50–60 g.

Organoleptic assessment by taste panel

Organoleptic scores for taste, texture, flavour, appearance and overall acceptability were evaluated at each tasting session by different panels of 400 consisting of persons, from Ife University campus, not trained formally in sensory assessment. The panel attended in groups of ten; and five randomly coded samples were presented to each member for assessment. The assessors described their opinions of the samples using a 5-point hedonic scale as previously described (Afolabi *et al* , 1981) (Table 1). Since it cannot be assumed that the hedonic scale is linear or that the data are distributed normally, the median was used as a measure of central tendency of the scores for each sample. The median was calculated as a point value assuming a continuous distribution.

Treated samples were warmed at 37°C in the oven for 1 hr before serving at each tasting session. Fresh fish used for sensory assessment were always freshly killed, boiled and salt added to taste.

Table 1. Sample taste panel score sheet

In this experiment we are interested in assessing the acceptability of a number of fish samples.

Would you please taste each sample and give your assessment of *taste*, *flavour*, *texture*, *appearance* and *overall acceptability* using the scale below:

Like much	=5
Like	=4
Neither like nor dislike	=3
Dislike	=2
Dislike much	=1

You may if you wish also add any comments you think appropriate.

	Attributes' score					
Sample code	Taste	Flavour	Texture	Appearance	Overall acceptability	Comments

Signature..... Date .....

Statistical analysis of results

Nutritive evaluation and proximate composition data were subjected to analysis of variance. The medians of hedonic score of organoleptic attributes were subjected to a median test (Hall & Nie, 1981).

Results

Proximate composition

The proximate composition of fresh, traditionally smoked (TS), traditionally solar

Table 2. Proximate composition of fresh (FF), traditionally smoked (TS), traditionally solar dried (TD), oven dried (OD), Ife solar dried (ISD), freshwater fish species used for palatability studies

Fish Species	Treat-ment	Proximate composition (%)				
		Moisture	Crude protein	Total lipid	Total ash	Fibre (modified acid detergent)
C. lazera	FF	78.13 (2.22)	18.63 (6.70)	4.22 (1.38)	0.71 (0.19)	0.13 (0.04)
	TS	59.47 (9.12)	27.74 (8.81)	6.15 (1.36)	1.03 (0.13)	1.20 (0.04)
	TD	54.78 (9.91)	25.36 (10.73)	6.57 (1.23)	1.49 (0.31)	0.21 (0.15)
	OD	12.04 (1.26)	74.45 (29.48)	21.90 (0.83)	3.36 (0.27)	0.67 (0.48)
	ISD	12.01 (2.25)	76.60 (21.19)	20.32 (1.63)	3.41 (0.18)	0.62 (0.21)
S. niloticus	FF	78.11 (5.78)	18.62 (6.11)	3.66 (0.67)	0.95 (0.13)	0.15 (0.02)
	TS	46.24 (6.40)	33.45 (6.54)	5.34 (0.16)	1.46 (0.13)	0.28 (0.24)
	TD	44.21 (5.50)	33.89 (11.23)	5.78 (1.53)	1.77 (0.23)	0.27 (0.08)
	OD	11.11 (1.99)	78.89 (27.56)	18.57 (0.95)	3.61 (0.31)	1.05 (0.89)
	ISD	10.84 (3.21)	78.19 (26.40)	19.54 (0.93)	3.70 (0.46)	1.02 (0.18)
S. galilaus	FF	78.11 (0.04)	18.03 (6.60)	3.60 (1.65)	0.86 (0.12)	0.08 (0.01)
	TS	51.72 (6.30)	32.72 (1.95)	4.82 (1.49)	1.19 (0.20)	0.12 (0.12)
	TD	45.21 (5.21)	30.15 (14.72)	5.86 (1.29)	1.23 (0.26)	0.13 (0.17)
	OD	10.26 (1.20)	77.26 (27.45)	19.88 (1.86)	3.70 (0.24)	0.18 (0.13)
	ISD	7.25 (2.1)	78.49 (38.78)	14.38 (1.37)	4.05 (0.59)	0.98 (0.50)
T. zilli	FF	77.56 (0.56)	18.88 (6.80)	2.75 (1.27)	0.95 (0.17)	0.10 (0.04)
	TS	48.31 (2.49)	30.94 (7.10)	4.48 (1.27)	1.35 (0.18)	0.26 (0.14)
	TD	45.68 (5.62)	37.52 (2.34)	4.84 (1.54)	1.81 (0.84)	0.37 (0.12)
	OD	10.87 (2.90)	76.71 (34.19)	19.62 (1.65)	3.78 (0.28)	0.71 (0.14)
	ISD	11.63 (2.90)	78.25 (27.38)	19.34 (1.76)	3.33 (0.10)	0.88 (0.61)
H. fasciatus	FF	74.37 (0.82)	18.41 (6.12)	0.25 (0.10)	0.82 (0.61)	0.03 (0.04)
	TS	33.41 (1.55)	47.60 (12.56)	0.61 (0.14)	0.83 (0.26)	0.07 (0.04)
	TD	30.01 (6.41)	51.17 (14.11)	0.53 (0.33)	1.07 (0.14)	0.07 (0.05)
	OD	4.48 (0.56)	79.34 (5.10)	3.68 (0.34)	12.32 (0.37)	0.61 (0.17)
	ISD	4.40 (1.20)	78.17 (6.10)	4.25 (0.91)	13.28 (1.34)	0.86 (0.14)

Values are means of ten determinations. Values in parentheses are standard deviations of mean.

dried (TD), oven dried (OD) and Ife solar dried (ISD) freshwater fish (FF) species used for palatability trials in the study is shown in Table 2. The data in Table 2 show that the different fish species, *C. lazera, S. niloticus, S. Galilaeus, T. zillii,* and *H. fasciatus* are of similar composition in terms of standard food chemistry and in close agreement with results obtained by previous workers, Hoffman *et al.* (1977). It can be observed that crude protein, total lipid, total ash, and crude fibre contents of the fish samples increase with decreasing moisture content.

Animal feeding trials

The results of the rat feeding trials are shown in Table 3. The data obtained show that all fish samples used in the feeding trials are of high nutritive value. All samples have superior biological value (BV), true digestibility (TD), net protein utilization (NPU) and protein efficiency ratio (PER) to casein ($P < 0.01$), although they are not significantly different from each other.

Table 3. Nutritive value of fresh (FF), traditionally smoked (TS), traditionally solar dried (TD), over dried (OD), Ife solar dried (ISD) freshwater fish species used for palatability studies.

Protein source	Treatment	BV	TD	NPU	PER
Casein		56.10 (0.2)	98.71 (2.0)	55.73	1.63 (0.1)
C. lazera	FF	93.87 (8.0)	98.06 (2.2)	92.04	2.9 (0.8)
	TS	72.94 (7.3)	94.15 (1.1)	68.67	2.5 (0.8)
	TD	77.46 (3.6)	94.30 (4.4)	73.04	2.2 (0.3)
	OD	93.61 (4.5)	96.35 (1.0)	90.19	3.3 (0.9)
	ISD	87.99 (1.1)	95.45 (4.1)	83.99	2.2 (0.9)
S. niloticus	FF	98.24 (4.1)	96.60 (8.3)	94.89	2.5 (0.1)
	TS	82.43 (1.8)	88.93 (6.4)	73.30	2.4 (0.6)
	TD	76.51 (8.2)	90.52 (6.0)	69.26	2.4 (0.5)
	OD	94.69 (2.1)	96.80 (1.2)	91.66	2.8 (0.6)
	ISD	86.97 (4.2)	92.20 (1.6)	80.19	2.4 (0.8)
S. galileus	FF	97.86 (4.7)	98.11 (8.6)	96.01	2.2 (1.1)
	TS	80.31 (9.0)	91.56 (6.9)	73.53	2.5 (0.9)
	TD	78.41 (1.3)	87.63 (3.1)	68.71	2.2 (0.6)
	OD	93.62 (3.6)	98.88 (3.5)	92.57	2.7 (0.5)
	ISD	91.69 (2.5)	90.03 (1.7)	82.55	2.1 (0.8)
T. zilli	FF	85.72 (1.2)	96.64 (9.1)	82.84	2.7 (0.5)
	TS	81.35 (3.4)	92.41 (1.5)	75.18	2.6 (0.7)
	TD	72.63 (1.2)	91.16 (1.5)	66.21	2.1 (0.6)
	OD	95.68 (2.4)	95.23 (3.1)	91.11	2.8 (0.4)
	ISD	91.45 (1.7)	94.34 (2.3)	86.27	2.6 (0.9)
H. fasciatus	FF	98.72 (1.9)	95.10 (5.2)	93.88	2.6 (0.8)
	TS	80.78 (2.2)	90.92 (2.0)	73.44	2.3 (0.5)
	TD	80.59 (1.6)	88.72 (1.3)	71.49	2.3 (0.5)
	OD	95.82 (3.3)	91.33 (1.2)	87.51	2.8 (0.1)
	ISD	96.21 (7.1)	91.67 (4.0)	88.91	2.4 (0.4)

Values are means of eight replicates. Values in parentheses are standard deviation of means.

Organoleptic assessment

Table 4 summarizes the taste panel results. From the consumer type taste panel no statistically significant differences between fish species and organoleptic attributes scored could be discerned (Tables 5 and 6). However, a comparison between fish treatment and organoleptic attributes showed obvious diffences.

Table 4. Medians of hedonic scores of taste, flavour, texture, appearance and overall acceptability of fresh (FF), traditionally smoked (TS), traditionally solar dried (TD), oven dried (OD), Ife solar dried (ISD) freshwater fish species.

Fish species	Treat-ment	Organoleptic attributes scored				
		Taste	Flavour	Texture	Appearance	Acceptability
C. lazera	FF	4.95	4.81	4.98	4.93	5.00
	TS	4.23	4.98	4.27	4.96	4.45
	TD	3.18	2.33	3.24	2.62	1.62
	OD	4.29	3.43	3.66	4.35	4.17
	ISD	4.11	3.12	3.87	3.70	3.83
S. niloticus	FF	4.92	4.98	4.95	4.87	4.95
	TS	4.49	3.82	3.72	4.64	4.63
	TD	2.58	1.73	3.47	3.83	1.29
	OD	3.98	3.34	4.15	3.71	4.13
	ISD	4.28	3.35	3.64	3.52	3.96
S. galilaeus	FF	4.89	4.83	4.96	4.88	4.92
	TS	4.04	4.10	3.62	4.38	4.31
	TD	1.87	1.54	2.34	1.43	1.81
	OD	3.81	3.49	4.00	4.71	4.11
	ISD	3.83	3.51	3.14	3.35	4.38
T. zilli	FF	4.98	4.87	4.94	4.87	4.96
	TS	4.97	4.32	4.46	4.50	4.72
	TD	2.04	1.73	2.28	1.61	1.48
	OD	3.85	3.59	4.37	4.16	4.44
	ISD	3.69	3.21	3.95	3.47	4.08
H. fasciatus	FF	4.85	3.99	4.63	3.16	3.45
	TS	3.96	3.92	3.61	2.34	3.43
	TD	2.51	3.03	2.23	3.50	2.29
	OD	3.79	3.75	3.36	3.77	3.32
	ISD	3.56	3.15	3.46	3.91	3.48

Table 5. Median test of hedonic scores for taste, flavour, texture, appearance and overall acceptability against fish species* (data from Table 4)

Fish species	Taste‡	Flavour‡	Texture‡	Appearance‡	Overall acceptability‡
C. lazera	5	2	3	3	3
S. niloticus	4	3	3	2	3
S. galilaeus	2	2	2	3	3
T. zillii	3	3	4	3	3
H. fasciatus	3	3	1	1	0
Median	4	4	4	4	4

* Values are No. of cases greater than the median.

‡ NS (not statistically significant).

Table 6. Median test of hedonic scores* for taste, flavour, texture, appearance, overall acceptability and fish species against treatment (data from Table 5)

Treatment	Taste†	Flavour†	Texture‡	Appearance‡	Overall acceptability‡	Fish species§
FF	5	5	5	4	4	2
TS	4	5	2	4	4	3
TD	0	0	0	0	0	2
OD	1	2	3	3	3	2
ISD	2	0	2	1	1	2
Median	4	4	4	4	4	3

* Values are No. of cases greater than the median.
Level of significance: †$P < 0.01$; ‡$P < 0.05$; §NS.

They rated the fish samples as a whole lower than FF. A median test of data in Table 4 shows FF always having the greatest number of cases greater than the median and TD the lowest for taste ($P < 0.01$), flavour ($P < 0.01$), texture ($P < 0.05$), appearance ($P < 0.05$) and overall acceptability ($P < 0.05$). The trend of scores for each of the attributes considered was:

Taste: FF > TS > ISD > OD > TD,
Flavour: FF = TS > OD > ISD = TD,
Texture: FF > OD > TS = ISD > TD,
Appearance: FF > TS > OD > ISD > TD,
Overall acceptability: FF = TS > OD > ISD > TD.

Discussion and conclusion

Results obtained for proximate analysis and feeding trial experiment suggest that the nutritive value of fish samples used were high. However, the animal assay technique could not discriminate between the different fish treatment and species. They have very similar nutritive values in this study.

The overall acceptability scores obtained using an untrained taste panel would closely relate to the overall preference of the consumer for the samples. An inspection of remarks made by individual members of the panel showed that smoked fish was the most preferred alternative to fresh fish in Nigeria. They preferred TS in all the attributes considered to any other treatment group. The species of fish does not seem to be related to panel scores. The seemingly poorer scores for *H. fasciatus* could be attributed to the very small size of the fish which made it unimpressive to the panellist.

H. fasciatus was included in this study because it always constituted 30% of our catch, and about 99% of the catch in this country was always consumed smoked. It appears the traditionally solar dried fish was not acceptable to the sensory assessors because it had some off flavour, pebbles and sometimes dust. Although the most acceptable fish product next to FF is TS, it contains more potentially toxic substances than ISD or OD (Lo & Sandi, 1978; Afolabi, Adesulu & Oke, 1983). The least expensive alternative process available is the Ife solar dryer which provides an hygienic environment and may be constructed at village level technology. Smoke flavour, free of potential toxicants, could be cheaply introduced to improve its organoleptic attributes.

In conclusion, though the taste panel could not show statistically significant

differences for the individual attributes examined and fish species, obvious differences exist between fish treatment and organoleptic attributes. It appears, as would be expected, that there is no correlation between nutritive value and organoleptic assessment as animal assay evaluates the protein quality of food only. However, reasonable correlation between taste panel scores and chemical/physical parameters of processed fish is emerging from this laboratory and elsewhere (Afolabi *et al.*, 1981; Afolabi, Salau & Oke, unpublished; Tatterson, Pollit & Wignall, 1980; Connell & Shewan, 1980).

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Quality changes of Nigerian traditionally processed freshwater fish species. II. Chemical composition

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Summary

Chemical changes in traditionally smoked (TS), traditionally solar dried (TD), oven dried (OD), Ife solar dried (ISD), *Clarias lazera, Sarotherodon niloticus, Sarotherodon galilaeus, Tilapia zillii* and *Hemichromis fasciatus* were determined.

Amino acid composition of fish treatment groups were similar. There was a decrease in percent available lysine in the order OD ≈ ISD > TS > TD. No systematic variation in fatty acid composition was observed.

Analysis of nucleotides and nucleotide related products revealed no systematic variation in the individual nucleotide content. However a pooled percent of the total nucleotides shows that TD has the highest value of hypoxanthine (HX).

Introduction

As a method of food preservation, smoking and solar drying play a significant role in the fishing industry of developing nations, such as African countries. There is much literature concerning the quality in processed fish from temperate or cold countries of the world (Connell, 1980), but relatively little information about quality changes in African fish, especially the traditionally processed.

In a previous work the proximate composition, nutritive value and organoleptic changes of traditionally smoked or solar dried fish were assessed for *Clarias, Sarotherodon, Tilapia* and *Hemichromis* species. These changes were compared to fish processed using a drying oven and a University of Ife designed and built solar dryer (A. M. A. Salau, unpublished). The present study attempts to estimate the chemical changes that occur in the same batch of fish used in our previous studies (Afolabi, Arawomo & Oke, 1984).

Materials and methods

Sample treatment

Fish samples were the same batch used in our previous work (Afolabi *et al.*, 1984).

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Amino acid analysis

Total amino acid analysis was carried out on the samples after acid hydrolysis by refluxing for 24 hr under nitrogen using an LKB amino acid analyser as previously described (Afolabi & Oke, 1981). Available lysine was determined by the method of Booth (1971).

Fatty acid analysis

Oil extraction was by the method of Bligh & Dyer (1959). Analysis of fatty acids was carried out by trans-esterification using BF_3/methanol followed by gas chromatography using a Hewellet Packard 5880 gas chromatograph (FID) on a 25×0.2 mm SP 1000 flexible fused silica column, coupled to a splitless injection mode autosampler (1.2 μl per injection). The column temperature was between 140 and 250°C at 3°C/min. Carrier gas was helium at a flow rate of 0.75 ml/min.

Nucleotide and nucleotide related product analysis

The method used for analysis was essentially that of J. Murray (private communication) using anion exchange high performance liquid chromatography. Reference nucleotides, nucleosides and bases were obtained from Sigma Chemical Co. A Waters model 6000 pump coupled to a variable volume U6K valve injector was used for delivery of eluate buffer to the column. Detection of UV absorbing materials in the eulate was made using a Cecil 212 Spectrometer fitted with a 10 μl flow cell. Column 25 cm Partisil SAC 10 μm, 260 nm, 0.1 AUFS, 4 μl aliquots used for injecion, eluate 0.04 M KH_2PO_4 containing 0.00187 M H_3PO_4 at 2 ml/min. Recorder chart speed 1 cm/min, 10 nV FSD. Twenty gram of fish was homogenized with 40 ml ice-cold 0.6 M perchloric acid for 1 min in an MSE homogeniser. After Buchner filtration into an ice-chilled flask, 20 ml of the filtrate was adjusted to pH 7.04 with 5 M KOH. Extracts were held in an ice bath for 30 min and portions of the supernatant were removed from the precipitated $KClO_4$ at −30°C prior to analysis.

Results

Amino acid analysis

Table 1 shows the amino acid composition of traditionally smoked (TS), traditionally solar dried (TD), oven dried (OD), Ife solar dried (ISD), *Clarias lazera, S. niloticus, S. galilaeus, T. zillii, H. fasciatus*. The amino acid patterns of all the processed fish species are similar. It appears there is a general loss of amino acids with processing. In all methods of preservation used, OD had a higher amino acid content, although not statistically significant.

The lysine content of different treatment groups are presented in Table 2. The results show that all the fish species have a high lysine content compared to whole hen's egg and other animal protein sources (Afolabi & Oke, 1981; Mitchell & Block, 1946).

Table 1. Amino acid composition of traditionally smoked (TS), traditionally solar dried (TD), oven dried (OD), Ife solar dried (ISD), freshwater water fish species.

Amino acids' (g/100 g protein)	*C. lazera*				*S. niloticus*				*S. galilaeus*				*T. zilli*				*H. fasciatus*			
	TS	TD	OD	ISD	TS	TD	OD	ISD	TS	TD	OD	ISD	TS	TD	OD	ISD	TS	TD	OD	ISD
Asp.	10.94	10.73	12.59	11.11	9.46	9.79	13.04	11.57	10.16	11.81	12.29	11.07	11.3	10.1	12.42	11.07	11.72	11.41	11.04	11.28
Thr.	3.65	3.83	3.93	3.67	3.38	3.15	4.09	3.88	3.28	3.47	4.01	3.63	3.55	3.60	4.15	3.74	3.19	3.24	3.59	3.27
Ser.	3.37	3.49	3.81	3.25	3.08	3.41	3.67	3.64	3.12	3.33	3.42	3.12	3.70	3.10	3.59	3.24	3.17	3.14	3.29	3.17
Glu.	15.35	15.03	16.98	15.04	13.61	14.26	16.64	16.24	14.35	14.15	17.33	15.67	16.36	17.14	17.54	15.64	15.54	15.11	15.07	15.69
Pro.	2.99	2.68	3.80	2.94	3.14	4.45	16.81	4.10	3.43	3.91	3.73	3.29	3.17	3.46	3.56	3.48	3.03	2.91	2.91	2.92
Gly.	6.05	7.16	6.72	6.46	7.08	7.79	7.25	7.22	7.11	6.61	5.87	6.78	5.58	5.91	5.56	6.56	6.48	6.51	6.32	6.14
Ala.	5.85	6.23	6.74	5.65	5.75	6.65	7.41	6.81	5.61	6.22	6.76	6.22	6.53	6.67	6.54	6.08	5.54	5.88	6.02	5.71
Val.	4.34	4.29	4.71	4.42	4.02	2.82	3.40	4.82	4.18	4.19	5.55	5.04	5.00	5.10	5.81	5.21	4.26	4.45	4.66	4.53
Cys.	0.67	0.28	0.29	0.27	0.53	0.43	0.44	0.53	0.52	0.53	0.52	0.49	0.50	0.58	0.63	0.59	0.59	0.56	0.69	0.56
Met.	3.15	2.79	3.79	3.15	2.65	2.74	3.06	3.25	3.02	3.18	3.56	3.14	3.10	3.26	3.71	3.35	3.10	3.19	3.35	3.19
Ile.	4.43	4.22	4.49	4.52	4.63	2.59	3.49	4.38	4.57	4.13	5.54	5.11	5.82	5.26	5.56	5.06	4.68	4.63	4.75	4.73
Leu.	7.68	7.55	8.32	7.54	6.61	6.16	7.97	7.74	8.28	8.45	8.59	7.81	8.46	8.11	8.67	7.86	7.82	7.90	7.77	7.61
Tyr.	3.14	2.91	3.28	2.90	2.71	2.49	2.86	3.31	2.88	2.71	3.42	3.09	3.02	3.19	3.50	3.17	3.09	3.11	3.12	2.95
Phe.	4.22	3.85	4.29	3.97	3.68	3.41	4.18	4.35	4.60	4.61	4.64	3.86	3.44	3.01	4.67	3.76	3.37	3.97	4.29	3.89
Lys.	13.24	12.80	13.57	13.14	10.39	10.14	13.19	12.65	11.27	12.57	14.82	12.76	11.37	11.96	14.93	13.68	11.31	11.50	13.51	12.29
His.	2.88	3.43	3.87	3.55	3.11	2.97	3.59	3.86	3.11	3.63	4.06	3.77	3.11	3.24	4.38	3.86	3.21	3.22	3.29	3.22
Arg.	4.87	5.23	5.81	5.03	5.03	5.04	5.19	5.99	5.10	5.49	6.03	5.14	5.36	5.01	5.92	5.48	5.61	5.05	5.32	5.52

Values are arithmetic means of duplicate determinations.

Table 2. Available lysine in traditionally smoked (TS), traditionally solar dried (TD), Oven dried (OD), Ife solar dried (ISD), freshwater fish species

Fish species	Treatment	Total lysine (g/100 g protein)	Available lysine (g/100 g protein)	% available
C. lazera	TS	13.24	10.50	79.3
	TD	12.80	9.33	72.9
	OD	13.57	12.86	94.8
	ISD	13.14	11.37	86.5
S. niloticus	TS	10.39	7.27	70.0
	TD	10.14	7.72	76.1
	OD	13.19	12.32	93.4
	ISD	12.65	10.66	84.3
S. galileus	TS	11.27	8.27	73.4
	TD	12.57	9.38	74.6
	OD	14.82	14.09	95.1
	ISD	12.76	11.24	88.1
T. zillii	TS	11.37	8.25	74.9
	TD	11.96	8.29	69.3
	OD	14.93	14.26	95.5
	ISD	13.68	12.09	88.4
H. fasciatus	TS	11.31	8.43	74.5
	TD	11.50	9.02	78.4
	OD	13.51	12.94	95.8
	ISD	12.29	10.97	89.3

Values are arithmetic means of four determinations.

However, there is a decrease in per cent available lysine with processing. In general, the order of available lysine is OD $\simeq$ ISD > TS > TD ($P < 0.05$).

Fatty acid analysis

Table 3 shows the comparison of the fatty acid composition of the various fish groups. There appears to be no systematic variation in the fatty acid composition with fish species or treatment groups. There are, however, higher proportions of unsaturated fatty acids in OD and ISD than in TS or TD, although not statistically significant.

Nucleotides and nucleotide related products analysis

The results of the nucleotide and nucleotide related product analysis are presented in Table 4. No systematic variation in the individual nucleotide content was observed. A pooled percentages of the total nucleotides (Table 5), however, shows the following relationship for:

(a) ATP+ADP: TS > OD > ISD > TD;
(b) AMP: OD > ISD > TD > TS;
(c) IMP: TS > OD > TD > ISD;
(d) INO: TS > OD > TD > ISD;
(e) Hx: TD > ISD > OD > TS ($P < 0.05$).

(See Tables 4 and 5 for abbreviations.)

Table 3. Percentage fatty acid composition of traditionally smoked (TS), traditionally solar dried (TD), oven dried (OD), Ife solar dried (ISD), freshwater fish species

Fatty acid	*C. lazera*				*S. niloticus*				*S. galileus*				*T. zilli*				*H. fasciatus*			
	TS	TD	OD	ISD	TS	TD	OD	ISD	TS	TD	OD	ISD	TS	TD	OD	ISD	TS	TD	OD	ISD
C14:0	2.7	2.7	2.9	2.9	4.2	2.4	6.5	13.7	2.1	2.6	6.2	6.7	4.1	2.2	2.7	2.8	2.8	4.3	2.1	2.3
C15:0	2.3	2.2	3.9	2.7	4.7	4.9	7.9	3.6	2.9	3.7	7.4	4.4	2.2	2.2	1.4	2.3	2.9	2.4	1.4	2.5
C16:0	22.5	22.8	24.4	23.1	21.4	26.5	23.0	28.8	22.2	22.1	23.3	19.9	23.8	22.5	19.6	22.8	22.8	23.5	21.7	23.3
C16:1	6.0	6.0	6.0	6.9	5.3	6.8	7.2	10.6	5.9	6.1	7.5	6.4	5.7	9.0	4.5	5.2	3.2	4.6	3.9	4.2
C17:0	2.7	2.5	2.4	2.8	2.1	1.3	2.5	1.6	2.9	2.1	2.8	1.7	1.9	2.1	2.0	2.3	1.3	1.9	2.0	1.5
C18:0	8.4	8.4	8.2	7.9	7.9	6.5	5.0	5.7	6.1	7.1	5.8	7.5	6.2	7.5	8.3	7.6	8.1	7.5	9.8	7.1
C18:1	15.9	16.0	15.7	15.7	13.6	8.1	8.3	8.9	8.2	8.2	8.2	8.5	4.9	11.8	16.5	16.3	16.3	16.1	16.2	16.1
C18:2	5.0	3.8	5.4	5.1	3.7	3.6	4.2	2.9	3.0	2.6	4.4	2.9	6.0	6.5	7.5	6.7	6.7	6.8	6.4	7.5
C18:3	3.8	4.3	4.0	4.1	1.9	1.3	4.1	3.3	1.3	1.2	4.0	1.7	2.0	2.0	4.5	3.1	3.1	3.8	4.2	3.5
C18:4	0.6	0.6	0.5	0.7	1.1	1.1	1.8	1.3	1.3	1.1	1.6	1.3	1.2	0.9	0.8	1.1	0.5	0.9	0.8	0.8
C20:4	3.7	3.9	3.9	3.7	5.3	2.0	2.0	1.5	2.1	2.6	2.8	4.8	3.8	3.8	3.9	3.8	3.5	4.2	5.7	5.9
C20:5	tr	3.6	3.2	3.2	tr	0.3	0.4	0.5	tr	0.7	0.7	0.5	tr	0.6	0.7	0.7	0.3	0.7	0.9	0.7
C22:5	1.0	0.9	0.8	0.8	1.9	1.6	0.9	1.7	2.0	2.8	2.5	2.4	1.7	1.8	1.8	1.9	2.4	2.3	2.6	2.8
C22:6	4.0	3.8	3.9	3.8	4.0	3.7	3.1	2.5	3.2	3.5	3.8	3.8	3.8	2.6	2.9	3.6	4.5	4.5	6.1	4.8
Minor components	21.4	18.5	14.5	16.6	22.9	29.9	23.1	13.4	36.8	33.6	19.0	27.5	22.7	24.5	22.9	19.8	21.6	16.5	16.2	17.0
% saturates	38.6	38.6	41.8	39.4	40.3	41.6	44.9	53.4	36.2	37.6	45.5	40.2	38.2	36.5	34.0	37.8	37.9	39.6	37.0	36.7
% mono unsaturates	21.9	22.0	21.7	22.6	18.9	14.9	15.5	19.5	14.1	14.3	15.7	14.9	20.6	20.8	21.0	21.5	19.5	20.7	20.1	20.3
% poly-unsaturates	18.1	20.9	21.7	21.4	17.9	13.6	16.5	13.7	12.9	14.5	19.8	17.4	18.5	18.2	22.1	20.9	21.0	23.2	26.7	26.0
Total % unsaturates	40.0	42.9	43.4	44.0	36.8	28.5	32.0	33.2	27.0	28.8	35.5	32.3	39.1	39.0	43.1	42.4	40.5	43.9	46.8	46.3

Values are means of duplicate determinations.

Table 4. Nucleotides and related products content of traditionally smoked (TS), traditionally solar dried (TD), oven dried (OD), Ife solar dried (ISD), freshwater fish species

		Nucleotides and related produces (μmol/g fish)					
Fish species	Treatment	ATP+ADP*	AMP*	IMP*	INO*	HX*	Total nucleotide
C. lazera	TS	0.25 (14.1)	0.06 (3.4)	0.37 (20.9)	0.70 (39.5)	0.39 (22.0)	1.77
	TD	0.09 (2.6)	0.21 (6.0)	0.80 (22.7)	1.49 (42.3)	0.93 (26.4)	3.52
	OD	0.46 (9.6)	1.97 (41.2)	0.67 (14.0)	1.28 (26.8)	0.40 (8.4)	4.78
	ISD	0.27 (14.9)	0.05 (2.8)	0.32 (17.7)	0.71 (39.2)	0.46 (25.4)	1.81
S. niloticus	TS	0.26 (18.8)	0.06 (4.3)	0.31 (22.5)	0.42 (30.4)	0.33 (23.9)	1.38
	TD	0.13 (4.9)	0.09 (3.4)	0.18 (6.7)	0.88 (32.8)	1.40 (52.2)	2.68
	OD	0.30 (14.2)	0.03 (1.4)	0.19 (9.0)	0.69 (32.7)	0.90 (42.7)	2.11
	ISD	0.21 (10.9)	0.04 (2.1)	0.14 (7.3)	0.60 (31.3)	0.93 (48.4)	1.92
S. galilaeus	TS	0.23 (15.3)	0.02 (1.3)	0.38 (25.3)	0.52 (34.7)	0.35 (23.3)	1.50
	TD	0.09 (4.9)	0.04 (2.2)	0.12 (6.5)	0.57 (30.8)	0.02 (55.6)	1.85
	OD	0.28 (13.4)	0.02 (1)	0.22 (10.5)	0.68 (32.5)	0.89 (42.6)	2.09
	ISD	0.14 (11.7)	0.02 (1.7)	0.06 (5.0)	0.39 (32.5)	0.59 (49.1)	1.20
T. zilli	TS	0.27 (15.6)	0.08 (4.6)	0.47 (27.1)	0.61 (35.3)	0.30 (17.3)	1.73
	TD	0.02 (1.9)	0.02 (1.9)	0.15 (14.4)	0.25 (24.0)	0.60 (57.7)	1.04
	OD	0.30 (10.8)	0.13 (4.7)	0.58 (20.9)	1.08 (39)	0.68 (24.5)	2.77
	ISD	0.13 (10.1)	0.08 (6.2)	0.12 (9.3)	0.31 (24.0)	0.65 (50.4)	1.29
H. fasciatus	TS	0.12 (10.3)	0.08 (6.9)	0.33 (28.5)	0.30 (25.9)	0.33 (28.4)	1.16
	TD	0.02 (1.9)	0.04 (3.7)	0.35 (32.4)	0.29 (26.9)	0.38 (35.2)	1.08
	OD	0.41 (11.2)	0.22 (6.0)	1.13 (30.9)	0.95 (26.0)	0.95 (26.0)	3.66
	ISD	0.11 (9.9)	0.07 (6.3)	0.35 (31.5)	0.30 (27.0)	0.28 (25.2)	1.11

Values are means of ten determinations. Values in parentheses are % of total nucleotides.

ATP, adenosine triphosphate; ADP, adenosine diphosphate; AMP, adenosine monophosphate; IMP, inosine monophosphate; INO, inosine; HX, hypoxanthine.

Table 5. Pooled mean percentages of nucleotides, nucleotide related product composition, and overall acceptability by a consumer type taste panel of traditionally smoked (TS), traditionally solar dried (TD), oven dried (OD) and Ife solar dried (ISD) freshwater fish species

	Pooled mean % nucleotides and related product					
Fish treatment	ATP+ADP	AMP	IMP	INO	HX	Overall acceptability*
TS	14.8	4.1	24.9	33.2	23.0	4
TD	3.2	3.4	16.5	31.4	45.4	0
OD	11.8	10.9	17.1	31.4	28.8	3
ISD	11.5	3.8	14.2	30.8	39.7	1

*Values are number of cases greater than the median (median = 4 and values are significant at $P < 0.05$. Data source: Afolabi *et al.*, 1984).

Discussion and conclusion

Drying or smoking of fish for preservation has been performed by man since the evolution of settlement in communities (Steinberg 1980) and still remains, today, the method of choice in traditional African society. The principal aim of fish processing is to preserve and/or enhance quality. However, these traditional process techniques have great problems of assuring the maintenance of the quality of the starting material, fresh fish, during and after processing. In this study the chemical changes of such traditionally preserved fish are examined.

Amino acid analysis

The results of the amino acid profiles (Table 1) of the various fish treatment groups suggest that there is no substantial loss during processing. Lysine is one of the major limiting amino acids in most protein sources and has a very reactive E-amino group which often reacts with reducing sugars (Maillard reaction) and other amino acids in the protein to yield a lysine complex which is no longer available for use by the body. The decrease in percent available lysine is clear evidence of damage to protein. This reaction may be accelerated by heat, moisture, storage and processing. However, other non-enzymic browning or oxidative type reactions may to a small extent be responsible for protein damage. The high loss in TS may be due to the high heat of processing (Labuza, 1973; Afolabi *et al.*, 1984) and the loss in TD largely due to biochemical and/or microbial deterioration during the relatively long drying time (4–6 days) (Shizunori, 1980). Comparing OD and ISD it appears drying at a sufficiently low and effective temperature of 70°C affected percent available lysine the least.

Fatty acid analysis

The fatty acid composition does not appear to change in any consistent direction with processing. However, the seemingly higher proportions of unsaturated fatty acids in OD and ISD suggest that they may go rancid faster in storage due to oxidation.

Nucleotides and nucleotide related products analysis

In the fish muscle, adenosine nucleotides occupy more than 90% of the total nucleotides and the main component is ATP (Shizunori, 1980). The principal course of ATP decomposition in fish *post mortem* is as follows (Tarr, 1966; Eskin, Henderson & Townsend, 1971):

$$\text{ATP} \rightarrow \text{ADP} \rightarrow \text{AMP} \rightarrow \text{IMP} \rightarrow \text{INO} \rightarrow \text{HX}.$$

In general the reactions ATP → IMP take place in a relatively early stage after death. In this period the reactions IMP → INO → HX occur more slowly than those in the sequence ATP → IMP. Accordingly IMP is readily accumulated in the fish meat in the early stage after death; INO and HX concentration increased with lapse of time or with decrease in freshness. A comparison of data obtained in this study and a previous one (Afolabi *et al.*, 1984; see Table 5) shows an increasing HX with a decreasing number of cases greater than the median of acceptability score (which included consideration for freshness) by a consumer type taste panel ($r = -0.9994$). Number of cases greater than the median was treated as a point value. The significantly higher percentage of IMP and lower HX in TS (Table 5) than in the other processed fish indicates that the smoked fish appear the freshest at the end of processing while TD is the least fresh.

In conclusion, although traditionally smoked fish could be argued to be the freshest

after processing because it contained the lowest percentage of HX, it is highly contaminated by potential toxicants such as polynuclear aromatic hydrocarbons (Afolabi, Adesulu & Oke, 1983). Traditional solar drying which is the cheapest method of preservation has low chemical. nutritional and organoleptic qualities as its disadvantage. Oven drying requires electric power supply and adequate equipment maintenance. Therefore, it is impractical in the villages where there is either no power supply or the cost of purchase is prohibitive to the local fisherman.

The nutritional. chemical and organoleptic qualities of ISD are comparable to OD. Sun is abundant in Africa and Ife solar dryer requires no electric power supply. It could be constructed with village level technology at the cost of N120.00 (US $200.00) each.

The practical implication of the findings in the present and previous (Afolabi *et al.*, 1983, 1984) studies is that the preservation of fish using the Ife solar (an Ife designed and built solar dryer) provides an alternative to smoking and traditional solar drying.

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Compositional analysis of liquid egg products using infrared transmission spectroscopy

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Summary

Calibration equations have been developed for the measurement of protein, total lipid and total solids content of liquid egg products using infrared transmission spetroscopy and the technique enables all these analyses to be carried out in under 20 min. Multiple correlation coefficients of 0.982, 0.988 and 0.997 and residual standard deviations of 0.37, 0.66 and 1.06% respectively were obtained for the infrared method compared with standard procedures. The results for total solids was not significantly different from that obtained by regression of total solids on combined protein and total lipid, all three determined by standard methods.

Introduction

A rapid method for the quantitative analysis of milk by infrared (IR) transmission spectroscopy was reported 20 years ago (Goulden, 1964) and has subsequently evolved into an officially recognized procedure (Horwitz, 1980). Much more recently, one of the commercial instruments utilized for IR milk analysis has been adapted for the analysis of meat products (Bjarnø, 1981, 1982; Steel & Bell, 1983). In view of the lack of rapid methods of analysis for liquid egg products, therefore, a study was carried out with the purpose of investigating the possible use of the instrument for that application.

Materials and methods

Samples

A total of thirty-five samples comprising liquid whole eggs, albumens, yolks and commercial blends were obtained from two different egg processors. The samples were analysed for protein (P), total lipid (L), and total solids (S) by official AOAC methods (Horwitz, 1980).

Infrared transmission spectroscopy

The instrument under study was a Super-Scan (N. Foss Electric A/S, Hillerød DK-3400, Denmark), and it was used in the following way. The flow system was bled to remove air bubbles and a zero check on each component was carried out prior to each set of measurements. Eleven g of sample was accurately weighed into the reaction vessel and the weight stored in the microprocessor interfaced to the balance. One hundred g 3% Decon-90 was added and the vessel placed in the reaction chamber, shaken for 2 min, removed and 1 ml antifoaming agent added with further shaking to ensure

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dispersion of the sample. The reaction vessel was heated to 50°C in a waterbath and placed in the instrument for the pre-programmed analysis cycle. Each sample was analysed in duplicate.

Statistical analysis

Multiple linear regression analysis was carried out for each of the sets of reference data on the four means of duplicate Super-Scan readings protein (P_s), fat (L_s), carbohydrate (C_s) and water (W_s). Outliers were eliminated on the basis of the Cook statistic (Cook, 1977) which combines information about residuals and leverage and hence identifies points which are both suspect and influential. Minimal adequate subsets (Aitkin, 1974) were then investigated in order to find equations involving the fewest number of Super-Scan terms which fit the reference data as well as those using all of them. Finally, for comparison, multiple linear regression of total solids on protein and total lipids by the AOAC method was carried out.

Results and discussion

Liquid egg products are usually traded on a total solids content specification and since in a commercial egg breaking operation there is often an imperfect separation of the yolk from the albumen, the total solids of these products and blends made from them will vary somewhat between batches. Therefore, in order to maintain consistent product quality a rapid control procedure for this constituent would be advantageous. The total solids content of liquid egg products essentially consists of protein plus total lipid (Egan, Kirk & Sawyer, 1981) and both of these have been successfully measured in milk and meat by IR transmission spectroscopy. This is achieved in the commercial instrument used by measuring the background corected absorption at 5730 nm for protein and 6500 nm for lipid. Additional measurements are available at 9500 nm for carbohydrate and 4800 nm for water. The instrument is pre-calibrated in the factory using protein and fat standards but requires the determination of small 'intercorrection' factors for each filter which are specific for the particular application under investigation. The precisions of the Super-Scan readings for protein and total lipid expressed as standard deviations of replicates were 0.25 and 0.35% respectively. These figures are identical to those reported for meat using the same instrument (Bjarnø, 1981).

The independent variable (Super-Scan data) correlation matrix for the samples under study is shown in Table 1. As all the Super-Scan readings have high inter-correlations they are not truly independent variables, although the correlation between the reference data for protein and lipid was also high ($r = 0.972$) so this does not mean that they are not independent readings. The use of too many terms in the calibration equation for each constituent under these circumstances, however, is not advisable

Table 1. Independent variable correlation matrix for Super-Scan readings on liquid egg products

	L_s	P_s	C_s	W_s
L_s	1.0000			
P_s	0.93855	1.0000		
C_s	0.93914	0.90515	1.0000	
W_s	−0.86967	−0.82980	−0.78610	1.0000

since no additional information is provided by the additional terms but long term error, on the other hand, may be introduced. The results obtained, therefore, are based on equations selected from minimal adequate subsets containing spectroscopically sensible terms. The relationships between protein, total lipid and total solids determined by standard methods and by the Super-Scan after removal of two outliers in each case are shown in Figs 1–3. The regression equation for lipid involves only the Super-Scan fat reading while those for protein and total solics involve both the protein and fat readings but in no case did the carbohydrate or water readings make a significant contribution to the measurement.

The results presented in Figs 1–3 should be interpreted with caution because although they are impressive on superficial inspection as a result of the high correlation coefficients, these are caused by the wide range of compositions of the samples under study. The standard deviation figures are not as good when considered within one

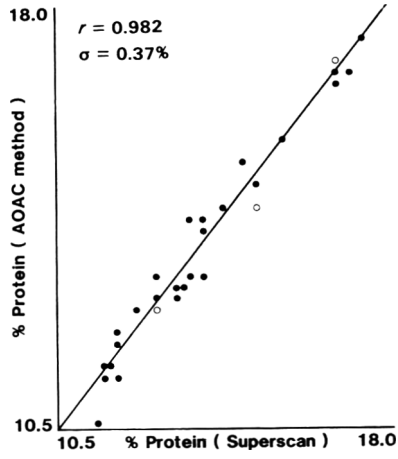

Figure 1. Plot of AOAC *versus* Super-Scan egg protein content using the regression equation $P = 5.64 + 0.115\ L_s + 0.498\ P_s$. ● = one observation; O = more than one coincident observation.

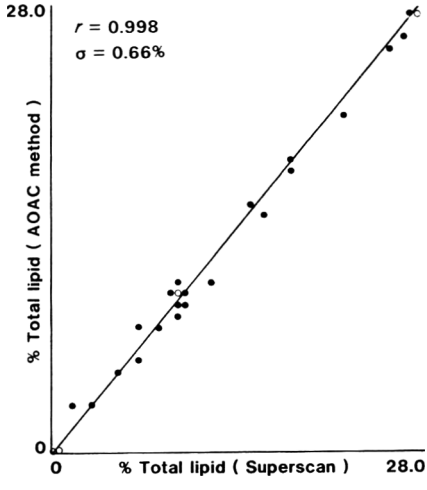

Figure 2. Plot of AOAC *versus* Super-Scan egg total lipid content using the regression equation $L = 0.63 + 0.975\ L_s$.

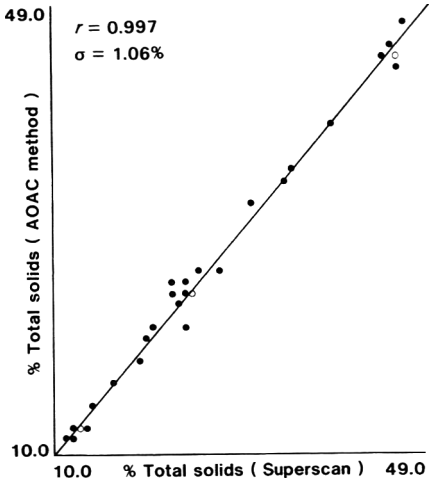

Figure 3. Plot of AOAC *versus* Super-Scan egg total solids content using the regression equation $S = 6.41 + 1.145\,L_s + 0.529\,P_s$.

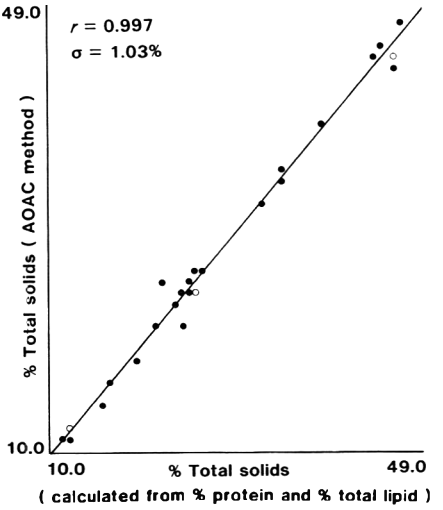

Figure 4. Plot of AOAC egg total tolids content *versus* egg total solids content predicted using the regression equation $S = 2.49 + 0.824\,P + 1.089\,L$.

product type instead of across the whole range. For example, the total solids equation has a standard deviation of 1.06% which is poor in relation to the range of values encountered for liquid whole egg only (24.5–26.0%). However, the result obtained using protein and total lipid determined by standard methods to predict total solids (Fig. 4) was almost identical. Therefore, the IR method is no worse than the standard methods for this purpose and should prove acceptable for commercial blends at least.

It was not possible with the number of samples studied to attempt separate calibrations for different product types—e.g. whole egg only—nor is it valid to speculate as to whether this might result in improved standard deviations.

Conclusion

In a limited feasibility study, an IR analyser designed for meat analysis has been successfully applied to the determination of protein and total lipid in liquid egg products.

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Synergistic killing of spores of *Bacillus subtilis* by peracetic acid and alcohol

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Summary

Synergistic sporicidal effects were observed when spores of *Bacillus subtilis* SA22 (NCA 72-52) were exposed to 0.08% (v/v) peracetic acid plus 9.9% (v/v) various primary alcohols. Synergistic sporicidal responses were also found when combinations or 0.04 or 0.08% (v/v) peracetic acid plus up to approximately 20% (v/v) ethanol were used. Higher concentrations of ethanol or peracetic acid did not yield further sporicidal action. Simultaneous use of ethanol and peracetic acid is a possible method of sterilization of aseptic packaging materials.

Introduction

Many aseptic packaging systems utilize high concentrations of hydrogen peroxide (25–35%) for sterilization of the packaging material, which is often applied at temperatures higher than ambient (von Bockelmann, 1973).

An alternative sterilant to such high concentrations of hydrogen peroxide has been sought which would reduce exposure time or reduce costs by operating at a lower concentration or be applied at a temperature closer to ambient. Bayliss & Waites (1982) have demonstrated a rapid kill of spores of *B. subtilis* (NCDO 2129 and 2130) with simultaneous UV irradiation and 2.5% (w/v) hydrogen peroxide although higher concentrations of hydrogen peroxide protected the spores from UV irradiation. Addition of a low level of peracetic acid to hydrogen peroxide has been found to reduce significantly the time required to achieve a given sporicidal effect (Dallyn, 1980), alternatively, for a given exposure time the hydrogen peroxide concentration could be reduced. Han, Shornick & Loncin (1980) compared the sporicidal effect of 30% hydrogen peroxide with low concentrations (0.3–1%) of peracetic acid at room temperature. The hydrogen peroxide had little effect on spores of *B. subtilis* var. *niger* (ATCC 9372) whereas the peracetic acid solutions showed considerable sporicidal activity. Similar effects were found with *B. stearothermophilus* (Oxoid Code BR 23) while *Clostridium sporogenes* (ATCC 19404) was more sensitive than the bacillus strains to both chemicals. Peracetic acid is rapidly decomposed at high temperatures to acetic acid, water and oxygen (Greenspan, Johnson & Trexler, 1955), which facilitates its removal and therefore is a suitable chemical sterilant for surface sterilization of packaging material.

In this study the chemically resistant spores of *B. subtilis* SA22 (NCA 72–52), as used by Toledo, Escher & Ayres (1973), have been used to determine the sporicidal effects of peracetic acid in the presence of primary alcohols, particularly ethanol.

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Materials and methods

Spore preparation

Bacillus subtilis SA22 (NCA 72–52) was grown at 30°C on plates of yeast dextrose tryptone agar plus starch (YDTAS) which contained per litre: 5 g peptone (Oxoid L37), 3 g beef extract (Oxoid L29), 2.5 g tryptone (Oxoid L42), 10 g glucose (BDH), 1.0 g yeast extract (Oxoid L21), 1.0 g soluble starch (BDH Analar), 15 g agar (Difco); nutrient agar (NA) (Oxoid CM3), or potato agar (PA) (Gould, Stubbs & King, 1970), for 3, 5 and 2 days respectively when > 75% sporulation, as observed microscopically, had occurred (Mottishaw, Brown & Leaper, 1982). The spores were harvested from 'lawn growths' by suspension in sterile distilled water, washed twice, resuspended in sterile distilled water and stored at 4°C until required. Storage of the spores was shown not to have any effect on their chemical resistance.

Treatment with chemicals

Measured volumes of methanol, ethanol or propan-1-ol (as appropriate) and sterile distilled water were added to sterile 150 ml conical flasks and allowed to equilibrate at 20°C in a shaking water bath (Grant Instruments, Cambridge, U.K.) at 120 strokes/min for approximately 20 min. The required volume of peracetic acid (Interox Chemicals Ltd, Widnes, Cheshire, U.K.) was added immediately prior to the addition of the appropriate spore suspension to give a total volume of 25 ml containing approximately 5×10^6 spores/ml. Aliquots (1 ml) of the mixture were aseptically removed at timed intervals and mixed with 1 ml of the appropriate sodium hydroxide solution. Sodium hydroxide solutions of 0.02, 0.025 and 0.035 M were used to neutralize 0.04, 0.08 and 0.1% or 0.12% peracetic acid solutions respectively. The first 10-fold dilution was carried out within 30 sec of removing the samples.

Recovery and enumeration

Treated spores were serially diluted in sterile distilled water and recovered using pour plates of YDTAS. Plates were incubated aerobically at 37°C for 48 hr prior to enumeration.

Results

In the presence of 0.08% (v/v) peracetic acid, the times taken to achieve one log reduction in survival (D value) of spores of *B. subtilis* SA22 were 47.2, 25.6 and 19.9 min for spores harvested from PA, YDTAS and NA respectively. This difference in chemical resistance between spores produced on different media was also maintained for mixtures of peracetic acid and 9.9% (v/v) alcohols (Table 1).

Table 1. Average D values (min) for the effect of 0.08% (v/v) peracetic acid in the absence or presence of 9.9% (v/v) primary alcohols on spores of *B. subtilis* SA22 (NCA 72–52)

	Spores harvested from		
Treatment	PA	YDTAS	NA
Peracetic acid	47.2	25.6	19.9
Peracetic acid + methanol	17.3	9.7	5.9
Peracetic acid + ethanol	4.7	1.9	2.1
Peracetic acid + propan-1-ol	1.6	0.9	0.7

The effects of ethanol concentration on spores of *B. subtilis* SA22, harvested from NA, exposed to 0.04, 0.08, 0.1 and 0.12% (v/v) peracetic acid are shown in Figs 1–4. Log mean D values and standard errors were calculated from up to five separate experiments for each combination of ethanol and peracetic acid. The D values were calculated from all the points obtained from the individual survival curves, thus where 'shouldering' was present this was taken into account. A decrease in the log D value resulted from the addition of increasing concentrations of ethanol to the peracetic acid. For 0.04% (v/v) peracetic acid the effect reached a plateau at concentrations of ethanol

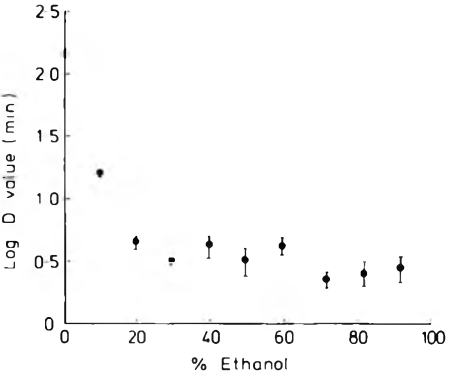

Figure 1. Effect of ethanol concentration on spores of *Bacillus subtilis* SA22 harvested from NA exposed to 0.04% (v/v) peracetic acid. Mean D values are plotted together with the standard error.

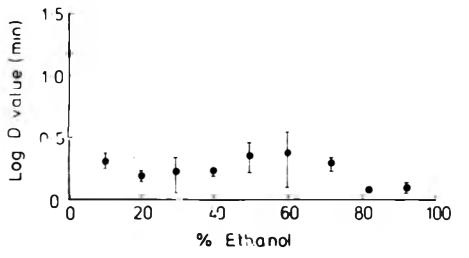

Figure 2. Effect of ethanol concentration on spores of *Bacillus subtilis* SA22 harvested from NA exposed to 0.08% (v/v) peracetic acid. Mean D values are plotted together with the standard error.

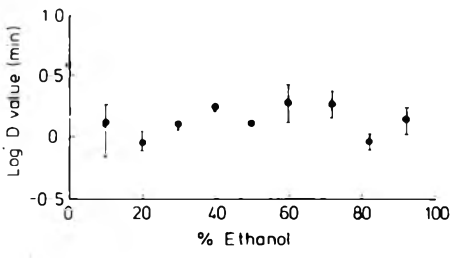

Figure 3. Effect of ethanol concentration on spores of *Bacillus subtilis* SA22 harvested from NA exposed to 0.1% (v/v) peracetic acid. Mean D values are plotted together with the standard error.

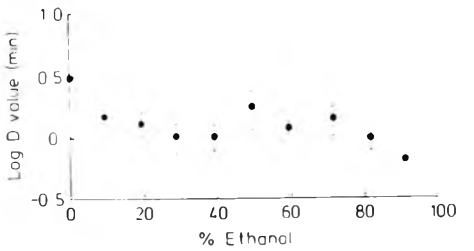

Figure 4. Effect of ethanol concentration on spores of *Bacillus subtilis* SA22 harvested from NA exposed to 0.12% (v/v) peracetic acid. Mean D values are plotted together with the standard error.

above 30% (v/v) whereas for 0.08% (v/v) peracetic acid no further decrease in the log D value was observed above 20% (v/v) ethanol. There was a decrease in the log D value when 0.1 or 0.12% (v/v) peracetic acid was tested with 9.9% (v/v) ethanol but this was not apparent at higher ethanol concentrations. Spores harvested from PA and YDTAS behaved in a similar manner. Although the resistance to 0.04% (v/v) peracetic acid alone varied between the spore crops this variation was not so apparent when ethanol/peracetic acid combinations were tested.

Discussion

Although Morton (1968) and Russell (1971) have reported ethanol to be ineffective as a germicidal agent against spore-forming bacteria, the present study indicates that a synergistic sporicidal effect is produced by the addition of primary alcohols to peracetic acid. This addition is reported to result in the formation of hydrogen peroxide and the corresponding acetate ester (Greenspan *et al.*, 1955). Although the precise mode of sporicidal action of the chemicals under investigation is unknown, Fraser (1980) suggested that peracetic acid may promote oxidation of cellular material by action upon the lipoprotein cytoplasmic membrane disrupting its physiological role. The cellular targets for the action of hydrogen peroxide, formed by the addition of alcohol to peracetic acid, are believed to be enzymes with thiol groups, ribosomes and thiol groups (Hugo, 1977). If hydrogen peroxide is formed in the system under investigation the spores might be destroyed by hydroxyl radicals formed by decomposition of the hydrogen peroxide, as suggested by King & Gould (1969) and Bayliss & Waites (1981).

Alcohols in low concentrations have been reported to have an inhibitory effect on the initial rate of *L*-alanine-initiated germination of *B. subtilis* spores (Yashuda-Yasaki, Namiki-Kanie & Hachisuka, 1978) and affect general membrane permeability of vegetative cells (Hugo, 1977).

The effect of the addition of ethanol to peracetic acid was further investigated since ethanol would be the most favoured alcohol as a sterilant component for the food packaging industry, as residues could be removed by an increase in temperature and would be the least likely to cause any tainting.

Spores of *B. subtilis* SA22 (NCA 72–52) produced on NA, are known to be approximately 40 times more resistant to 0.04% peracetic acid than spores of *B. subtilis* (NIRD 1069) and approximately 100 times more resistant than spores of *B. subtilis* var. *niger* (NCIB 8058) and an NCA strain of *B. subtilis* var. *globigii* (Mottishaw *et al.*, 1982). Spores of *C. sporogenes* (3679), used as an anaerobic reference strain in place of

C. botulinum, appeared to be extremely sensitive to peracetic acid with 0.004% (v/v) proving sporicidal after only a few minutes treatment at 20°C whereas bacillus strains could survive 0.04% (v/v) peracetic acid for periods from several minutes to several hours (Mottishaw *et al.*, 1982). However no synergistic sporicidal effect was demonstrated with spores of *C. sporogenes* exposed to 9.5% (v/v) ethanol and 0.002% (v/v) peracetic acid.

The 'shouldering' as noted on the survival curves for ethanol/peracetic acid combinations was also reported by Swartling & Lindgren (1968) for the effect of hydrogen peroxide at 50 or 60°C but was less marked at 70 or 80°C. Since there is no agreed method or satisfactory explanation for dealing with 'shouldering', all data points from the survival curves in the present study were considered in the D value calculations (cf. Swartling & Lindgren, 1968). Toledo *et al.* (1973) reported a lag in inactivation of *B. subtilis* var. *globigii* by hydrogen peroxide which could be reduced by an increase in hydrogen peroxide concentration or by an increase in temperature. They also noted that the more resistent the organism—e.g. *B. subtilis* SA22 and *B. subtilis* var. *globigii*—the longer the lag period, whereas less resistant organisms—e.g. *C. sporogenes* (3679) and *Staphylococcus aureus*—followed first-order reaction kinetics.

Changes in the composition of the sporulation media have been shown to alter the resistance of *B. subtilis* (NCDO 2130) spores to hydrogen peroxide, peracetic acid, choline, heat and UV irradiation (Bayliss, Waites & King, 1981) which may be due to changes in the spore coat properties. This present data indicate that spores of *B. subtilis* SA22 produced on different media show a difference in resistance to peracetic acid and 9.9% (v/v) alcohol/peracetic acid combinations. However the difference was not apparent at all ethanol concentrations.

This study has shown, therefore, that peracetic acid in combination with low levels of ethanol is an effective sterilant since it proved sporicidal against the chemical-resistant spores of *B. subtilis* SA22.

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Effect of berry maturation on some chemical constituents of black, green and white pepper (*Piper nigrum* L.) from three cultivars

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Summary

The accumulation pattern of three commercially important constituents (starch, non-volatile ether extract and piperine) in black, green and white pepper products of a local Sri Lankan cultivar has been compared with that for the Panniyur and Kuching cultivars introduced from India and Sarawak respectively. The local cultivar contained a higher level of starch and lower levels of non-volatile ether extract and piperine than the other two cultivars. The local cultivar has a composition satisfactory for its use in the whole berry form or for processing into a powdered spice.

Introduction

Pepper (*Piper nigrum*), known as the king of spices, is one of the earliest spices known to man. Today it is the largest commodity in the international spice trade, contributing about 40% or 77 000 t to total world trade, valued at about US $100m per year (Bavappa, 1979). The two major primary products which trade internationally are black and white pepper. Black pepper is prepared by drying the whole, unripe but fully developed berry while white pepper consists of fully ripe dried fruit from which the pericarp has been removed. Green pepper is another form of pepper which was introduced only recently to the world market and is prepared from unripe but fully developed berries which are artificially dried or preserved in the wet form in brine, acetic acid or citric acid (Pruthi *et al.*, 1976). The stage of harvesting pepper berries is dependent on the product to be prepared. In Sri Lanka, berries are harvested at 5–6 months, 4.5–5 months and 6 months for the preparation of black, green and white pepper products respectively. Fully-matured pepper berries can be obtained 6 months after fruit setting.

Oleoresin constitutes a solvent-extracted spice preparation possessing particular flavour characteristics and containing both volatile and non-volatile substances. In the case of pepper oleoresin, the odour and flavour are determined by the composition of its aromatic volatile oil, while the characteristic pungency is produced by non-volatile alkaloids of which piperine is the most important (Cripps, 1973; Genest, Smith & Chapman, 1963; Purseglove *et al.*, 1981; Wijesekara, Senanayake & Jayewardene, 1972). Pepper oleoresin is a popular and important flavouring agent in the food industry since it is easy to handle commercially and the risk of biodeterioration and microbial contamination is reduced. An oleoresin can be used to replace 20–25 times its weight of spice for an equivalent flavouring effect. When oleoresin is extracted from pepper berries, the high pungency level and volatile oils are important constituents for food

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manufacturers. Although starch does not contribute towards the flavour qualities or pungency of pepper, it is important in the manufacture of powdered spices (Mathai, 1981). Pepper berries intended for direct use as a spice in the whole or ground form should be high in starch content. All of these quality characteristics vary with variety or cultivar, maturity of the berries and method of preparation of the pepper product (Genest *et al.*, 1963; Jose & Nambiar, 1972; Lewis *et al.*, 1969; Mathai, 1981; Mathai, Kumaran & Chandy, 1981; Nambudiri *et al.*, 1970; Purseglove *et al.*, 1981).

In this study we have evaluated a local Sri Lankan pepper cultivar and two cultivars introduced into Sri Lanka for some commercially important chemical constituents as related to the maturity of the berries and the type of preparation.

Materials and methods

Black, green and white pepper samples each derived from a local Sri Lankan cultivar, an Indian hybrid (Panniyur-1) and a cultivar introduced from Sarawak (Kuching), and harvested at different maturities (see Table 1) were obtained from the Minor Export Crops Research Station, Matale, Sri Lanka. These samples had been prepared as described below.

Table 1. Starch content of three pepper cultivars

Age of berries (months)	Starch content (% dry weight basis)		
	Panniyur	Kuching	Local
Black pepper			
2	13.2	14.9	15.9
3	14.2	15.9	16.2
4	15.3	17.8	19.5
5	37.3	40.8	44.0
6	47.8	50.1	51.9
Green pepper			
4	13.8	13.9	17.0
4.5	15.8	16.4	18.3
5	23.8	26.3	29.3
5.5	44.5	46.9	49.3
White pepper			
5	50.6	51.9	52.6
5.5	56.0	57.5	59.4
6	57.9	59.8	60.7

Methods of preparation

Black pepper. After harvesting, the browning fermentation was initiated by piling the berry clusters in a heap 30–40 cm high and exposing them to the sun for several hours followed by spreading the berries on to a cement drying floor in the sun for 3–5 days.

Green pepper. After harvesting the berries were cooked in boiling water for 15 min and then oven dried for 6–8 hr at 55°C.

White pepper. After harvesting, the berries were cooked in boiling water for 5 min, the pericarp was removed by hand and the seed sun dried for 2–3 days.

Preparation of dried berries for analysis

All pepper samples were ground to a fine powder in a coffee grinder and stored in glass jars at room temperature for immediate chemical analysis.

Analysis

Moisture was determined by Method 30.005 of the Association of Official Analytical Chemists (AOAC, 1980). Starch was determined by the American Spice Trade Association (ASTA) analytical method 8.0 (Anon., 1968). For determination of non-volatile ether extract (NVEE), pepper samples (5.00 g) were extracted for 20 hr with diethyl ether in a soxhlet apparatus, the ether removed by vacuum distillation and the extract heated to constant weight at 100°C. Piperine was determined by high performance liquid chromatography of the non-volatile ether extract by the method of Rathnawathie & Buckle (1983). All chemicals and solvents were of analytical reagent grade. Water was double distilled in glass.

Results and discussion

The three types of berries analysed were of different maturity, with five maturities (2, 3, 4, 5 and 6 months) examined for black pepper. Since green pepper is prepared in Sri Lanka from berries which are three-quarters mature, berries aged 4, 4.5, 5 and 5.5 months were analysed. The white pepper analysed had been prepared from berries harvested at three maturity stages (5, 5.5 and 6 months).

Starch

The proportion of starch (Table 1) increased as the berries reached maturity for black, green and white pepper products, in agreement with the observation of Mathai (1981) for black pepper of the Panniyur cultivar. Mathai (1981) analysed berries up to 8.5 months of age but under Sri Lankan climatic conditions fully matured black pepper berries are normally obtained 6 months after fruit setting. All black pepper samples showed a gradual increase in starch content up to 4 months and then the level increased rapidly. While all green pepper samples showed a rapid increase of starch content after 4.5 months, white pepper samples showed only a gradual increase in starch content with maturation, since berries were harvested only after 5–6 months. Of the three cultivars examined, the local Sri Lankan cultivar showed the highest starch content for all types of pepper of comparable maturity, while the Panniyur cultivar contained the lowest. The outer skin forms about 25% of the dry weight of pepper and consists mainly of fibre and some of the essential-oil cells (Purseglove *et al.*, 1981). Since the fibrous skin is removed during the preparation of white pepper, these samples, as expected, contained higher starch levels than the other two products, while green pepper contained the lowest starch levels.

Non-volatile ether extract

Results for NVEE (Table 2) showed no common trends for the three cultivars examined. Black pepper showed a substantial rise in NVEE content between 3 and 4 months' maturation and thereafter the level gradually decreased. Mathai (1981) found similar behaviour with the oleoresin content of black pepper. The Panniyur cultivar

Table 2. Non-volatile ether extract content of three pepper cultivars

Age of berries (months)	Non-volatile ether extract content (% dry weight basis)		
	Panniyur	Kuching	Local
Black pepper			
2	6.7	6.0	6.6
3	7.2	6.7	7.1
4	14.2	12.5	10.2
5	13.8	11.8	8.8
6	7.6	7.6	7.4
Green pepper			
4	15.0	13.6	12.1
4.5	15.8	14.6	13.8
5	14.8	12.4	11.7
5.5	8.1	8.1	7.9
White pepper			
5	8.9	8.5	8.1
5.5	8.2	7.9	7.6
6	7.7	7.5	7.4

contained the highest level of NVEE while the local cultivar contained the lowest after 4 months' maturation. Further maturation resulted in a decrease in NVEE content for all cultivars to a level between 7.4 and 7.6% (dry weight basis). For green pepper, the NVEE content reached a maximum level at 4.5 months and then decreased with increasing maturation in all three cultivars. For white pepper samples at the fully matured stage, NVEE content decreased with further maturation from 5 to 6 months, at which stage the local Sri Lankan cultivar contained slightly less than the Panniyur and Kuching cultivars, an observation in common for both black and green pepper samples. Green pepper samples contained a higher level of NVEE than did black or white pepper, irrespective of the cultivar.

Piperine in NVEE

The piperine content of NVEE of black pepper (Table 3) increased in berries up to 4 months of age and then decreased, in line with the content of NVEE. Although the Panniyur cultivar contained the highest level of NVEE in black pepper, the Kuching cultivar showed the highest piperine content and the local Sri Lankan cultivar contained the lowest. Mathai (1981) has also shown a relationship between the piperine content and maturity of black pepper berries of the Panniyur cultivar. The piperine content of green pepper reached a maximum level after 4.5 months and then gradually decreased, except for the Kuching cultivar which showed the highest level of piperine after 5 months before a reduction at 5.5 months' maturity. For white pepper samples, the piperine content decreased between 5 and 6 months' maturation. The highest levels of piperine in white pepper, as for the other two pepper products, were found in the Kuching cultivar. Since the NVEE of pepper contains fatty oils, pungent alkaloids and other nitrogenous but non-pungent extractives (Purseglove *et al.*, 1981), cultivars which have the highest levels of NVEE may not necessarily also show the highest level of piperine. In our study, the highest levels of NVEE and piperine were shown by the

Table 3. Piperine in non-volatile ether extract of three pepper cultivars

Age of berries (months)	Piperine content (% NVEE)		
	Panniyur	Kuching	Local
Black pepper			
2	36.1	36.0	34.9
3	46.7	49.1	46.7
4	51.5	55.4	49.8
5	47.5	53.3	48.3
6	45.5	49.3	44.9
Green pepper			
4	52.7	58.3	51.3
4.5	54.4	59.9	54.1
5	52.4	62.8	52.4
5.5	48.3	52.9	48.2
White pepper			
5	47.6	49.5	46.2
5.5	44.3	48.1	42.4
6	43.3	46.7	40.9

Panniyur and Kuching cultivars, respectively, for each of the black, green and white pepper products. Green pepper showed the highest levels of piperine in all samples tested while the white pepper products contained the lowest. The general pattern of accumulation of piperine during maturation of berries of the three cultivars analysed appears to be similar to that for NVEE. The sudden rise of the starch content in pepper berries towards the end of maturation is related to the proportionate fall in the NVEE content during the same stage of development.

Table 4. Comparison of pungency products for three pepper cultivars

Age of berries (months)	Panniyur			Kuching			Local		
	NVEE	Piperine	Prod.*	NVEE	Piperine	Prod.	NVEE	Piperine	Prod.
Black pepper									
2	6.7	36.1	241.9	6.0	36.0	216.0	6.6	34.9	230.3
3	7.2	46.7	336.2	6.7	49.1	329.0	7.1	46.7	331.6
4	14.2	51.5	731.3	12.5	55.4	692.5	10.2	49.8	508.0
5	13.8	47.5	655.5	11.8	53.3	628.9	8.8	48.3	425.0
6	7.6	45.5	345.8	7.6	49.3	374.7	7.4	44.9	332.3
Green pepper									
4	15.0	52.7	790.5	13.6	58.3	792.9	12.1	51.3	620.7
4.5	15.8	54.4	859.5	14.6	59.9	874.5	13.8	54.1	746.6
5	14.8	52.4	775.5	12.4	62.8	778.7	11.7	52.4	613.1
5.5	8.1	48.3	424.4	8.1	52.9	428.5	7.9	48.2	380.8
White pepper									
5	8.9	47.6	423.6	8.5	49.5	420.8	8.1	46.2	374.2
5.5	8.2	44.3	363.3	7.9	48.1	379.9	7.6	42.4	322.2
6	7.7	43.3	333.4	7.5	46.7	350.3	7.4	40.9	302.6

*Pungency product = NVEE (% dry weight basis) × piperine content (% NVEE).

Table 4 compares the pungency product (i.e. NVEE × piperine content) for the three cultivars examined, and clearly shows that each of the three cultivars give maximum figures after 4 months for black pepper, 4.5 months for green pepper and 5 months for white pepper. For each of the three types of pepper product, the local cultivar gave a pungency product lower than for the Panniyur and Kuching cultivars, especially for black pepper. The differences between cultivars were greatest at the maturity at which the highest pungency product occurred. The differences in pungency product between the imported varieties were small when compared to the differences between either of these cultivars and the local cultivar.

The selection of the proper cultivar for processing is dependent on whether the berries are intended primarily for oleoresin production or for use as a powdered spice. The local Sri Lankan cultivar containing a slightly higher level of starch and a lower level of NVEE compared to the introduced cultivars is considered to have a composition satisfactory for processing into the ground spice, while the Panniyur and Kuching cultivars have properties suitable for both end purposes, but are considered more suitable for oleoresin production.

Further studies are in progress to establish the qualitative and quantitative differences in volatile flavouring constituents in pepper products derived from the three cultivars grown in Sri Lanka and harvested at various stages of maturation.

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Preparation and properties of a new generation of in-can sterilized skim-milk based concentrates

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Summary

Pilot-scale production of a range of novel heat stable concentrates is described. The concentrates containing *circa* 40% total solids—largely milk protein and various sugars—were favourably compared with conventional evaporated milk and exhibited exceptional storage stability.

Introduction

It has long been recognized that the mineral balance of milk is an important determinant of the heat stability of concentrate (e.g. Sommer & Hart, 1926). Although the detail of the mechanism of heat coagulation of concentrated milk remains unresolved, various studies have indicated that soluble salts, especially calcium, are particularly important and that the partial removal of soluble milk salts can promote very large increases in heat stability (e.g. Rose, 1961; Fox & Hearn, 1978; Muir & Sweetsur, 1978a; 1984; Sweetsur & Muir, 1980a,b). Laboratory studies have indicated that the process of ultrafiltration (UF) has potential for producing heat stable concentrates, because ultrafiltration selectivity concentrates milk protein with a concomitant removal of soluble salts (Sweetsur & Muir, 1980a). Furthermore, the protein : carbohydrate ratio of the concentrate so prepared can be restored by addition of carbohydrate to that of original milk (*circa* 0.7) with no serious loss in stability (Sweetsur & Muir, 1980b). This paper describes the extension of the laboratory studies to pilot-scale production of a range of novel heat stable concentrates. The properties of these concentrates containing *circa* 40% total solids—comprised of milk protein and various sugars—were also considered.

Materials and methods

Samples

Milk was collected from the Hannah Research Institute farm bulk tank and after warming to 40°C the fat was removed using a small, open, centrifugal cream separator. When quantities of skim-milk in excess of 150 l were required, high quality skim milk was purchased from a nearby creamery.

Lactose, glucose and sorbitol (Analar grade) were purchased from Laboratory suppliers and sucrose was obtained from local retail outlets. Glucose syrup (~ 27 dextrose equivalent) was obtained from CPC Ltd.

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Ultrafiltration of milk

The processes found to be of practical application for the production of concentrates are shown in Fig. 1.

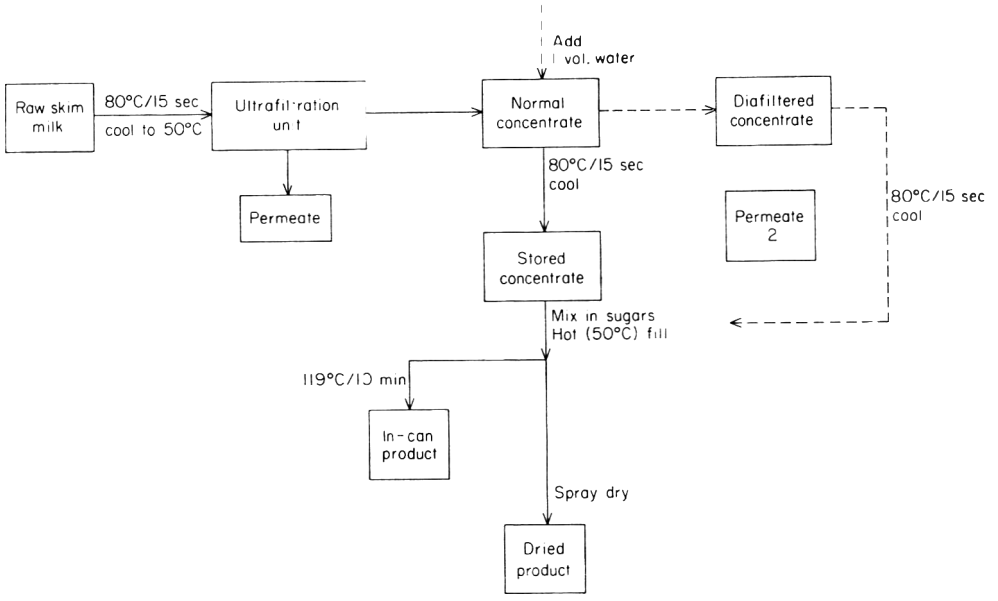

Figure 1. A schematic diagram of the process for production of sterile and dried concentrates.

Cold skim-milk was continuously pasteurized (80°C, 15 sec) using a pilot-scale heat exchanger (APV Co. Ltd, Junior Paraflow, 540 l/hr) and taken from the plant at 40–50°C immediately prior to concentration. The skim-milk was then concentrated to between one-fifth and one-sixth volume using a pilot-scale ultrafiltration plant (Paterson Candy Ltd, Whitchurch, Herts, type BX6 membranes, 20.8 m^2 membrane). During concentration the temperature of the milk was held at 50±2°C by means of a trim cooler/heater. The total solids content of the concentrate was monitored with a hand-held sugar refractometer (Atago Ltd, Chemlab Instruments, London) which had been previously calibrated against UF concentrates of known composition.

The process of diafiltration was also tested. Water is added to the milk (or concentrate) to dilute the aqueous phase and after concentration to the same level of total solids—necessitating removal of a greater volume of permeate—the concentrate contains a significantly lower level of soluble low molecular weight constituents. In this work, an equal volume of water was added to the concentrate and the process of ultrafiltration continued until the original degree of concentration was re-attained (cf. Muir & Sweetsur, 1984).

After concentration, the product was re-pasteurized (80°C/15 sec) and cooled to 4°C for storage. Provided the concentrate was heat treated , the product was both bacteriologically and chemically stable for up to 5 days.

Formulation of protein–carbohydrate mixtures

Prior to sterilization the concentrates were warmed to 40°C and carbohydrate (as solid) was added with vigorous stirring using a high speed mixer (Silverson Machines,

Chesham, Bucks). When lactose was being added to concentrate it was necessary to heat the mixture *circa* 70°C to achieve solubilization in a reasonable time. The hot concentrate was then filled into standard unlacquered three piece tins (*circa* 420 ml capacity) and the ends seamed on with a pilot-scale can seamer (Metal Box Co. Ltd).

Sterilization procedure

The filled and seamed cans (9×420 ml) were placed in a small rotary autoclave (38 rev/min) and the temperature raised to 119°C over a period of 5 min. When this processing temperature was attained the product was cooked for exactly 10 min, after which the cans were rapidly cooled (over *circa* 3 min) to room temperature in running water. The product was then stored at 15, 37 and 45°C.

Since the autoclave was not fitted with a facility for measurement of internal can temperature (i.e. a slip ring and thermocouples) it was necessary to devise an alternative technique for monitoring sterilization effect. Bunn & Sykes (1981) reported that a chemical indicator strip (Thermalog S, Bennett & Co., Brimpton Common, Berks) could be used effectively in processes where the total lethality F_0 lay in the range 4–23 F_0 units. (The sterilization value of a process is generally expressed as the F_0 value which is equivalent to the number of minutes required to destroy a specified number of spores at 250°F, i.e. 121.1°C. For example, at an $F_0 = 2.45$ the heat process would expect to reduce the survival population of spores of *Clostridium botulinum* in phosphate buffer by a factor of 12 decimal values.) Whilst Bunn & Sykes (1981) placed the indicator strip into specially adapted vials containing a little water, it was found that the strip could be immersed in water, milk or concentrate with no loss in reproducibility or readability. Preliminary experiments using a continuous commercial autoclave justified the claims of Bunn & Sykes (1981) for the readings from the indicator strips corresponded well with results from a full scale telemetric calibration. Subsequently, the indicator strips have been used routinely to monitor each heat process. First, the position of a can in the autoclave was shown to be unimportant for the range of values obtained at F_0 values of 10.6 (s.e. = 0.12) and 12.6 (s.e. = 0.16) was within the repeatability of values from a number of strips treated together in a single can. Second, the autoclave was calibrated for a range of heating times at 119°C. The results are shown in Table 1. For routine treatments, a heating time of 10 min was chosen since this corresponds to $F_0 = 5$, i.e. the normal commercial treatment for full-cream evaporated milk. In practice, milk treated by this process was sterile when tested bacteriologically (after 1 week pre-incubation at 30 or 37°C).

Product evaluation

The total solids contents of concentrates were measured by the hot plate method described in Muir & Sweetsur (1978b) after preliminary 4-fold dilution with distilled water.

Alcohol stability was assessed by diluting equal volumes of concentrate (2 ml) with alcohol solutions of different composition. The results are expressed as the concentration of alcohol (v/v) at which visible clots are formed.

The pH of the concentrates was estimated using a previously standardized pH combination electrode and a digital pH meter (Phillips model PW9410).

The stability of various products in hot coffee was measured by a modification of the test described by Sweetsur (1976). Concentrate (10 ml) was added to freshly prepared coffee solution at 85°C (100 ml, 1.3% coffee solids, pH *circa* 4.8). After 10 sec, the

coffee was stirred briskly for 10 rev and the extent of formation of visible scum or floccules noted.

Product viscosity was evaluated by means of a 'Rheolog' viscometer (Brookfield Engineering Laboratories Inc., Stoughton, U.S.A.) in a thermostatted (30°C) small sample adaptor. Measurements were normally made at a shear rate of 79/sec.

Organoleptic and colour assessments were made by a small panel of trained and experienced tasters. Products were given a score for a given attribute (0–5) and the mean score (rounded to the nearest integer) reported. Since the object of this work was to obtain an overall impression of the new products extensive tasting and statistical analysis were not carried out.

Results and discussion

A comparison of the properties of concentrates containing different sugars

A range of concentrates containing 40% total solids was prepared in which milk protein concentrate was combined with sucrose, glucose, lactose, sorbitol and corn syrup (27 D.E.). Typical results are shown in Table 2. A clear pattern emerged, for without exception concentrates containing non-reducing sugars could be sterilized without difficulty to yield a fluid, slightly coloured product. The products were similar in viscosity to conventional evaporated milk although the solids content was almost one-third greater and, more significantly, the solid-not-fat content was almost double. concentrates containing sucrose and sorbitol were always stable after 10 min sterilization (F_0 = 5) and often stable after a 15 min treatment ($F_0 \sim 10$, Table 1).

In direct contrast, it has not been possible to prepare analogous concentrates containing reducing sugars. When lactose, glucose or corn syrup were used as the carbohydrate component, products were grainy and had gelled and could not be safely sterilized (Table 2). Furthermore, the intensity of brown coloration was related to the reducing power of the sugar. It is noteworthy that concentrates formulated with corn syrup (D.E. = 27) were particularly unstable although the pH drop during sterilization was not particularly marked. These results confirm our earlier laboratory findings (Sweetsur & Muir, 1980a) and suggest that the stability of concentrates is highly dependent on the presence of small quantities of reducing sugar. This aspect of the product formulation was then considered in detail.

Table 1. Processing time and F_0 value for concentrates*

Heating time† (min)	Indicator strip (mm)	F_0 values†
2.5	< 5.0	< 1
5.0	< 5.0	< 1
7.5	10.0	1–2
10	16.0	5
15	28.0	10.5
20	35.8	14.5

*Measurements were replicated on both sorbitol and sucrose type concentrates.

†Temperature of steam = 119°C.

†From a practical viewpoint $F_0 \leq 4$ was normally taken to indicate a lack of sterility.

Table 2. A comparison of the properties of concentrates containing different sugars

Added sugar	TS (%)	Stable heating time (min)	Properties after standard heating (F_0 = 5) Viscosity (cp)	pH	Colour*	Gel/precipitate formation*
Sucrose	40.7	10–15	140	6.56	+	Nil
Sorbitol	40.9	10–15	120	6.79	+	Nil
Lactose	40.9	2.5–5.0	—	6.39	++	+++
Glucose	40.7	5.0–7.5	—	6.07	++++	++++
Corn syrup	40.7	< 2.5	—	6.49	+++	++++
Conventional evaporated milk	31.0	10–15	~ 100	6.28	+	Nil

*+ → ++++ indicates intensity of colour or precipitate formation.

Properties of concentrates containing mixtures of non-reducing and reducing sugars

Experiments were than carried out in which mixtures of non-reducing and reducing sugars were used as the carbohydrate component of concentrates. Representative results are shown in Table 3. There appears to be no practical way in which reducing sugars can be used in significant quantities for all the mixtures tested had coagulated after sterilization.

This result was corroborated by another series of experiments in which various sorbitol–glucose mixtures were used as the sugar portion of concentrates (Table 4). As

Table 3. Properties of sterile concentrates containing mixtures of reducing and non-reducing sugars

	TS (%)	Properties after standard heat treatment (F_0 = 5) pH	Colour	Gel/precipitate formation
Lactose : sucrose				
25 : 75	40.6	6.49	++	+++
50 : 50	40.7	6.42	++	+++
75 : 25	40.6	6.29	++	+++
Glucose syrup : sorbitol				
50 : 50	41.0	6.54	++	+++
Glucose syrup : sucrose				
50 : 50	41.0	6.45	++	+++

Table 4. Sterile concentrates containing mixtures of glucose and sorbitol

Glucose (%)	Sorbitol (%)	Properties after standard heating (F_0 = 5) Viscosity (cp)	pH	Colour	Gel/precipitate formation
0	100	120	6.79	+	Nil
5	95	270	6.72	++	Nil
10	90	2400	6.65	+++	+/−
20	80	—	6.49	+++	++
50	50	—	6.18	++++	++++

the proportions of glucose in the sugar fraction increased the viscosity of the sterile product also increased until, at over 10% incorporation of glucose gel formation occurred. This decrease in stability was associated with a corresponding drop in pH after heating and by an increase in brown coloration.

Clearly, the proportion of reducing sugar in the carbohydrate component of sterile concentrates is an important determinant of stability. Although the results suggest that no reducing sugar should be incorporated for maximum stability, some residual lactose is present from the original milk. This residual lactose comprises between 10 and 14% of the sugar component and is therefore equivalent to between 5 and 7% in terms of reducing power. This fact may be a contributing explanation of our observation, made under laboratory conditions (Muir & Sweetsur, 1984), that diafiltration improves heat stability. The diafiltration technique was therefore considered on the pilot scale.

Effect of diafiltration on concentrate stability

Concentrates containing sorbitol and sucrose were manufactured from milk concentrated by ultrafiltration and from the same concentrate subsequently diafiltrated (see Materials and methods for technique used) and the properties of the concentrates examined. In every case, as the severity of the sterilization process increased, the pH of the concentrate fell and viscosity increased until a gel was formed (Table 5). However, diafiltration did significantly improve heat stability irrespective of the sugar used. There was no readily perceptible difference in either taste or colour of the corresponding ultrafiltered and diafiltered products, although the samples were not subjected to a rigorous organoleptic testing.

Table 5. A comparison of concentrates prepared by conventional ultrafiltration and diafiltration

		Ultrafiltered			Diafiltered		
Heating time (min)		Viscosity (cp)	pH	Gel/ppt*	Viscosity (cp)	pH	Gel/ppt*
7.5	Sorbitol	90	6.84	−	115	6.87	−
10.0		120	6.79	−	135	6.83	−
15.0		3300	6.73	+	720	6.75	−
20.0		—	6.66	+++	—	6.67	++
7.5	Sucrose	80	6.63	−	115	6.67	−
10.0		140	6.56	−	135	6.63	−
15.0		—	6.50	++	1700	6.56	+/−
20.0		—	6.41	+++	—	6.50	+++

* − no gel or ppt; + → ++++ increasing intensity of gel or precipitate formation.

The increase in heat stability brought about by diafiltration was thus of limited practical value since the untreated concentrates already survived the minimum effect sterilization. Nevertheless, in circumstances where the initial bacteriological quality of milk was poor and a higher degree of sterilization was required diafiltration is a useful technique.

Storage stability of concentrates

One important feature of any sterilized product is its shelf life, for however attractive its other properties the product must be physically stable to allow adequate

distribution. Samples of concentrates containing sorbitol and sucrose (and a control sample of the milk protein concentrate without added sugar) were stored for 7 months at 15, 37 and 45°C. The properties of the products after storage are shown in Table 6. The sucrose and sorbitol containing samples behaved in an almost identical manner, becoming darker in colour as the storage temperature increased. Nevertheless, even after storage at 45°C there was no appreciable gel formation and the products had become markedly less viscous during storage (cf. Table 5). It is noteworthy that the samples with no added sugar showed various degrees of aggregation during storage with heavy precipitates being formed at the higher storage temperatures.

Table 6. The changes in properties of novel concentrates during storage for 7 months at various temperatures

	Control			Sorbitol/sucrose		
	15°C	37°C	45°C	15°C	37°C	45°C
Colour	+	++	+++	+	++	+++
Viscosity (cp)	1800	ppt	ppt	50	45	45
pH	6.46	6.15	6.00	6.38	6.06	5.81

ppt = precipitate formed.

By comparison with conventional evaporated milk, the novel concentrates are very stable, since evaporated milk exhibits greater change under these very severe test conditions.

A comparison of the properties of the novel concentrates with conventional evaporated milk

A limited comparison of the properties of a range of sterile concentrates was made with evaporated milk. Typical results are shown in Table 7. All concentrates were highly stable when added to hot coffee and had maximum scores in a 'coffee' test. The alcohol stability of the new range of concentrates was higher than that of conventional evaporated milk but this may in fact reflect differences in pH (Table 2).

Table 7. A comparison of novel concentrates with conventional evaporated milk

			Stability indices		Organoleptic properties*		
Product	Colour	TS (%)	Alcohol (%)	Coffee	Sweet	Caramel	Chalk
Evaporated milk	+	31	70	+5	0	2	2
Sucrose UFC†	+	41	80	+5	4	0	2
Sorbitol UFC	−	40	80	+5	2	0	2
Glucose (5%)/Sorbitol	++	41	80	+5	2	1	2
Lactose†	−	40	80	+5	1	0	2

*The properties were scored 0–5 by a small panel of trained experts where 0 − absent, 5 = overpowering.

†UFC = concentrate prepared by ultrafiltration.

‡This sample had a minimum heat treatment equivalent to F_0 = 1 and was used because at F_0 = 5 clotting occurred.

When the concentrates were subjected to organoleptic assessment (undiluted) some clear differences emerged. A small panel of experts attributed scores for sweetness, caramel/cooked flavour and chalkiness (Table 7). The overriding attribute of all the novel concentrates was that of sweetness. Even when a lactose containing concentrate was tasted it was perceptibly sweet. Caramel/cooked flavour was present only in the samples of conventional evaporated milk and in that containing 5% glucose. All samples had some chalky flavour/mouthfeel, although this attribute was not particularly marked. The overall impression was favourable and no off-flavours or taints were detected.

Notwithstanding the results of the above evaluation, the organoleptic assessment was of limited value since it is not anticipated that the concentrates would be consumed directly. Rather it is likely that they might be used in coffee, confectionery, in desserts, and as an adjunct when baking. In the first of these roles the new range of concentrates have attractive properties but their potential use in confectionery and baking has yet to be evaluated.

Conclusions

The experimental results discussed in this paper show that the laboratory findings of our earlier work may be translated into a new generation of high solids, sterile concentrate based on milk protein. The limitations of the formulation of such material are described and especial attention has been drawn to the hitherto unsuspected deleterious role played by small amounts of reducing sugar. The novel concentrates prepared in this work were favourably compared with conventional evaporated milk and exhibited exceptional storage stability. Further work is in progress to evaluate end uses of the products and also to produce a range of concentrates containing fat.

Acknowledgments

Miss A. G. Wilson and Mr J. Abbot are thanked for their expert technical assistance. This work was supported in part by a grant to Miss A. G. Wilson and Mr J. Abbot by E.E.C. Commission under Regulation No. 2935/79 contract No. 60.

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Book Reviews

Food Carbohydrates. Ed. by David R. Lineback and George E. Inglett. Westport, Conn.: AVI, 1982. Pp. xii+494. ISBN 0 87055 400 X. US$49.50.

This substantial text is a compilation of the papers, twenty-three in all, presented at a joint meeting of the Institute of Food Technologists and the International Union of Food Science and Technology, held in June 1981. The book presents chapters on: 'Modern Methods of Analysis of Corn-derived Sweeteners' (Bernetti), 'Corn Syrup Selections in Food Applications' (Strickler), 'Taste Receptor Origin of Sweetness' (Beidler), 'Fructose—A Regulatory Perspective' (Frattali), 'Polyols: Chemistry and Application' (Emodi), 'Sucrose and Health' (Bollenback), 'Lactose and the Sugars of Honey and Maple' (Donner and Hicks), 'Sugar Dehydration Reactions' (Feather), 'The Influence of Food Carbohydrate on Dental Caries' (De Paola), 'Carbohydrates, Sweet Taste and Obesity' (Drewnowski, Gruen and Grinker), 'Health Implications of Food Carbohydrate' (Reiser), 'Lectins: Carbohydrate-binding Proteins in Search of a Function' (Goldstein), 'Current Concepts of Starch Structure' (Hood), 'Starch Derivatives and Their Use in Foods' (Smith), 'Food Applications of Gums' (Glicksman), 'Dietary Fibre in Health and Disease' (Kritchevsky), 'Effect of Preparative Procedure on the Evaluation of *in vitro* Indigestible Residue' (Rasper), 'Analysis of Polysaccharides' (Aspinall), 'Protein—Polysaccharide Interactions' (Cherry), 'Hydrocolloid Interactions with Starches' (Christianson), 'Polysaccharide Conformation in Solutions and Gels' (Dea) and 'Metabolism and Mutagenic Effects of Maillard Reaction Products' (Perkins, Becher, Genther and Martin).

The book aims to assemble the recent work on chemistry, analysis, functional properties and nutritional considerations over the whole spectrum of food carbohydrates, and the editors achieve this task admirably. One criticism of the book is that twenty-one of the twenty-three chapters are by American authors and this bias is highlighted in that much of the information is presenting data/views based on American research, which is misleading. For example, in the chapter on 'Protein—Polysaccharide Interactions' (Chapter 20) an unbalanced view is presented on the question of composition and functional (breadmaking) properties. I would also like to have seen a chapter devoted to carbohydrate analysis. Although this is covered to some extent in Chapter One, new techniques, such as mass detection with liquid chromatography or the use of short capillary columns for carbohydrate analysis, have not been mentioned.

All chapters are adequately referenced and although, as may be expected from such a wide coverage, they do not deal with their subject in depth, this book provides a very adequate starting point. The book is reasonably priced and is essential reading for all scientists and industrialists working in this field.

Stanley Z. Dziedzic

Developments in Dairy Chemistry, Vol. 1. Ed. by P. F. Fox. Barking, Essex: Applied Science, 1982. Pp. x+409. ISBN 0 85334 142 7. £44.00.

This book, the first in an advanced series to be published on dairy chemistry, is a welcome addition to books in the English language in this field and fills an important gap

in the literature of food science. This volume is concerned only with milk proteins and is the most informative book on the subject known to the reviewer.

Fourteen authors contribute twelve chapters on the structure of the milk proteins of bovine and other species of mammal, their biosynthesis, the enzymic and heat induced mechanisms of coagulation of milk, the gelation of sterilized milks in ageing, changes during storage of raw milk, nutritional aspects, manufacture of casein, caseinates and coprecipitates, isolation of whey proteins, and finally their functional properties and use as ingredients in food.

The colloidal stability of milk has always been a fascinating and industrially important problem and the authors give much information and many suggestive comments in this field. The reasons for, and mechanisms of, forewarming or preheating milk are of long standing importance for chemists in the dairy manufacturing industries and this book sheds much light on the problem.

Today there is no more important subject than the factors controlling the gelation of ultra high temperature treated (UHT) milk.

The author of Chapter 7 gives several facts and references but there seems to be no complete answer. The findings on the effect of temperature of storage are inconsistent, and why should manganous sulphate delay gelation? There seem to be many peculiar facts about this obviously complex phenomenon. The nature of the peculiar flavour of UHT milk has still to be finalized. It is unfortunate that such an ungrammatical term as UHT should have been adopted but I suppose it is too late to change it now. The expression HT-ST is used in different senses in different countries, a point to be watched.

The increasing interest in the whey proteins is a welcome trend in the dairy and food industries. It is not generally realized that whey protein is superior to bovine milk, human milk and egg protein in its content of essential amino acids. There is only a bare mention of some aspects of practical importance such as allergies, mastitis and colostrum. The numerous comments and suggestions made by the authors, who are recognized authorities in their respective fields, will stimulate the reader, although the book is definitely designed for reference rather than for the general reader. A study of all the facts presented in this book serves to emphasize what a complex material milk is, and why no two samples are ever exactly alike.

The book is as comprehensive and up to date as is practicable. A particularly useful feature is the large number of references which vary from fifty-four to 216 in each chapter, but the index could be more detailed in view of the enormous amount of information in the book. The full name of lesser known substances and properties should be given the first time initials are used because not all readers will be experts in this field. Although the book will be of most use to research workers in the subject, chemists in the dairy industry will find Chapters 5–8 of great interest, and Chapters 10–12 will be very useful to industrial chemists in the food industry.

The book is well produced, the type clear and the illustrations are explicit and appropriate. The high price may result in relatively few individual purchasers, but this is a book which should be available in every university, college, research institute and firm with an interest in dairy chemistry and, of course, in scientific libraries.

J. G. Davis

Advanced Sugar Chemistry: Principles of Sugar Stereochemistry. By Robert S. Shallenberger.
Chichester: Ellis Horwood, 1982. Pp. xxi+323. ISBN 0 85312 541 4. £31.50.

The opening chapter is concerned with introductory stereochemical material including definitions of sugars and the systematic nomenclature for carbohydrates and their derivatives. There are included numerous comments and examples which make the chapter very readable and easy to follow. Chapter 2, on the chiral nature of sugars, discusses the basic concepts of chirality and symmetry and develops these with regard to sugars. Chapters 3 and 4 are concerned with pseudosugars such as dihydroxyacetone, and the acyclic structures respectively, involving Fischer projections and establishing, through proof of structure, absolute configuration for the acyclic form. Chapter 5, on the ring forms, concentrates on Haworth structures for furanose and pyranose forms of aldo- and keto- pentoses and hexoses, and permissible chiral operations with these structures. Chapter 6, on conformational structure, discusses the puckered ring forms, the Reeves convention, chair, boat and skew conformations, envelopes and twists and the governing principles of multiple chirality. Chapter 7 shows how the optical rotatory power is a function of the sugar's stereochemistry and how this, through simple calculation, can be predicted. Chapter 8 is concerned with tautomeric composition, mutarotation, enolization and reversion products. Chapter 9 discusses the structure/linkage of oligosaccharides with relation to their optical rotatory power. The concluding chapter, Chapter 10, is concerned with sweetness. Until the early 1960s little was known of the structure/activity relationships involving sugar structure and taste—since then, mainly due to Professor Shallenberger's work, a theory has developed showing that, like optical rotation, the inherent characteristic of sweetness of a sugar molecule can be related to its stereochemical disposition. A synopsis of the theory is given in this chapter.

On the whole the book is well presented with many drawings to help understand the many difficult conceptual problems involved. I found the profuse listing of structures and their nomenclature extremely useful and illustrative. There are a number of references included and also a useful glossary of stereochemical terms. This book will be of great benefit to carbohydrate chemists and those students who find this 'esoteric subject' of sugar stereochemistry difficult to comprehend.

The title *Advanced Sugar Chemistry*, is slightly misleading for those people expecting synthetic sugar chemistry. The sub-title, *Principles of Sugar Stereochemistry,* is more definitive of the contents. The book is reasonably priced and is highly recommended.

Stanley Z. Dziedzic

Energy Management in Foodservice. By Nan and Kenneth Unklesbay.
Chichester: Ellis Horwood, 1982 (and Westport, Conn.: AVI, 1982). Pp. ix+437. ISBN 0 85312 526 0. £28.50.

The increasing cost of energy has generated much research and reams of literature concerned with the better use and management of this resource. This book has been written with the intention of providing a practical college text on the understanding and management of energy use in catering and is aimed at a readership which would include both students and practitioners of catering science.

The text is presented in five parts. The first of these deals with introductory and basic concepts such as energy sources and energy consumption in agriculture, food processing and distribution. The section also includes an overview of energy use in catering. Although a considerable amount of interesting matter is presented, much of this is concerned with the U.S. scenario and is, therefore, of reduced relevance to European readers. Part II presents the scientific and engineering aspects governing the generation and transfer of energy and sets out five basic energy managerial policies. The attempt to cover this large and important area in only fifty pages is perhaps overambitious, taking into account the intended readership. Understanding is not assisted by the use of non-S.I. units. The problems of achieving energy efficiency in processes such as chilling, freezing, thawing and heating of food are considered in Part III. Unit systems notwithstanding, this section contains much useful, practical information about ways of saving energy in the formulation, preparation, holding and service of food. In part IV indirect uses of energy in support areas such as air conditioning, sanitation and lighting are considered. In general this is an interesting section although the intended readership may well feel that the treatment is, on occasions, too detailed. The establishment of energy accounting systems and the implementation of conservation programmes is dealt with in Part V. Only nine pages are devoted to the subject of energy data collection and analysis—aspects which might be regarded as vital in a text book concerned with energy management and therefore worthy of more extensive treatment.

The book is well produced and is relatively free from typographical errors. Each chapter is well supported by up-to-date references to further reading. Diagrams are well drawn and whilst the majority of the tabulated information (which is perhaps the most valuable part of the book) is interesting, in a few cases more intelligent editing is indicated. It is expected that this book of 430+ pages with its wealth of data and at its relatively modest price will appeal to technologists with interests in the field of catering.

A. E. V. Lilly

Food Service Sanitation Handbook. By Gerald and Nancy E. Cohen. New Jersey: Hayden Book Co., 1982. Pp. viii+161. ISBN 0 8104 9478 7. £11.35 (softback).

This book is written for food industry and catering establishment operators to provide an informative, useful and understandable background in food hygiene. It is, however, specifically directed towards the food/catering industry in the U.S., providing in the last two chapters a guide to the health and sanitation codes and the role and operation of agencies which enforce the codes in the U.S.A. The substance of the book is, of course, of universal interest.

In many ways the book is successful in meeting its objectives, as it is written in an unsophisticated, direct and sometimes quaint manner, covering the many facets of hygiene at a fairly low level. Even so, it is doubtful whether food industry operators would be able to understand much of the science presented in the book.

There are many errors of fact, largely in the initial chapters relating to basic microbiology (Chapter 2), food-borne diseases (Chapter 3) and investigation of food-borne diseases (Chapter 4). Further, in an attempt to convey information in a form acceptable to the layman, the text is sometimes oversimplified to the extent of being inexact, inaccurate and misleading. This is a great pity, since there is much useful

information contained in the subsequent chapters which cover the topics of personal hygiene, food protection and quality control, rodent and insect control, construction and design of equipment and facilities, effective cleaning, and special food operations.

Of the many errors, it was noted with interest that wood is an acceptable cutting surface for foods, moulds make yoghurt, typhoid bacillus form spores, vibrio can cause syphilis, hot water dissolves fat, and that bacteria are so small that 25 000 can fit into an area of one square inch—the last is, of course, true! The latter point also highlights the use of Imperial units of measurement throughout the book. There is also one interesting photograph of a line of jacketed vessels—upside down!

In conclusion, the objectives of the authors are well-founded and no doubt a market does exist for such a book. It is tragic, however, that they did not seek guidance on the microbiological aspects; their attempts in this area make it difficult to recommend the book, even to the non-scientific readers for whom it is intended.

A. J. Reynolds

Brown Sugar and Health. By I. I. Brekhman and I. F. Nesterenko, translated by J. H. Appleby.
Oxford: Pergamon, 1983. Pp. v+ 96. ISBN 0 080268374. £10.00.

Professor Brekhman and his colleagues have developed the theory that there is something extraordinarily potent in brown sugar which is responsible for its health-giving properties. In his previous book, *Man and Biologically Active Substances* (BAS), the author drew attention to the pharmacology of health, or 'pharmacosanation', and the role of BAS in 'building up the inner ecology of the organism'. Whereas brown sugar has BAS, white sugar does not. This follow-up book of ten chapters and ninety-six pages explores these hypotheses further.

The book sets out the results of experiments designed to compare the effects of feeding white and brown sugar to rats. Professor Brekhman describes the tests as being rigorous because the animals received diets containing 50–90% sugar, and the experiments were continued for several months, and in some cases for the entire lifespan of the animal. His conclusions are that white sugar, when taken in amounts closely approximating those usually consumed by people, leads to premature ageing, and that rats which ate white sugar were less clean and mobile, showed less interest in the opposite sex, and lost their reproductive functions at an earlier stage.

In similar experiments with brown sugar, Professor Brekhman claims that this 'valuable' and 'health-promoting' product increases the animal's work capacity and resistance to stress, as well as eliminating many of the 'negative aspects' of white sugar.

The effects of brown sugar on man are described briefly in Chapter 10. The text here consists of only three and a half pages which describe the studies of two other groups of researchers. In the first study, it is stated that the replacement of 20 g of refined white sugar with brown sugar in the diet of young men adapting to mountainous conditions facilitated their more rapid adjustment to high altitude. In a second study, 237 people with abnormal blood chemistries were fed daily 70 g brown sugar in place of white sugar. After 4 months on the diet, the blood sugar, β-lipoproteins and cholesterol levels in the blood were found to be normal. Unfortunately in both the studies, insufficient information was provided by Professor Brekhman as to how he came to his conclusions. No other written work describing brown sugar's valuable effects on people is described.

On the basis of the animal and human studies available, the author states that after

struggling for 10 years to answer the question—'What is better—to give up sugar altogether or to eat brown sugar?'—he can now conclude emphatically that it is better for us to eat brown sugar throughout life.

In Chapter 5—'What we know about brown sugar?'—the reader may be disappointed to learn that, despite the author's investigations, it was not possible to identify a substance or group of substances which represent the chief active principle of brown sugar. Despite scouring the pages for any explanation of brown sugar's key to health, your reviewer could come up only with the phrase 'stimulating units of action' and the newly coined word 'staminator' (derived from stamina), both of which are used in Chapter 6 to describe the undefined 'principles' in the 'natural complex' in brown sugar.

In conclusion, your reviewer remains unconvinced that there is any nutritional or health advantage in consuming brown sugar in place of white sugar. It would have been useful if the book had given some descriptions of processing and specifications of the brown sugar(s) used in the animal and human studies, and had provided a more detailed account of the experiments and the rationale behind them.

Although the book is interesting and provocative, the result and conclusions must still be looked upon with some scepticism. Everyone is perfectly entitled to believe in what they will, but the author has not provided the reader with sufficient facts to support either the theory that brown sugar has beneficial effects in man or the validity of the experimental procedures.

David P. Richardson

Dietary Fibre. Ed. by G. G. Birch and K. J. Parker.
London: Applied Science, 1983. Pp. xi+304. ISBN 0 85334 178 8. £28.00.

The book is a compilation of sixteen papers presented to the 13th International Symposium held at the University of Reading in 1982. The papers cover the role of fibre in food product formulation, the effect of fibre on nutrient bioavailability, the nature and physiological effects of fibre and methods of analysis.

The whole subject of dietary fibre is undergoing rapid development and this book is welcome as an up-to-date review. It is aimed specifically at the food technologist but will also be useful to nutritionists, dietitians and physicians. The papers are also of value for the extensive and up-to-date references they contain.

The opening two chapters trace the development of the dietary fibre hypothesis over the past decade. In the wake of the recent huge popularity of the F plan diet there has inevitably been a backlash suggesting that dietary fibre inhibits the absorption of minerals like calcium and zinc. The book warns that, although there is only a marginal effect on mineral absorption, care needs to be taken in diets that are only just adequate in these nutrients. The section on the effect of fibre on energy-containing nutrients is also interesting in the light of recent claims.

Chapters on the effect of dietary fibre on bile acid metabolism, water holding capacity, and stool weight are detailed and probably better suited to the specialist researcher than the dietitian. But they are well worth the effort needed to read them if this is not your speciality. Similarly, the chapters on the chemical nature and methods of analysis of dietary fibre are somewhat esoteric, but useful to specific groups of workers. The chapter on 'Dietary Fibre as A Tool For the Clinician' takes the bold step taken by

so few others of recommending a target intake of fibre of 40 g a day. The one subject which is not covered in detail is the relationship (if any) between fibre and cancers. Brief mention is made in one or two places, but one might have expected that this would have a chapter of its own.

This book certainly contains something for everyone, no matter how much or how little one already knows about or deals with dietary fibre.

Jenny Salmon

Recent Advances in Food Irradiation. Ed. by P. S. Elias and A. J. Cohen. Amsterdam: Elsevier Biomedical Press, 1983. Pp. viii+361. ISBN 0 444 80499 4. US$76.50.

This important new book has chapter headings as follows:
'General Introduction' (Elias and Cohen), 'Advances in Radiation Chemistry of Food and Food Components—An Overview' (Basson), 'Commonality and Predictability of Radiolytic Products in Irradiated Meats' (Merritt and Taub), 'Chemiclearance Approach to Evaluation of Safety of Irradiated Fruits' (Basson *et al.*), 'Development of a Computer Program for "Steady State" Calculations—Model Fruit' (Ehlermann), 'Recent Advances in Radiation Chemistry of Lipids' (Delincee), 'Comparison of Chemical Consequences of Heat and Irradiation Treatment of Lipids' (Nawar), 'Recent Advances in Radiation Chemistry of Proteins' (Delincee), 'Recent Developments in Radiation Chemistry of Carbohydrates' (Adam), 'Chemical Consequences of Irradiation of Sub-Tropical Fruits' (Beyers *et al.*), 'Recent Advances in Radiation Chemistry of Vitamins' (Basson), 'Nutritional Aspects of Food Irradiation' (Murray), 'Microbiological Aspects of Food Irradiation' (Teufel), 'Toxicological Aspects of Food Irradiation' (Elias), 'Evaluation of the Irradiation of Animal Feedstuffs' (Conning), 'Absence of Induced Radioactivity in Irradiated Foods' (Becker), 'Estimated Radiation Chemical Changes in Irradiated Food' (Vas), 'Recent Advances in Irradiation Dosimetry and Processing Conditions' (Vas) and 'Future Prospects for Radiation Processing of Food' (Ehlermann).

The book is very much an up-dating of previous work and thus portrays the most recent advances in food irradiation. To this end the eminent authors are from many parts of the world but reflect, in particular, the great lead given by the Karlsruhe and South African workers.

Although of great interest to all food scientists and technologists interested in this expanding subject, it emphasizes chemical studies and data afforded by a number of model systems.

Chemical changes in food components are clearly so complicated that freshly emerging data are commonplace and too numerous to discuss in this review. However, an interesting example is that of sugars, well known to protect other fruit components from radiation damage, yet susceptible to breakdown into uloses and other carbonyl compounds, some of which are mutagenic. Evidently radiation is less severe than heat treatment in this respect.

One drawback of the book is the absence of any abstract heading for each chapter. This necessitates some delving and searching for information. However, the contents abound in succinct and valuable information and it is clear that prospects for radiation preservation of food are good. The book is entirely recommendable.

G. G. Birch

Demulsification: Industrial Applications (Surfactant Science Series Vol. 13). By K.J. Lissant.
New York: Marcel Dekker. 1983. Pp. vii + 162. ISBN 0 8247 1802 X. SFr100.

Ensuring long term stability is a problem with which all emulsion commodity formulators are familiar. How to achieve this goal is now well documented in many books and published papers, even though the precise nature of the mechanism involved is not fully understood. The reverse process of deemulsification is, however, far less well documented and it remains very much a matter of trial and error.
Deemulsification has been tackled very seriously by the petroleum industry because of its importance in oil recovery, and the techniques it has developed to solve this problem have been adopted by other industries. The author quotes a few examples where deemulsification is necessary in the food industry. For example, processing of olives to obtain olive oil leads to the formation of an undesirable emulsion when the olive paste is diluted with water, and the effluents from citrus processing plants may contain emulsified oil.

In order to develop deemulsification techniques it is necessary to understand the principles of emulsion stability. These are reviewed briefly by the author along with related topics such as emulsion types, nature of the oil–water interface at the drop surfaces, emulsifiers, drop coalescence and methods for testing emulsions. Two chapters then describe the deemulsification of oil-in-water and water-in-oil emulsions, respectively. These are followed by a review of the deemulsification of petroleum emulsions which details a large number of methods which have been utilized and gives many line diagrams of the equipment involved. The final chapter covers additional methods and areas of deemulsification.

The author adopts a very informal and critical style which suggests that the text reproduces verbatim material which has been presented on previous occasions in a series of lectures. The mathematical background to the various theories associated with emulsion stabilization and destabilization is avoided. Although the text will, consequently, be more readily understood by those less well versed in mathematics it inevitably means that the presentation of some facts is oversimplified with a resulting loss of precision and accuracy. For example, the author states (p. 80) that when butter is heated '. . . the viscosity will be reduced and most of the water will fall out'!

The author remarks in the preface that he has tried '. . . to show the breadth of the areas where deemulsification is of concern and to outline general approaches to the problem'. This he has achieved but there is no guarantee that the methods reviewed will ensure the solution of any deemulsification problem with which the reader is confronted. Indeed, as the author points out 'I wish that a nice, neat formula could be given for the solution of every deemulsification problem'.

Each chapter ends with a comprehensive list of references to papers, books and patents for further reference, the publication dates extending into 1981. In view of the time taken to publish a book, following submission of the manuscript, the literature coverage is very satisfactory.

The book is printed by the reproduction process now generally used by Dekker. Although it provides clarity of the printed word and illustrations it is less pleasing to the eye than the traditional way of printing.

P. Sherman

Developments in Food Proteins, Vol. 2. Ed. by B. J. F. Hudson.
London: Applied Science, 1983. Pp. x + 339. ISBN 0 85334 176 1. £36.00.

This is a series of related essays on topics in the protein area written by authors with long experience in their subjects. The technological significance of wheat proteins is dealt with in some detail, covering much recent research (over 200 references) but easily understandable by the non-specialist. Soy proteins are similarly treated ranging from the structure of the bean to its use in textured products. In view of the number of soy products on the market that appear to have impaired protein quality it is interesting to note the report that 'properly processed' soya has a PER close to that of casein, and to see the quality/heat processing curve, known for at least half a century. Consumers will be pleased to read of a 1981 paper stating that soy processing technology is now able to produce flatus free products.

Rape seed, a major source of oil, has not so far been used for its protein and the chapter devoted to this subject discusses improved varieties useful for animal feed, but unfortunately the concentration of thyroactive substances is still too high to allow its use for human food. The author does not believe that concentration or isolation of the protein along the lines used for soya is economically feasible.

Lupin seed protein first used in cereal products as long ago as 1919, is similarly discussed. Its potential depends more on economic than nutritional considerations.

Similar economic issues control the use of legume proteins. In a chapter on air classifications of field beans and peas it is pointed out that while the protein can be increased by up to 60%, the starch fractions do not have very useful properties in food technology.

One chapter goes into the animal field with a discussion of fish proteins, although it has never been made clear in such discussions why the fish cannot be consumed as such. The author contrasts the failure of fish protein concentrates (can that be wondered at when the price of the product exhibited at the 1970 International Congress of Food Science approached that of top quality beef?) with the acceptance of fish gel in Japan. The subject has been well researched over the years, with, as the author states, more failures than successes, but he deals here with newer approaches.

The last protein discussed is yeast; refining procedures bring it 'closer to use in human food'. Yeast proteins have limited functionality but can be improved by a variety of procedures.

In all these discussions of novel sources of protein over the years their nutritional value, quite separately from their functional properties, has been a major item (in these essays this is not so). They have mostly started their laboratory lives as potential human foods, intended to supplement diets which, it was later accepted, are not short of protein anyway. Another point of major importance is that measurements of the quality of any one protein source do not provide any information of the value of such foods when added to a diet. For example, it is likely that a protein completely lacking one essential amino acid, and so with all protein quality measurements at zero, yet a valuable supplement because it was very rich in the two most frequently limiting amino acids (lysine and the sulphur amino acids), would have been discarded at the very start.

One of the major uses of protein quality measurements is probably in control of processing but the vast expense of animal methods precludes their use in any possible routine control systems. So the final paper on estimation of protein quality with its

emphasis on relatively brief indices based on amino acid profiles and *in vitro* methods rounds off the series of essays rather nicely.

Overall a well produced book useful to those whose interests lie in this area.

A. E. Bender

Food Texture and Viscosity: Concept and Measurement. By M. C. Bourne. New York: Academic Press, 1982. Pp. xii + 325. ISBN 0 12 119060 9. US$36.00.

The opening chapter covers the importance of textural properties, definitions of texture, texture related terms, an introduction to rheology and a history of some of the early rheological work.

Chapter 2 deals with body–texture interactions. It provides a comprehensive study of mastication processes, with some interesting quantitative data on chewing efficiency, chewing rates, saliva production and composition, maximum forces exerted between teeth (including dentures) and average forces exerted when chewing different foods. This offered part of the explanation as to why I broke my denture on a Yugoslavian bread roll!

Principles of objective texture measurement are discussed in Chapter 3. Fundamental tests, which measure well defined rheological properties, are briefly covered, but most of the space is devoted to empirical tests, particularly imitative tests. These empirical and imitative tests are classified according to the principle of the test rather than the type of food. Such principles include the measurement of force, distance, time, energy, or combinations of these. In the force section, puncture testing, compression extrusion tests, shear tests, tensile tests and torsion methods are described. The different types of distance measuring instruments are then discussed, along with time aspects of deformation. Finally, multiple measurement techniques which have the ability to measure several variables under controlled conditions are described. A section on units of measurement appears in the middle of the chapter; these are mainly S.I., but occasionally c.g.s. Table 11 contains reference to several units, named after eminent scientists, all beginning with a small letter, e.g. watt, joule, pascal, newton and hertz.

Chapter 4 deals in considerable detail with the practical aspects of objective texture measurement. Puncture testers are given reasonable prominence. Experimental data, linking instrumental readings with firmness and ripeness, are given for apples and several other fruit. 'Motorized' puncture testers are mentioned briefly, such as the Bloom Gelometer, the Stevens LFRA texture analyser, the Marine Colloids Gel Tester, the Maturometer, the Christel texture meter and the Armour Tenderometer. Also described are shear testing equipment, the Farinograph and a number of lesser known (to me at least) instruments with such interesting names as the Bostwick Consistometer, the Hilker–Guthrie Plummet, a Ridgelmeter, a Haugh meter, the Adams Consistometer, a TUC Cream Corn Meter and a Succulometer; excellent photographs are supplied of most of this equipment. In the multiple measuring instruments the General Foods Texturometer is described, along with a wide range of food applications, summarized in tabular form. Information on the Food Technology Corporation Test System (55) the Instron (76) and the Ottowa Measuring System (46) is presented in similar fashion (numbers in parentheses represent the number of food applications mentoned).

Chapter 5 covers viscosity and consistency. Laminar and turbulent flow are discussed; viscosity values are given for common substances and sugar solutions; interesting values for shear rates that fluids are subjected to are given for, in the mouth, flow over inclined surfaces, pouring and mixing operations. Time dependent and independent non-Newtonian fluids are discussed. Methods for measuring viscosity such as capillary flow techniques, falling ball methods and rotational viscometers are well described. Both S.I. and c.g.s. units (i.e. centipoise and centistoke) are used throughout this chapter. However, the author suddenly starts abbreviating second as sec. rather than s, which is preferred in the earlier chapters.

Chapter 6 on sensory methods of texture and viscosity emphasizes the importance of sensory judgements of quality and states that sensory methods are the ultimate method of calibrating instrumental methods of texture measurement. Sensory texture profiling is described under the following headings; selection of panel members, training of panel, establishing standard rating scales. Examples of reference materials are given for establishing scales for hardness, fracturability, chewiness, gumminess, adhesiveness and viscosity. Development of score sheets and comparative texture profiles are also discussed, and useful practical examples are given. The author makes a strong case that well conducted texture profile techniques are objective tests: that is they are free from personal bias and results from different panels are reproducible to a high degree. Finally the types of correlation between subjective perceptions and objective measurements are discussed; measured power law exponents are given for a wide variety of stimulus and their respective sensory response.

The concluding chapter gives guidelines for selection of a suitable test procedure and the factors that require consideration before commencing experimental work are reviewed.

There is a useful appendix on suppliers of texture and viscosity measuring instruments. All cited references are collected at the end of the book, and a 'Suggestions for Further Reading' section appears at the end of the introductory chapter. A subject index is provided; however, no foods are mentioned in this. The book is easy to read and well presented. The approach tends to be descriptive and analytical rather than mathematical, but direction is provided to more advanced treatments. I am convinced that both students and experienced rheologists will find this book extremely valuable.

M. J. Lewis

Sensory Quality in Foods and Beverages: Definition, Measurement and Control.
Ed. by A. A. Williams and R. K. Atkin.
Chichester: Ellis Horwood, 1983. Pp. 488. ISBN 0 85312 480 9. £22.50.

As a record of the proceedings of the Symposium organized jointly by the Society of Chemical Industry's Food Group (Sensory Panel) and Long Ashton Research Station in April 1982 this volume should more than suffice for those who attended. Whether the contents will be equally acceptable to a wider readership is questionable. The book contains the thirty papers presented at the Symposium and is unusual in allocating space to fifteen posters that were on display. Many of these have been written up in the form of short research communications and detailed experimental procedures. The inclusion of one poster which would be better described as a commercial technical data sheet is surprising and unnecessary.

A wide range of subject matter has been covered and the editors have done well to find a title that embraces the majority of the contents. The book is arranged in five sections. The first of these on understanding and defining sensory quality consists of four papers but only two address themselves to this particular topic. The second and third sections relate to measurement and sensory quality, one being devoted to sensory and the other to instrumental methods. The fourth section, by far the largest, covers the application of sensory and instrumental methods to specific products. The book concludes with a section on the influence of sensory quality on food choice and intake. There are only three papers in this section but the review on sensory qualities, palatability of food and overweight by J. E. R. Frijters and the paper on marketing and sensory quality by D. Lesser are in the reviewer's opinion two of the better contributions to the book. In general the quality of the papers and their scientific content are ordinary but a noteworthy exception is that due to D. M. H. Thomson and H. J. H. Macfie which poses the question 'Is there an alternative to descriptive sensory assessment?' and then proceeds in a very original and elegant way to suggest that there might be.

With few exceptions the papers provide adequate literature coverage and the authors have ensured that recent references are included. Food scientists and technologists and others associated with the quality of food will find something of interest in this volume which could provide a source for further reading. The book has been produced from camera-ready typescript supplied by the authors and is remarkably free from typographical errors although alterations to the typescript have in places led to smudged typeface. The legend to Fig. 1, p. 301 contains a double entry. An adequate index is included but more useful are the summaries which introduce each paper.

D. McHale

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 (three complete copies) should be sent to the Editor, *Journal of Food Technology*, 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 1½ 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. Authors should consult the current issue in order to ensure that their manuscript conforms to the Journal conventions on such things as subheadings, layout of tables, etc.

References. Only papers closely related to the author's work should be included; exhaustive lists should be avoided. References in the text 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 paper; (d) the title of the journal given in full and not abbreviated, set in italics (underlined once in typescript); (e) the volume number; (f) the first and last page numbers of the paper—e.g.

Steiner, E. H. (1966). Sequential procedures for triangular and paired comparison tasting tests. *Journal of Food Technology*, **1**, 41–53.

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) place of publication and publisher—e.g.

Lawrie, R. A. (1979). *Meat Science*, 3rd edition. Oxford: Pergamon Press.

In the case of edited multi-author monographs, the editor(s) should be indicated in parentheses after the book title—e.g.

Hawthorn, J. (1980). Scientific basis of food control. In *Food Control in Action* (edited by P. O. Dennis, J. R. Blanchfield & A. G. Ward). Pp. 17–33. Barking, Essex: Applied Science.

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		

Chemical formulae and solutions. The correct molecular formula should be used indicating whether the substance is anhydrous or hydrated. Solutions should be given in terms of molarity. The arithmetic basis of other expressions of concentration must be clearly defined.

Species names. In addition to common or trivial names of plants, animals or microorganisms, the species should normally also be indicated by use of the latin binomial at least once. This should be given in full and italicized (underlined once in typescript). In subsequent use, the generic name may be abbreviated to a single letter provided that this does not result in ambiguity.

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.

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