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# Approaches to the effective utilization of *Haplochromis* spp. from Lake Victoria. II. Production and utilization of dried, salted minced fish cakes

C. M. DHATEMWA*, S. W. HANSON† AND M. J. KNOWLES†‡

## Summary

The yields and composition of *Haplochromis* mince prepared by passing whole fish or 'nobbed' (beheaded and eviscerated) fish through a flesh–bone separator were investigated. Salted minced fish cakes were prepared using a dry salt to minced fish mixture (40 : 100). The dried cakes were assessed chemically, microbiologically and organoleptically over a 14 week period stored at an ambient temperature of 20–25°C. Reconstitution was effected by desalting in boiling water. The technique developed offers a viable alternative method for processing and utilization of small, bony fish species which are difficult to process by traditional methods.

## Introduction

Catches of *Haplochromis* spp. from Lake Victoria are likely to increase substantially over the next few years, necessitating the development of methods for their effective utilization as food for direct human consumption (CIFA, 1982). The chemical composition and distribution of nutrients in *Haplochromis* have been investigated, providing a basis for evaluating the nutritional implications of processing methods (Ssali, Hanson & Knowles, 1984).

The small size of *Haplochromis* renders it impractical to employ hand gutting. At the present time the main method of processing is sun drying of the whole fish. The dried *Haplochromis* are then usually placed on sticks which are linked together in parallel strings for sale as a 'mat of fish'. Such products are not as popular as larger species of dried fish and it is envisaged that as catches of *Haplochromis* increase, additional methods for their effective utilization will have to be found.

Some other methods of processing *Haplochromis* for direct human consumption have been investigated. Canning has not proved viable owing to the non-uniformity with respect to colour and size of the many species present in any typical catch and the prolonged retorting times needed in order to soften the bones sufficiently (Beatty, 1964). Mechanical drying methods using a diesel fired tunnel drier have also been tried (Beatty, 1964), however, it is doubtful whether fuel prices make this method an attractive proposition. Jiwani & Dhatemwa (1972) produced a fish powder from gutted, beheaded and smoked fish, which was of superior quality to the traditional *Haplochromis* fish powder produced from ground sun dried whole fish. However, the process-

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ing method, involving hand gutting, beheading, smoking, hammer milling and sieving, proved too labour intensive and the price of the final product was uneconomic.

Therefore, in view of the difficulties encountered with the above methods, and as there are inadequate refrigeration facilities for the storage of fresh fish and distribution to outlying areas, alternative processing methods need to be developed. Production of salted minced cakes from *Haplochromis* offers one possible method of utilizing the whole catch without distinction of size, species or colour.

## Materials and methods

*Fish samples*

Four batches of *Haplochromis* were caught by bottom trawling (depth 25–45 m) from the Kitubulu–Nsazi inshore fishing ground of Lake Victoria during October –December 1980 and February 1981. The fish were frozen, packed in insulated boxes and flown to the U.K. They were kept in cold store (−25°C) until processed. (Some of the analytical work described in the previous paper (Ssali *et al.*, 1984) was carried out on the same October, November and February batches.) On arrival the quality of each batch was assessed and found to be acceptable organoleptically (neutral odours, no belly burst) and microbiologically—i.e. within acceptable I.C.M.S.F. (International Commission on Microbiological Specification for Foods) limits for total counts (Nickelson *et al.*, 1980).

*Production of minced fish*

Thawed, washed, whole *Haplochromis* were fed into a Baader 694 flesh–bone separator, using a standard drum with 3 mm diameter orifices. The mince was washed 3 times in cold tapwater in the ratio 2 : 1 (water : mince, by weight) in order to separate and remove visceral material. The excess water was drained off through a cheesecloth, using a technique similar to that described by Tableros & Young (1981) for washing Mexican shrimp by-catch.

A comparative experiment was carried out in which thawed washed *Haplochromis* were 'nobbed' (beheaded and eviscerated) and fish mince obtained directly by passing the nobbed fish through the flesh–bone separator without further washing stages.

*Salting of mince and production of minced fish cakes*

Washed mince was mixed thoroughly with food grade salt in various concentrations (5–60% expressed as percentage proportions of the weight of mince). The mixture was allowed to stand for 15 min and the excess liquor was expressed through a cheesecloth. Portions of salted mince were moulded into minced fish cakes (about 100 g) using a simple hamburger press (10 cm diameter). This procedure was a modification of that developed by Del Valle (1974). The salted cakes were placed on plastic screens and dried in circulating air at 40°C in a Mini-Torry kiln for 40 hr, in order to simulate as far as possible natural sun drying.

*Storage of minced fish cakes*

Samples of the cakes were randomly divided into three lots and stored under ambient conditions (20–25°C) for 14 weeks. Lot UN (denoting unpackaged) were stored without any packaging material. Lot LDP (denoting low density polythene) were individually packed in LDP bags (35.5 g/m$^2$), without excluding air, and lot VP (denoting vacuum pack) were individually sealed in vacuum pouches (cellulose MXDT/

A) coated on outside only with polyvinylidene 35 g/m², laminated to low density polyethylene 46 g/m².

*Desalting procedure*

Fish cakes were desalted by soaking in tapwater (ratio of 1:10, fish weight: water volume) for 30 min. An equal volume of fresh water was then used to boil the cakes for 15 min (Young *et al.*, 1979).

*Proximate analysis*

The methods used for moisture, protein, lipid and ash are as given in the previous paper (Ssali *et al.*, 1984). Salt was determined on ashed samples by titration with 0.1 M silver nitrate solution using potassium chromate as indicator, or by using a Corning Eel chloride meter standardized against the titration method.

*Microbiological determinations*

Total Viable Counts (TVC) and *Staphylococci* were determined as described by Del Valle (1974).

*Sensory panel assessment*

The desalted boiled samples were served in shallow dishes to a trained taste panel of four judges. Samples were evaluated on a 1–5 score range for colour (degree of brown-ness), odour, fish taste, rancid taste, and texture (mouth feel: soft to granular). Water was used to clear carryover taints between samples.

## Results and discussion

*Minced fish*

Preliminary work on several species of fish including mackerel (*Scomber scombrus*), haddock (*Melanogrammus aeglefinus*), and sprat (*Sprattus sprattus*) had shown that higher yields of minced fish were obtained using a 3 mm orifice drum rather than a 5 mm orifice drum on the Baader 694 flesh–bone separator. Additionally, bone and skin content were found to be lower in mince produced using the smaller orifice drum (Dhatemwa, 1981). Similar results were obtained with *Haplochromis*. With the 5 mm orifice drum an average yield of 63.5% of mince was obtained compared with an average yield of 69.1% using the 3 mm orifice drum. The 3 mm orifice drum was therefore used for all the subsequent experimental work.

Table 1 gives the yields of unwashed mince from four batches of *Haplochromis* and the corresponding yields after washing the mince samples 3 times with water. Washing reduced the average mince yield from 69.1 to 37.3% (49.3 to 21.4% d.w.b.). A comparative experiment using nobbed fish as raw material produced an average yield of

**Table 1.** Mince yields (as % of whole fish weight) for four batches of *Haplochromis*

| | October batch | November batch | December batch | February batch | Average* |
|---|---|---|---|---|---|
| Unwashed mince | 68.1 | 70.9 | 67.5 | 69.7 | 69.1 (49.3) |
| Washed mince | 40.2 | 35.5 | 39.2 | 34.2 | 37.3 (21.4) |

*Average mince yields on a dry weight basis (d.w.b.) are given in parentheses.

unwashed mince of 37.3% as a percentage of whole fish weight (37.7% d.w.b.). This mince did not require washing. Although nobbed fish gave higher yields when considered on a dry weight basis, the method was discarded as being too slow in terms of fish preparation.

Table 2 indicates how the washing process affected the chemical composition and microbiological quality of the minced fish. Washing reduced the microbial load of the minced fish by 4-fold for one sample and 14-fold for a second, whilst at the same time considerably lightening the colour of the mince as a result of removal of water soluble blood and gut pigments. Washing the mince increased water content, thus reducing the percentages of crude protein, lipid and ash on a wet weight basis. Over 50% of the crude protein is lost in the washing process, although subsequent work has shown that losses of true protein are less as washing removes a higher proportion of non-protein nitrogenous compounds (Bigueras, 1983). Lipid and ash losses during washing are about 70 and 65%, respectively, thus considerably reducing the potential of the mince for rancidity development.

**Table 2.** Composition and total viable counts for mince samples from two batches of *Haplochromis**

| | December batch | | February batch | |
|---|---|---|---|---|
| | Unwashed | Washed | Unwashed | Washed |
| % moisture | 80.1 (1.4) | 85.2 (1.0) | 80.3 (3.3) | 83.8 (0.2) |
| % crude protein | 14.4 (0.5) | 11.1 (0.4) | 15.3 (0.2) | 11.6 (0.3) |
| % lipid | 5.6 (0.1) | 3.1 (0.2) | 5.2 (0.1) | 3.2 (0.1) |
| % ash | 1.3 (0.1) | 0.8 (0.1) | 1.2 (0.0) | 0.8 (0.1) |
| TVC/g (25° for 48 hr) | $1.8 \times 10^5$ | $1.3 \times 10^4$ | $6.2 \times 10^5$ | $1.7 \times 10^5$ |

*Percentages are the means of four determinations: standard deviations are given in parentheses.

*Salted minced fish cakes*

The salted cakes were found to change from a gelatinous mass to a firm coherent cake as the salt : fish ratio increased (Table 3). For salting ratios up to 20%, no liquor was lost from the salted mince, which gelatinized and retained its water holding capacity. As the salt ratio increased further, fluid losses occurred and the volume of liquor released from the salted mince increased up to an approximately 40% salting ratio. Subsequent salt increases above this salting ratio gave only marginal increases in liquor loss (see Fig. 1). The minced fish cakes produced from 40% salting ratio were coherent and firm and did not disintegrate on desalting. A salt ratio of 40% was therefore chosen as the concentration for cake formation from *Haplochromis*. This is within the range of optimum salt ratios found by Del Valle & Gonzalez–Inigo (1968), although other workers (Young *et al.*, 1979; Poulter & Poulter, 1984), have shown that lower levels of salt incorporation (10–20%) may be used with other fish species and normally in conjunction with more sophisticated drying regimes—i.e. an initial high temperature–short time drying period (e.g. 2 hr at 70°C, or 1 hr at 100°C), prior to sun-drying or controlled low temperature drying at 40°C.

The chemical composition data for the undried salted *Haplochromis* cakes (Table 4) illustrates that using an initial salt : fish mixture of 40 : 100 produces an undried salted cake with minimum moisture content and highest crude protein when calculated on the salt-free fraction.

**Table 3.** Description of salted *Haplochromis* mince*

| Salt as % of mince | Pressed mince | 100 g mince cakes (undried) | Cakes after drying for 40 hr at 40°C |
|---|---|---|---|
| 10% | Gelatinous, no fluid exuded | Gelatinous | Brown pliable, no visible salt particles |
| 20% | Gelatinous, no fluid exuded | Gelatinous | White cakes with rough striated surfaces, pliable |
| 30% | A bit gelatinous, crumbly, fluid exuded | Crumbly cake lacking consistency | White porous structure, lacking binding |
| 40% | No gelatinization, consistency of mince, fluid exuded | Consistent cake | White consistent with some salt particles on surface. |
| 50% | No gelatinization, excessive salt particles, fluid exuded | Consistent cake, but with excessive salt particles | White cakes showing consistency but with excessive salt particles |
| 60% | No gelatinization, excessive salt particles, fluid exuded | Consistent cake, but with excessive salt particles | White cakes showing consistency but with excessive salt particles |
| 70% | No gelatinization, excessive salt particles, fluid exuded | Consistent cake, but with excessive salt particles | White cakes showing consistency but with excessive salt particles |

*Five hundred grams of washed mince used for each salt concentration and three 100 g cakes made from each lot.

Salted cakes all made from an original salt:fish mixture (40:100) were dried in a Mini-Torry kiln for 40 hr at 40°C; the average chemical composition after drying was 17% moisture, 6% lipid, 26% protein and 49% salt.

*Storage trials*

The mean sensory panel scores awarded to the three lots of desalted samples (UN, LDP and VP) are summarized in Table 5. Only rancid taste appeared to increase

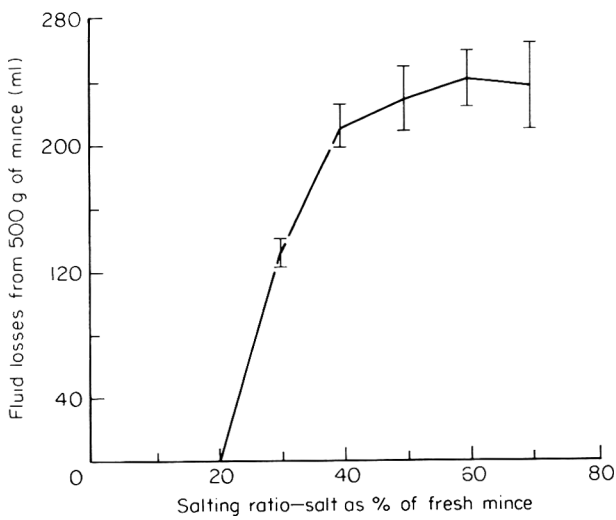


**Figure 1.** Fluid losses during salting of *Haplochromis* mince at different salting ratios. Mean values are plotted with vertical lines representing ± 1 s.d.

**Table 4.** Composition of undried salted *Haplochromis* cakes in different salt concentrations

| Salt as % of fresh mince | % moisture* | | % crude protein* | | % Salt in mince* |
|---|---|---|---|---|---|
| | Whole weight | Salt-free | Whole weight | Salt-free | |
| Unwashed mince | 80.4 (2.0) | 80.6 | 14.7 (0.27) | 14.7 | 0.20 (0) |
| Washed mince | | | | | |
| 0% | 83.4 (2.0) | 83.6 | 12.4 (0.25) | 12.4 | 0.20 (0) |
| 5% | 79.1 (1.6) | 82.7 | 11.8 (0.55) | 12.3 | 4.3 (0.37) |
| 10% | 75.8 (0.10) | 83.3 | 11.3 (0.17) | 12.4 | 9.0 (0.36) |
| 25% | 66.4 (0.10) | 80.4 | 10.6 (0.10) | 12.8 | 17.4 (0.12) |
| 30% | 58.4 (4.2) | 75.5 | 13.2 (0.2) | 17.1 | 22.6 (0.71) |
| 40% | 46.3 (2.50) | 64.8 | 15.2 (0.18) | 21.3 | 28.6 (2.26) |
| 50% | 41.5 (1.3) | 67.2 | 12.5 (0.32) | 20.2 | 38.2 (0.35) |
| 65% | 36.7 (1.5) | 68.5 | 11.3 (0.60) | 21.1 | 46.4 (0.33) |

*Percentages are the means of three determinations: standard deviations are given in parentheses.

significantly with storage time. Colour, odour, fish taste and texture remained relatively stable over the 14 week storage period. The vacuum packed samples tended to be less brown and have lower rancid taste scores than the other samples.

During the storage trial the UN cakes became harder with storage, whereas the VP and LDP cakes were relatively softer, and this was reflected in the different moisture contents. At the end of the 14 week trial the moisture level of the UN cakes had reduced to about 4%, whereas for the LDP and VP cakes it remained fairly similar to the initial level of 17%.

Microbiologically the salted minced fish cakes were stable and remained within acceptable I.C.M.S.F. limits for total counts (Nickelson *et al.*, 1980). Over the whole storage period, microbiological counts tended to decrease with storage time (Table 6). The presence of *Staphyllococcus* would emphasize the need for hygienic handling and immediate cooking of the fish cakes after desalting.

## Conclusion

Flesh–bone separation and the subsequent production of dried salted mince cakes offers a viable alternative technique for the utilization of whole small bony fish such as *Haplochromis* spp.

Salted minced fish cakes prepared from a salt : minced fish mixture (40 : 100) were found to be stable, except for a small increase in rancid taste, over a 14 week period, which would allow adequate time for the distribution of products even at warm ambient temperatures (20–25°C).

Further work on the process is continuing. Some aspects that are being considered include the use of high temperature–short time drying periods prior to low temperature dehydration of minced fish cakes containing lower levels of salt addition.

**Table 5.** Sensory panel scores for salted *Haplochromis* cakes*

| Storage time (weeks) | Colour | | | Odour | | | Fish taste | | | Rancid taste | | | Texture | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | UN | LDP | VP | UN | LDP | VP | UN | LDP | VP | UN | LDP | VP | UN | LDP | VP |
| 1 | 2.50 | 3.00 | 3.25 | 2.75 | 2.25 | 2.75 | 2.50 | 1.50 | 2.75 | 1.50 | 1.25 | 1.50 | 2.25 | 2.00 | 2.75 |
| 2 | 4.00 | 3.75 | 2.50 | 2.50 | 3.00 | 2.75 | 2.25 | 3.00 | 2.75 | 1.50 | 2.00 | 1.75 | 3.25 | 3.75 | 3.25 |
| 3 | 3.25 | 4.00 | 2.25 | 3.00 | 3.00 | 2.75 | 2.50 | 1.75 | 2.75 | 2.25 | 3.25 | 1.50 | 3.00 | 3.75 | 2.75 |
| 4 | 3.00 | 4.25 | 2.50 | 2.75 | 3.00 | 3.50 | 2.25 | 2.00 | 2.75 | 2.75 | 3.25 | 2.00 | 3.50 | 3.50 | 3.50 |
| 5 | 3.75 | 3.75 | 3.50 | 2.25 | 3.25 | 2.25 | 2.25 | 3.25 | 2.75 | 3.00 | 3.25 | 2.75 | 3.50 | 3.50 | 3.25 |
| 6 | 3.75 | 4.50 | 2.75 | 2.25 | 2.50 | 2.50 | 1.75 | 2.25 | 2.50 | 2.75 | 3.50 | 2.25 | 3.25 | 3.75 | 3.50 |
| 8 | 3.00 | 4.00 | 2.33 | 2.33 | 3.33 | 2.00 | 2.67 | 4.00 | 3.00 | 2.67 | 3.67 | 2.33 | 3.33 | 3.33 | 3.33 |
| 9 | 4.00 | 3.50 | 2.75 | 2.50 | 2.00 | 2.25 | 3.25 | 3.25 | 2.00 | 3.75 | 3.75 | 2.25 | 3.75 | 3.50 | 3.50 |
| 10 | 4.25 | 3.25 | 2.25 | 3.50 | 2.50 | 2.25 | 2.25 | 3.00 | 3.25 | 3.25 | 3.50 | 2.75 | 3.25 | 3.50 | 3.50 |
| 11 | 4.25 | 4.75 | 2.75 | 3.50 | 3.50 | 2.75 | 2.75 | 2.25 | 3.25 | 3.33 | 3.00 | 3.25 | 4.50 | 4.25 | 4.00 |
| 13 | 2.00 | 4.50 | 3.75 | 2.75 | 3.75 | 2.25 | 2.75 | 2.00 | 2.50 | 3.00 | 4.25 | 3.75 | 3.00 | 4.00 | 3.75 |
| 14 | 4.25 | 4.00 | 3.50 | 3.00 | 3.50 | 3.00 | 2.75 | 2.25 | 2.75 | 3.25 | 3.75 | 3.25 | 3.00 | 3.50 | 3.25 |
| Grand mean | 3.5 | 3.9 | 2.8 | 2.8 | 3.0 | 2.6 | 2.5 | 2.5 | 2.8 | 2.8 | 3.4 | 2.4 | 3.3 | 3.5 | 3.4 |

*Score ranges: colour, 1 = light brown to 5 = dark brown; odour, 1 = no odour to 5 = strong odour; fish taste, 1 = no fish taste to 5 = very strong fish taste; rancid taste, 1 = not rancid to 5 = very rancid; texture, 1 = soft to 5 = granular. The values given are the means of the scores for four judges.

**Table 6.** Microbiological content of stored salted *Haplochromis* cakes

| Storage time (weeks) | Colony forming units/g of salted cake | | | | | |
|---|---|---|---|---|---|---|
| | UN | | LDP | | VP | |
| | TVC | Staphs | TVC | Staphs | TVC | Staphs |
| 1 | $6.3\times10^{4}$ | $5.9\times10^{3}$ | $4\times10^{3}$ | $3.8\times10^{3}$ | $8.0\times10^{4}$ | $3.3\times10^{3}$ |
| 2 | $3.1\times10^{3}$ | $3.5\times10^{3}$ | + | + | $1.6\times10^{4}$ | $4.0\times10^{3}$ |
| 3 | $1.8\times10^{4}$ | $3.0\times10^{4}$ | + | NG | $4.0\times10^{3}$ | + |
| 4 | + | NG | + | NG | $2.6\times10^{4}$ | NG |
| 5 | $3.1\times10^{3}$ | NG | + | NG | + | + |
| 6 | $5.1\times10^{3}$ | + | + | NG | + | NG |
| 8 | + | NG | NG | NG | NG | NG |
| 9 | + | NG | + | NG | + | NG |
| 10 | + | NG | + | + | + | + |
| 11 | + | NG | NG | NG | + | NG |
| 13 | + | NG | NG | NG | + | NG |
| 14 | + | NG | + | NG | NG | NG |

NG = no growth. + = growth, but colonies less than thirty per plate.

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# Effect of purging on quality changes of ice-chilled freshwater prawn, *Macrobrachium rosenbergii**

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## Summary

Post-harvest purging of freshwater prawn by partly clearing the animal's stomach and guts has been considered as a quality improvement measure before delivery to the market. Duplicate experiments of 18 hr overnight purging of freshwater prawns were conducted to determine the effect of purging on the animal's muscle pH value, ammonia content, soluble/insoluble collagen ratio, texture profile (peak height/plateau height ratio) and their overall appearance. Experimental results showed no significant difference ($P \leq 0.05$) between the purged prawns and the control in their muscle pH values, ammonia content, soluble/insoluble collagen ratios, and peak height/plateau height ratios. However, with both control and purged prawns stored on ice, the decrease in peak height/plateau height, increase in pH, ammonia content and the soluble/insoluble collagen ratio with ice-chilling time were highly significant ($P \leq 0.01$). Purging helped to improve the appearance slightly.

## Introduction

The eating quality of freshwater prawn (*Macrobrachium rosenbergii*) commercially produced in Hawaii can be maintained for only about 3 days under ice-chilled condition. After that period, the cooked abdominal tissues become mushy, which is a serious marketing problem. The cause or causes of mushiness are not fully understood. It has been postulated that the mushiness might be due to the effect of a collagenolytic enzyme acting on the prawn tissues during ice-chilling or refrigeration (Baranowski, Nip & Moy, 1984; Nip, Lan & Moy, 1983). This textural change was not recognized by Angel *et al.* (1981) or only casually investigated (Waters & Hale, 1981).

One of the prevailing methods used by the prawn industry to extend the shelf life and improve the quality and texture of prawns is purging. Prawns are held overnight without feed in clean circulating water. This allows the animals time to 'purge' or clean their gut of food, thus improving the general eating quality. In addition, this method allows prawns to clean themselves of silt which becomes attached to their gills during harvesting (Lee, 1979). However, data on the effect of purging on the quality of prawns are not available. The purpose of this study was to determine the effects of purging on some chemical, physical and visual characteristics of ice-chilled prawns.

## Materials and methods

### *Raw materials*

Live prawns (weighing 50–60 g per animal) were obtained from a local producer.

*Contribution from Hawaii Institute of Tropical Agriculture and Human Resources, Jour. Series No. 2620.

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*Purging*

About sixty animals were placed in a 110 litre round-bottom tank (diameter 76 cm, depth 60 cm) with non-chlorinated, running tap water at about 22°C overflowing for 18 hr at a rate of 300 l/hr, after which surviving prawns were killed in an ice-water slurry, packed with shredded ice in sealed insulated containers, and stored in a walk-in refrigerator at 5°C for 7 days. Preliminary experiments indicated this flow discharge system could maintain the water much cleaner than bubbling air into the tank or recirculating the water in the tank (an open system) even though the latter two procedures also maintain similar dissolved oxygen (about 5 ppm) in the system to keep the prawns alive and active over 18 hr.

*Non-purged (control) prawns*

Non-purged prawns from the same delivery were killed in an ice slurry and stored as described above.

*Sampling schedule*

Prawn samples were removed from the insulated containers for various analyses at the end of 0, 3, 5 and 7 days. The experiment was duplicated using another delivery of live prawns.

*Chemical analyses*

The pHs of prawn samples were measured by means of an Orion surface pH electrode connected to an Orion 407A meter (Orion, Inc., Cambridge, Massachusetts, U.S.A.). Five animals were used for each sampling with each animal tested at three locations of its tail. Any significant increase in pH is an indirect indication of muscle (protein) degradation due to the production of alkaline substances such as ammonia and other amines.

The ammonia content of each animal was measured with an Orion ammonia-specific ion electrode connected to an Orion 901 meter according to the procedure of Ward, Finne & Nickelson (1979) with slight modification. Five grams of raw prawn tissue was blended with 50 ml distilled water and 10 ml of this slurry was diluted with 50 ml of distilled water for ammonia determination. Five animals were used for each sampling. Any significant increase in ammonia content is an indication of deterioration in the prawn tissue.

The soluble/insoluble collagen ratio was calculated as the ratio of collagen content in raw prawn tail (first abdominal segment) extracted with distilled water at 77°C for 3 hr and the residual collagen content (Nip & Moy, 1981). The first abdominal sections of four animals were analysed separately at each sampling. The increase in this ratio is an indication of solubilization of the collagen which is the main component of the connective tissue.

*Physical analysis*

The texture of cooked prawn abdominal tissue (tail) was measured with a PEP texture tester (PEP Co., Houston, Texas, U.S.A.) according to established procedure (Nip & Moy, 1979) with a slight modification. Only the first abdominal segment of the tail (closest to the head) was used to calculate the shear force to push through the shear cell (peak height/plateau height ratio). Five animals were used for each sampling.

*Statistical analyses*

Data from chemical and physical measurements were analysed for differences between the purged group and control using analysis of variance. Differences between holding times were analysed by Duncan's new multiple range test (Steel & Torrie, 1960).

*Visual observation*

Cooked prawn abdominal tissues used for textural measurement were also used for visual observation of mushiness, presence of black vein and total integrity by the three authors. The heads of these animals were also used for the examination of presence or absence of feed in the stomach. Mushiness is defined as the flaking of the cooked prawn abdominal tissue, especially in the first abdominal section, and integrity means the wholeness of the peeled, cooked prawn tail. A total of forty animals for each group were examined.

## Results and discussion

*Chemical analyses*

Results of the pH measurement are summarized in Table 1. Statistical analyses of these data showed no significant difference ($P \leq 0.05$) between the control and purged prawns. However, the gradual increase in pH values with ice-chilling time up to 7 days is highly significant ($P \leq 0.01$). This was expected and probably was due to the degradation of proteins and amino acids in the abdominal tissues during ice-chilled storage. This gradual increase in pH was also in agreement with the increase in ammonia content in prawn abdominal tissues during ice-chilled storage. Waters & Hale (1981) reported slightly higher pHs in *M. rosenbergii* during the first few days of iced storage.

**Table 1.** Comparison of pH values on non-purged (control) and purged prawns held under ice-chilled conditions*

| Treatment† | Holding time (day)‡ | pH§ | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Control | 0 | 6.87±0.06 | A | | | | | |
| | 3 | 6.88±0.28 | | B | | | | |
| | 5 | 7.02±0.21 | | | C | | | |
| | 7 | 7.31±0.33 | A | B | C | | | |
| Purged | 0 | 6.90±0.17 | D | E | | | | |
| | 3 | 7.04±0.23 | | | | F | | |
| | 5 | 7.14±0.32 | D | | | | | G |
| | 7 | 7.30±0.21 | E | E | F | G | | |

*Values indicate mean values of duplicate experiments with five animals per sampling.

†Not significant ($P \leq 0.05$) between control and purged prawns.

‡Highly significant ($P \leq 0.01$) with holding time.

§Means followed by a common letter differ significantly at $P \leq 0.05$ (C, D, F, G) or highly significant at $P \leq 0.01$ (A, B, E).

Results of ammonia analyses are summarized in Table 2. No significant difference ($P \leq 0.05$) was observed between the purged prawns and the control. However, the

**Table 2.** Comparison of ammonia content (ppm) on non-purged (control) and purged prawns held under ice-chilled conditions*

| Treatment† | Holding time (day)‡ | Ammonia content (ppm)§ | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Control | 0 | 75.6 ± 11.0 | A | B | | | | | | |
| | 3 | 87.4 ± 16.4 | | | | C | D | | | |
| | 5 | 114.8 ± 7.6 | A | | | C | | | | |
| | 7 | 127.1 ± 8.99 | | B | | | D | | | |
| Purged | 0 | 60.2 ± 7.3 | E | F | G | | | | | |
| | 3 | 95.9 ± 21.7 | E | | | | H | | | |
| | 5 | 110.2 ± 15.9 | | F | | | | | | I |
| | 7 | 140.6 ± 17.5 | | | | G | H | I | | |

*Values indicate mean values of duplicate experiments with five animals per sampling.

†Not significant ($P \leq 0.05$) between control and purged prawns.

‡Highly significant ($P \leq 0.01$) with holding time.

§Means followed by a common letter differ significantly at $P \leq 0.05$ (C) or highly significant at $P \leq 0.01$ (A, B, D–I).

increase in ammonia content with ice-chilling time was highly significant ($P \leq 0.01$) and was in agreement with the pH measurement. This increase in ammonia with holding time was also in agreement with those found in marine shrimp (Ward *et al.*, 1979).

Data on soluble collagen ratio are presented in Table 3. The soluble/insoluble collagen ratios were not significantly different ($P \leq 0.05$) between the control and the purged prawns. The increase in soluble/insoluble collagen ratio with ice-chilling time was highly significant ($P \leq 0.01$). This indicates the conversion of the insoluble collagen, the major component in the animal connective tissues, into soluble collagen during ice-chilled storage and thus leading to the degradation of the prawn abdominal tissues. The presence of a collagenolytic enzyme and its partial characterization have been studied (Baranowski *et al.*, 1984; Nip *et al.*, 1983). The increase in soluble/insoluble

**Table 3.** Comparison of soluble/insoluble collagen ratios on non-purged (control) and purged prawns held under ice-chilled conditions*

| Treatment† | Holding time (day)‡ | Soluble/insoluble collagen ratio§ | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Control | 0 | 0.59 ± 0.17 | A | B | C | | | | | |
| | 3 | 0.93 ± 0.03 | A | | | | D | | | |
| | 5 | 1.09 ± 0.59 | | B | | | | | | E |
| | 7 | 1.35 ± 0.55 | | | | C | D | E | | |
| Purged | 0 | 0.70 ± 0.15 | F | G | H | | | | | |
| | 3 | 0.91 ± 0.22 | F | | | | I | | | |
| | 5 | 0.99 ± 0.33 | | G | | | | | | J |
| | 7 | 1.21 ± 0.48 | | | | H | I | J | | |

*Values indicate mean values of duplicate experiments with five animals per sampling.

†Not significant ($P \leq 0.05$) between control and purged prawns.

‡Highly significant ($P \leq 0.01$) with holding time.

§Means followed by a common letter differ significantly at $P \leq 0.05$.

collagen ratio was also in agreement with the softening in the prawn abdominal tissue as expressed by the decrease in peak height/plateau height ratio of the textural profile (Table 4).

**Table 4.** Comparison of textural measurement (peak/plateau ratio) on non-purged (control) and purged prawns held under ice-chilled conditions*

| Treatment† | Holding time (day)‡ | Peak/plateau ratio§ | | | |
|---|---|---|---|---|---|
| Control | 0 | 2.57 ± 0.43 | A | B | C |
| | 3 | 2.06 ± 0.35 | A | | |
| | 5 | 2.08 ± 0.70 | | B | |
| | 7 | 1.94 ± 0.38 | | | C |
| Purged | 0 | 3.00 ± 0.71 | D | E | F |
| | 3 | 2.22 ± 0.59 | D | | |
| | 5 | 2.12 ± 0.80 | | E | |
| | 7 | 1.92 ± 0.45 | | | F |

*Values indicate mean values of duplicate experiments with five animals per sampling.

†Not significant ($P \leq 0.05$) between control and purged prawns.

‡Highly significant ($P \leq 0.01$) with holding time.

§Means followed by a common letter differ significantly at $P \leq 0.05$ (A–E) or highly significant at $P \leq 0.01$ (F).

Textural measurement of the cooked prawn tails are summarized in Table 4. No significant difference ($P \leq 0.05$) was observed in the purging treatment *versus* the control. However, the peak height/plateau height ratios showed highly significant difference ($P \leq 0.01$) with ice-chilling time. This is in agreement with the data collected on soluble/insoluble collagen ratios (Table 3). This means that as the ice-chilled time increases, the prawn tails soften gradually and eventually degraded to the mushy form. It is interesting to note that significant differences ($P \leq 0.05$) exist between 0 and 3 day samples for peak height/plateau height ratio and soluble/insoluble ratio. This confirms the observation that after 3 days of ice-chilled storage significant softening has been developed in the prawn tails.

*Visual observation*

The benefits of purging are believed to be the clearing of animal's head and gastrointestinal tract due to starving the animals and providing a longer shelf life. Our experimental data (Table 5) showed that the presence of black vein in the tail had decreased from 82.5% in the control group to 47.5% in the purged group, and the presence of feed in the head had decreased from 80% in the control group to 55% in the purged group during the purging period. This confirms the belief that purging can clear the feed in the head and the vein to some extent and improve the appearance of the product. However, the degree of integrity and mushiness in the purged prawns was improved by an average of only 20% as compared to the control. In addition, purging the prawns overnight always induces a certain degree of mortality (8% in our experiments) in the population. The mortality, additional handling cost and capital investment in the purging system makes the economic benefits of purging questionable.

**Table 5.** Comparison of visual observations on non-purged (control) and purged prawns held under ice-chilled conditions*

| Treatmen: | Holding time (days) | Visual observation | | | |
|---|---|---|---|---|---|
| | | Mushiness (%) | Presence of feed in vein (%) | Presence of feed in head (%) | Integrity (%) |
| Control | 0 | 0 | 90 | 90 | 100 |
| | 3 | 20 | 80 | 90 | 90 |
| | 5 | 30 | 80 | 70 | 70 |
| | 7 | 50 | 80 | 70 | 60 |
| Purged | 0 | 0 | 60 | 60 | 90 |
| | 3 | 10 | 40 | 40 | 90 |
| | 5 | 20 | 20 | 60 | 70 |
| | 7 | 30 | 70 | 60 | 80 |

*Values indicate mean values of duplicate experiments with five animals per sampling.

## Conclusion

Based on the experimental results, it can be concluded that purging has no significant effect on the quality factors of freshwater prawn (*M. rosenbergii*) such as pHs, ammonia content, soluble/insoluble collagen ratio, and peak height/plateau height ratio (textural measurement). Ice-chilling time showed significant effects on these measurements. Purging did help to improve the quality of cooked prawn by clearing the vein in the tail and the feed in the stomach of some animals, but it did not considerably reduce the tendency to become mushy after 3 days under ice-chilled conditions. This partial improvement in quality is obtained through increasing the unit handling cost of the product. Tissue mushiness, the main problem in the quality of freshwater prawn, is still not resolved by this process.

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# Intermediate technology for fish cracker ('keropok') production

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## Summary

'Keropok' production is an important cottage industry in Malaysia. However, production methods used are poor, resulting in inferior quality products which have uneven expansion characteristics, dark objectionable colours and varying shapes, sizes and thicknesses. This paper describes a method that can be used to upgrade 'keropok' production. Basically it is an adaptation from sausage production technology. Products from this method were superior in terms of appearance, shape and linear expansion and were more acceptable to taste panellists.

## Introduction

Crackers (in Malaysia known as 'keropok') are popular snack foods in Malaysia and the ASEAN countries. In the West, they would be classified as 'half-products' or 'intermediate' (Lachmann, 1969) and expanded snack products (Cumminford & Beck, 1972). Basically, they are produced by gelatinization of starch with water, to form a dough which is shaped, cooked and then sliced. The slices are then dried and expanded into a low density porous product upon immersion in hot oil. Fish, prawns or other food ingredients are usually added.

'Keropok' production in Peninsular Malaysia is usually confined to the coastal fishing areas along the east coast states (Siaw & Yu, 1978). In the states of Trengganu and Kelantan, 'keropok' production is a seasonal activity and is usually processed during the months of April to October. September and October are the peak production periods. A considerable proportion of the population is involved in 'keropok' production in these two states (Maarof, 1976). Basically, production still follows traditional methods and remains a cottage industry.

## Materials and methods

*Traditional method*

The traditional method is the most common process used and is heavily dependent on manual labour. Many varieties of fish are used, the most common being *Clupea leiogaster*. This is mainly consumed by the rural communities.

The fish is deboned manually and mixed with flour. Generally, sago flour (*Metroxylon sagu*) and/or tapioca flour (*Manihot utilissima*) is used. Salt, monosodium glutamate, water and sometimes sugar are added. This mixture is then kneaded manually or pounded using long wooden poles in a wooden mortar, 30–50 cm in diameter and 20–25 cm in depth, placed on the ground.

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After the appropriate consistency has been obtained, the dough is shaped manually by rolling into cylindrical rods, 25–30 cm long and 4–6 cm in diameter. To facilitate the rolling process, more flour is added to the dough during this procedure. The rolls are then boiled for about 1.5 hr until cooked. The cooked roll is then allowed to cool at room temperature. On the east coast of Peninsular Malaysia, the dough at this stage can be either fried or sliced. The fried product is known as 'keropok lekur' and the sliced keropok as 'keropok hiris'. The sliced variety is more popular.

Slicing is carried out manually using a knife and each slice has a thickness of 3–5 mm. The sliced pieces of dough are then dried in the sun. The drying period varies and can be as long as 2–3 days, depending on prevailing weather conditions.

It is hardly surprising then that 'keropok' produced this way are of poor quality. Product compositions vary among processors and are largely dependent on the desired profit margin. If fish is expensive, less fish or less expensive species will be used. There is no form of control for consistency or quality. The mixing process does not ensure a homogeneous dough and the rolls subsequently produced are of varying diameters. The centre of the rolls are very often still uncooked after boiling. Manual slicing results in pieces of cooked dough with different thicknesses within and between slices and the irregular diameter of the rolls causes different shapes and sizes to be produced. The process of sun drying is also uncontrollable and variable. This results in fluctuations in the moisture content of the dried slices. Usually, the moisture content is high as the product is sold by weight.

A previous paper (Yu, Mitchell & Abdullah, 1981) has described the production of 'keropok' using the extrusion technique. However, it was felt that although extruded 'keropok' was accepted by taste panellists, the method is an expensive one. High initial capital outlay is necessary and the method also involves the requirement of understanding the technology used. As the vast majority of 'keropok' processors in Malaysia are at the cottage industry level, a less sophisticated method has been developed, which involves considerably less initial capital investment and requires cheaper and less complicated equipment. This method involves technology that is intermediate in the level of sophistication between the traditional and the extrusion methods. This paper describes the method developed and compares the organoleptic qualities of 'keropok' produced by this 'new' method and those prepared traditionally.

*Processing method for 'keropok'*

The fish (*Clupea leiogaster*) was obtained fresh from the market. After filleting, the flesh and other ingredients were processed as outlined in Fig. 1.

The formulation used was 1:1 fish to flour, 2% salt, 1% sugar and 25–30% water. The fish:flour mixture and the other ingredients are mixed in a bowl mixer until a homogeneous mixture is obtained. The dough-like mixture is then stuffed into cellulose casings using a sausage stuffer (Dick, West Germany). The stuffed rolls are then steamed to gelatinize the starch granules for 60–90 min under ordinary pressure, when the temperature of the granules reaches 90–95°C. After steaming, the cooked roll is immersed in iced water in order to prevent shrinkage, reduce cook loss and to facilitate separation from the casing. It is then chilled overnight at 5–10°C before being sliced using a gravity slicer. A thickness of about 3 mm was found to be more acceptable in terms of packing, drying and expansion properties. For oven drying, an initial lower temperature of 40–45°C was used to prevent case hardening which leads to poor expansion. A final temperature range not exceeding 65–70°C was used and the product dried to a final moisture content of about 10% (by weight).

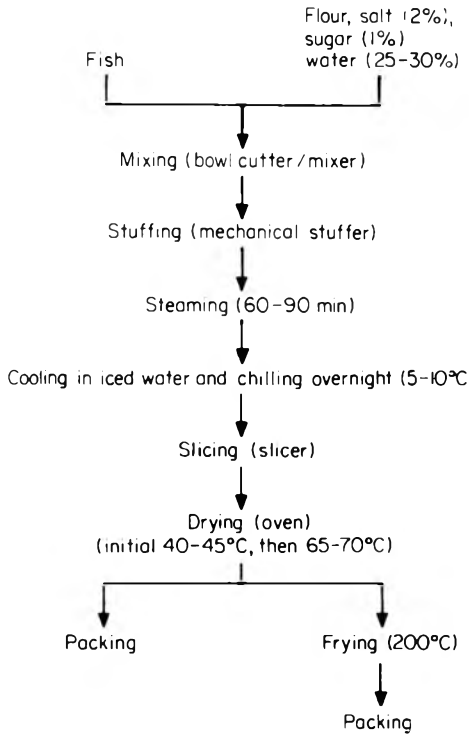


**Figure 1.** The modified traditional method of 'keropok' processing.

*Chemical analyses*

Analyses for moisture, fat, crude protein, ash and salt contents were carried out according to Pearson (1970).

*Linear expansion*

The percentage linear expansion was calculated as in Yu *et al.* (1981).

*Organoleptic evaluation*

The acceptability of 'keropok' produced by the modified method was compared to traditional 'keropok' following the method of Amerine, Pangborn & Roessler (1965). The samples were fried in hot oil at 200°C and evaluated by a taste panel of twenty-five people. The panel consisted of consumers selected from the University (UPM) and the general public. No specific training was given to any of the panel members.

Intensity scales of 5 for excellent and 1 for poor were used to evaluate the appearance and crispiness of the samples.

## Results and discussion

*Chemical composition and expansion characteristics*

The chemical analyses and expansion properties of four samples of 'keropok' processed traditionally compared to those prepared using the modified method (UPM 'keropok') are shown in Table 1. There is slight variation in chemical composition

**Table 1.** Chemical composition and linear expansion of fish (*Clupea leiogaster*) 'keropok' in Malaysia

| | | Samples | | | | |
|---|---|---|---|---|---|---|
| | | A | B | C | D | UPM |
| Chemical composition | Moisture | 13.3 | 11.9 | 12.8 | 13.1 | 9.5 |
| | Crude protein (N×6.25) | 20.3 | 20.6 | 20.0 | 21.7 | 21.6 |
| | Fat | 1.4 | 1.5 | 1.1 | 1.0 | 1.6 |
| | Salt (Cl) | 2.0 | 2.6 | 2.6 | 2.4 | 2.6 |
| | Ash | 2.9 | 2.4 | 2.8 | 2.7 | 2.8 |
| Expansion properties | Linear expansion | 77.7 | 46.9 | 34.3 | 64.3 | 95.4 |
| | S.D. (±) | 7.2 | 9.6 | 9.8 | 8.1 | 6.7 |

whereas linear expansion varies considerably. Chemical composition depends on formulation whereas linear expansion depends on the physical properties of the fish-flour mixture. Traditional products have much lower expansion and this is due to poor mixing, variation in the thicknesses of the sliced product and uneven drying. Basically this results from a lack of knowledge of the functional properties of the ingredients utilized and processing technology involved. 'Keropok' processing by the modified method begins with a homogeneous mixture of fish and flour that can only be achieved mechanically. A well mixed structure will result in smooth texture and good expansion. Mixing that is not homogeneous causes decreased gelatinization and therefore the expansion ratio is lowered. Processors should realize that only a well mixed structure will gelatinize fully when cooked. Ungelatinized or semi-gelatinized starch granules will result in poor expansion characteristics. In the modified method, controlled cooking also ensures adequate gelatinization of the starch granules.

*Organoleptic evaluation*

From Table 2, it can be seen that the appearance and shape score for UPM 'keropok' was rated higher and was more acceptable compared to those prepared traditionally. The UPM samples were round and flat and obtained the highest mean score. In contrast, the shape of traditionally processed 'keropok' varied considerably from elongated and pointed to an oval shape. This inconsistency is due to the use of the

**Table 2.** Mean score for appearance and shape of 'keropok' *

| | Samples | | | | |
|---|---|---|---|---|---|
| | A | B | C | D | UPM |
| Mean score | 3.4 | 2.4 | 2.9 | 3.9 | 4.5 |

* All samples showed significant difference at $P$ 0.05.

hand rolling method as no casings or moulds are used to standardize shape. Hand slicing also causes variation in thickness. The main advantage of using a mechanical slicer is that thickness can be controlled.

The crispiness ratings for UPM 'keropok' were also much higher (Table 3) compared to most traditional samples. For the latter, poor cooking procedures and uneven drying in the sun causes the samples to crinkle upon frying. This is caused by the fact that some portions expand to a greater extent than others. The starch in the unexpanded portion has not been fully gelatinized. Starch granules that are fully gelatinized will result in better rupture of the starch cells during frying. A linear expansion greater than 77% was found to be the ideal level of crispiness. This was achieved in all UPM samples. In comparison, less than 20% of the traditional samples had expansion ratios greater than 60%.

**Table 3.** Duncan's multiple range test (DMRT) for crispiness ratings of 'keropok'

| | Samples | | | | |
|---|---|---|---|---|---|
| | A | B | C | D | UPM |
| Mean score | 2.6 | 2.6 | 2.7 | 3.2 | 3.3 |

Samples joined by lines are not significantly different at 0.05 under the DMRT.

## Conclusion

The modified method produces 'keropok' that are superior in shape and appearance as well as crispiness. Processors should realize that the only way to improve efficiency and increase production is to incorporate some degree of mechanization into their current methods. The method described in this paper could be one way in which processors can produce a better 'keropok' and thereby capture a larger market for their product. Steps taken to introduce the improved technique to 'keropok' processors include publications in the local press and items on the local radio networks.

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# Use of low resolution NMR for determining fat content in meat products

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## Summary

The fat content in meat samples was estimated using a pulsed nuclear magnetic resonance (NMR) spectrometer.

The proton spin-lattice relaxation data for various samples have been analysed. The results have been interpreted in terms of a two-component system. The relaxation rate and amplitude of each component could be determined. The NMR results correlated significantly with chemical data. Correlation of fat content with water content was also investigated.

## Introduction

The NMR analytical method has been used successfully throughout the food industry for both research and quality control purposes (Brosio, 1982). The aim of this article is to provide an application of pulsed NMR to determine the fat content in meat products.

The X-ray instrument (Young, Motula & Twigg, 1976) which analyses 6 kg samples causes no structural damage to meat and provides a direct reading of fat percentage within 2 sec. The instrument is based on the principle that X-ray absorption is inversely proportional to fat content. Although samples do not have to be ground for this procedure, they must be free of salt and additives which might substantially influence the reading.

The near infrared reflectance (NIR) spectrometry can provide fast and easy determinations of all major chemical constituents after a definite preparation and careful handling of the samples. In commercial NIR instruments, light reflectance is measured very accurately from the surface of a sample at several different wavelengths (1400–2600 nm) and combined in a microprocessor to yield the concentration of the food constituents—e.g. water, fat, protein. To recognize the fat, water and protein components from the complex NIR spectra, the microprocessor must be calibrated. The calibration procedure is toilsome because many samples must be analysed by conventional chemical methods. The quality of the calibration is decisive and each meat subgroup may need its own calibration set. But, once calibrated, the NIR instrument is considered to be fast and easy to use (Martens, Bakker & Hildrum, 1981).

When the fat content of meat is determined with NMR, water and oil interfere within the liquid component of the nuclear resonance signal. Casey & Miles (1974) determined the fat content (1–12%) of minced and freeze dried meat at 70°C. The technique used required that the spectrometer be calibrated with purified beef fat. More recently (Tipping, 1982), the protein content of fresh meat was estimated using pulsed NMR spectroscopy. Adding a protein to the alkali copper relaxation reagent in order to form a copper complex, caused quantitative changes in spin-spin relaxation rates. This procedure did not determine fat or water content.

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This study of the spin-lattice relaxation by NMR pulsed technique has been performed to determine the fat content of intact meat.

## Materials and methods

The fresh meat—either beef (*Triceps brachii*) or pork (ham)—was trimmed of all visible fat. Then it was processed through a cutter which yielded flaked meat. This was added to trim in varying amounts, depending on desired fat content. The meat was ground after mixing and then frozen at −30°C in plastic bags. The samples were kept at room temperature for 3 hr to thaw before proceeding with measurements.

The emulsions were prepared by a standard method in a cutter ($R_t$ robot cutter) with salts (NaCl 2%, sodium caseinate 1%) and additives (tripolyphosphate (0.5%)); 0–12% pork rind was also added.

The NMR relaxation measurements were performed at 10 and 40°C with a Bruker PC20 pulsed spectrometer at a frequency of 20 MHz. The samples took up a volume of 1 $cm^3$ in 7.5 mm tubes (external diameter). Before beginning the measurements, the tubes were kept in the probehead for 10 min to reach temperature equilibrium. The present work consisted of a study of the proton spin-lattice relaxation in meats. When the nuclei are exposed to a static magnetic field ($H_0$), they become magnetized. The electromagnetic field ($H_1$) absorption destroys the nuclear magnetization. The component of the magnetization along the static field returns with a characteristic time $T_1$ to its equilibrium position under the influence of the lattice oscillating microscopic fields produced by Brownian tumbling. This NMR method can also detect the different species of the same nuclei in different lattices.

**Table 1.** NMR results for fats

| Sample | Temperature (°C) | $T_{1a}$ ± s.d. (msec) | $T_{1b}$ ± s.d. (msec) | Pa ± s.d. (%) | $N$ |
|---|---|---|---|---|---|
| Pork fat | 10 | 66 ± 2 | 309 ± 10 | 58 ± 1 | 4 |
| Pork fat | 40 | 122 ± 3 | 436 ± 11 | 55 ± 2 | 3 |
| Beef fat | 10 | 67 ± 1 | 452 ± 17 | 55 ± 2 | 3 |
| Beef fat | 40 | 120 ± 2 | 478 ± 5 | 58 ± 2 | 3 |

Pa: percentage of the $T_{1a}$ population (Pa = 100 × A/(A + B)); $N$: number of measurements; s.d.: standard deviation estimate.

Spin-lattice relaxation times were obtained by the (180°, $t$, 90°) sequence (Farrar & Becker, 1971). The magnetization is rotated by the first pulse in the opposite way (180°) and varies with time ($t$) under the effect of spin-lattice relaxation. The third pulse (90°) is used to measure the amplitude of the magnetization. The intensity was measured at 11 $\mu$sec after the end of the 90° pulse. A series of thirty-one $t$-values ranging between 4 and 3584 msec in a geometric progression like sequence was used for all $T_1$ measurements, each value was an average of nine measurements. The time lapse between two consecutive measurements was 3 sec to allow the nuclear magnetization to return to its equilibrium value.

In all cases a double exponential was fitted to the time dependence of the non-equilibrium magnetization and $T_{1a}$ and $T_{1b}$ were thus obtained. The computer data were obtained by using a non-linear regression program adapted from Atkins (1971) carried out on an HP 85 calculator.

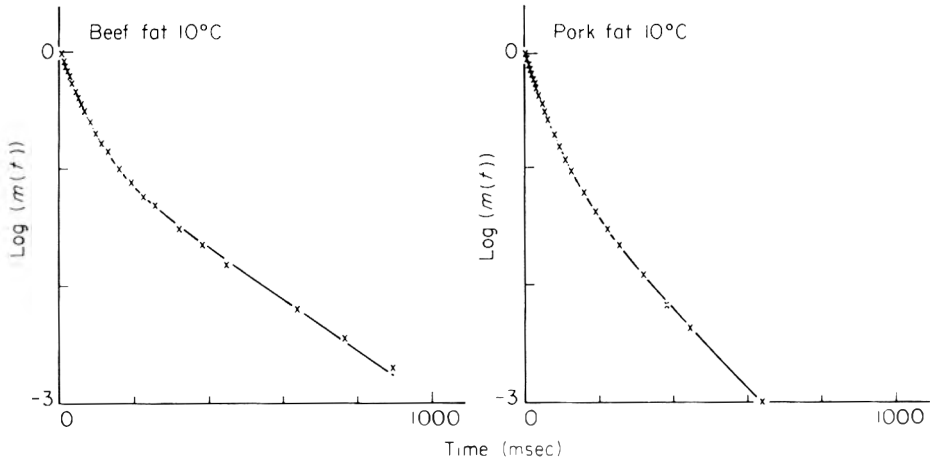


**Figure 1.** Proton spin-lattice relaxation curves in fats. *Experimental data: ——— fitted curve.

The lipid contents of beef and pork samples were determined by the standard Folch method. Emulsion lipid contents were determined by the Soxhlet method. Meat samples (2 g) were subjected to an oven drying procedure (48 hr at 105°C) to determine their water content. The samples were cooled in a dessicator before re-weighing in dishes. In both cases the methods were carried out in duplicate. The average values were used for comparisons with the NMR data.

## Results and discussion

A single component spin-lattice relaxation time was observed at all temperatures for muscle (Pearson *et al.*, 1974). Because of cross-relaxation between water and proteins, the relaxation time of both water and muscle protons was the same provided that the initial 10 msec of the relaxation decays were ignored (Edzes & Samulski, 1978). The values of the relaxation times of muscle protons were in the vicinity of 400 and 600 msec, at respectively 10 and 40°C for pork. The corresponding values for beef were 300 and 500 msec. By contrast, a double component spin-lattice relaxation time was found in fat (Table 1). Figure 1 shows the proton spin-relaxation curves in fats. The magnetization was normalized by using the reduced magnetization:

$$m(t) = (\mathrm{Meq} - \mathrm{Mz}(t))/\beta\mathrm{Meq},$$

where Meq is the magnetization equilibrium value, Mz($t$) is its value at time $t$, $\beta$ is a coefficient owing to pulse error.

This may be explained if different proton lattices in lipids are taken into account, namely hydrophobic hydrocarbon chains and polar groups. At least two components in the spin-lattice relaxation measurements were expected to be found in these samples. Given the accuracy of the methods, a third decay was therefore inseparable. Five parameters can be determined by a non-linear least squares repression program: the relaxation times $T_{1a}$ and $T_{1b}$, the A and B amplitudes of each component, and the coefficient $\beta$ owing to pulse error.

$$\mathrm{Mz}(t) = \mathrm{A}\,(1-\beta\exp^{(-t/T_{1a})}) + \mathrm{B}(1-\beta\exp^{(-t/T_{1b})}).$$

Tables 2–4 report the $T_1$ values for samples with different fat contents, at 10 and 40°C. The percentages of water and fat are also listed in these tables. Within experimental error the relaxation times can therefore be assumed to be constant for each set. The population Pa = $100 \times A/(A+B)$ determined the percentage of the $T_{1a}$ population. The Pa parameter must be directly linked to the fat content because it appeared only in the presence of fat in the sample. Table 5 shows a good linear relationship between the population Pa and the lipid contents (%). For emulsions, the correlation coefficient was not as high. This could be explained by the fact that pork rind and salt addition were not accounted for. However, the NMR results correlated significantly with conventional methods (Fig. 2). Table 6 shows the linear regression equations of water percent against fat percent from the NMR, Soxhlet or Folch methods. Within these series, the water and fat contents were closely correlated. A lower correlation appeared in the emulsion series for both techniques (Folch: $r = 0.985$; NMR: r = 0.957). Recently, Casey & Crosland (1982) determined the fat content of beef mince by direct measurement of moisture content evaluated by infrared heating.

It should be pointed out that this technique does not require tight control of the sample temperature, although $T_1$ values are significantly different. The regression lines

**Table 2.** NMR results for pork samples

| | Temperature | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | 10°C | | | 40°C | | | | |
| Sample No. | $T_{1a}$ (msec) | $T_{1b}$ (msec) | Pa (%) | $T_{1a}$ (msec) | $T_{1b}$ (msec) | Pa (%) | Fat (%) | Water (%) |
| 1 | 65.8 | 398 | 6.2 | 94.0 | 616 | 6.1 | 4.76 | 74.65 |
| 2 | 68.4 | 375 | 5.4 | 89.1 | 595 | 6.5 | | 73.55 |
| 3 | 72.0 | 398 | 6.3 | 91.0 | 639 | 6.7 | 6.37 | 73.25 |
| 4 | 68.4 | 367 | 7.0 | 98.7 | 599 | 7.7 | | 72.70 |
| 5 | 66.5 | 391 | 7.3 | 102.6 | 631 | 8.2 | 7.89 | 71.40 |
| 6 | 62.7 | 383 | 7.2 | 108.5 | 643 | 8.7 | | 71.30 |
| 7 | 67.6 | 407 | 8.1 | 97.7 | 632 | 8.2 | 9.42 | 70.90 |
| 8 | 79.4 | 403 | 9.0 | 111.1 | 638 | 9.7 | | 70.00 |
| 9 | 67.1 | 400 | 9.8 | 103.6 | 653 | 9.7 | 10.96 | 69.50 |
| 10 | 67.5 | 399 | 10.1 | 104.6 | 649 | 10.3 | | 68.85 |
| 11 | 66.1 | 381 | 10.5 | 109.5 | 632 | 11.4 | 12.44 | 67.85 |
| 12 | 67.9 | 383 | 11.4 | 109.5 | 634 | 11.8 | | 67.70 |
| 13 | 66.1 | 381 | 11.5 | 115.8 | 632 | 12.7 | 14.00 | 66.80 |
| 14 | 65.6 | 386 | 12.5 | 112.1 | 634 | 13.0 | | 66.50 |
| 15 | 68.9 | 391 | 12.8 | 125.7 | 646 | 14.7 | | 66.25 |
| 16 | 71.1 | 380 | 13.3 | 115.2 | 631 | 13.9 | 16.28 | 65.30 |
| 17 | 67.2 | 363 | 15.7 | 114.1 | 629 | 15.9 | 19.97 | 62.05 |
| 18 | 72.5 | 354 | 20.4 | 117.2 | 595 | 19.5 | | 58.25 |
| 19 | 64.4 | 355 | 21.2 | 125.0 | 633 | 22.6 | 28.08 | 56.30 |
| 20 | 68.9 | 356 | 25.8 | 123.0 | 582 | 25.6 | | 54.00 |
| 21 | 70.8 | 352 | 28.9 | 122.9 | 586 | 28.0 | 36.86 | 50.20 |
| Average | 68.3 | 381 | | 109.0 | 625 | | | |
| s.d. | 3.5 | 17 | | 10.9 | 21 | | | |

Pa: percentage of the $T_{1a}$ population (Pa = $100 \times A/(A+B)$); population size: eight readings (two measurements × four sub-samples).

**Table 3.** NMR results for beef samples at 40°C

| Sample no. | $T_{1a}$ (msec) | $T_{1b}$ (msec) | Pa (%) | Fat (%) | Water (%) |
|---|---|---|---|---|---|
| 1 | 63 | 406 | 3.4 | 3.74 | 74.2 |
| 2 | 93 | 410 | 4.2 | | 73.8 |
| 3 | 98 | 410 | 5.4 | | 73.1 |
| 4 | 103 | 430 | 6.7 | | 72.3 |
| 5 | 106 | 450 | 6.7 | | 71.7 |
| 6 | 95 | 431 | 7.6 | 7.92 | 70.7 |
| 7 | 95 | 440 | 7.1 | | 70.9 |
| 8 | 86 | 425 | 7.5 | | 70.3 |
| 9 | 102 | 470 | 8.9 | | 69.7 |
| 10 | 101 | 471 | 9.2 | | 68.6 |
| 11 | 111 | 431 | 9.1 | 11.16 | 68.0 |
| 12 | 121 | 400 | 12.1 | | 67.4 |
| 13 | 111 | 445 | 11.4 | | 66.6 |
| 14 | 122 | 412 | 13.0 | | 66.1 |
| 15 | 108 | 440 | 12.0 | | 65.9 |
| 16 | 115 | 457 | 13.2 | 13.90 | 65.8 |
| Average | 101.8 | 433 | | | |
| s.d. | 14.4 | 22 | | | |

Pa: percentage of the $T_{1a}$ population ($Pa = 100 \times A/(A+B)$); population size: eight readings (two measurements × four subsamples).

**Table 4.** NMR results for emulsions at 10°C

| Sample no. | $T_{1a}$ (msec) | $T_{1b}$ (msec) | Pa (%) | Fat (%) | Water (%) |
|---|---|---|---|---|---|
| 1 | 68.6 | 411 | 29.1 | 37.5 | 50.2 |
| 2 | 69.2 | 433 | 28.4 | 37.2 | 50.1 |
| 3 | 67.0 | 433 | 28.1 | 36.9 | 51.3 |
| 4 | 69.0 | 432 | 27.0 | 35.2 | 51.6 |
| 5 | 66.6 | 425 | 25.9 | 35.1 | 52.5 |
| 6 | 70.6 | 441 | 25.1 | 34.9 | 53.0 |
| 7 | 64.6 | 409 | 23.4 | 34.0 | 53.5 |
| 8 | 69.8 | 436 | 24.7 | 33.7 | 53.7 |
| 9 | 68.7 | 433 | 24.9 | 33.2 | 53.5 |
| 10 | 66.1 | 416 | 22.6 | 32.3 | 54.7 |
| 11 | 67.8 | 432 | 24.5 | 31.9 | 55.3 |
| 12 | 68.7 | 434 | 23.3 | 31.3 | 55.1 |
| 13 | 67.9 | 429 | 22.3 | 29.8 | 56.0 |
| 14 | 67.6 | 442 | 21.9 | 29.7 | 56.9 |
| 15 | 66.2 | 421 | 21.2 | 28.8 | 56.6 |
| 16 | 63.6 | 412 | 20.2 | 28.0 | 58.4 |
| 17 | 66.7 | 433 | 20.3 | 27.7 | 58.4 |
| 18 | 70.1 | 426 | 21.3 | 27.4 | 57.9 |
| 19 | 60.2 | 410 | 17.3 | 26.6 | 59.2 |
| 20 | 71.4 | 454 | 19.8 | 25.7 | 61.3 |
| 21 | 68.8 | 424 | 19.3 | 25.6 | 59.0 |
| Average | 67.5 | 427 | | | |
| s.d. | 2.5 | 11 | | | |

Pa: percentage of the $T_{1a}$ population ($Pa = 100 \times A/(A+B)$); population size: four readings (two measurements × two subsamples).

**Table 5.** Linear regression equations

| Sample | Temperature (°C) | $a \pm$ s.d. | $b \pm$ s.d. | $N$ | $r$ | $t$ | SIG |
|---|---|---|---|---|---|---|---|
| Emulsion | 10 | 1.15 ± 0.08 | 4.7 ± 1.9 | 21 | 0.957 | 14.33 | *** |
| Pork | 10 | 1.39 ± 0.04 | −2.4 ± 0.5 | 11 | 0.997 | 40.14 | *** |
| Pork | 40 | 1.43 ± 0.02 | −3.5 ± 0.4 | 11 | 0.999 | 59.99 | *** |
| Beef | 40 | 1.06 ± 0.14 | 0.3 ± 1.2 | 4 | 0.984 | 7.78 | * |

Fat (%) = $a \times$ Pa (%) + $b$: Pa: percentage of the $T_{1a}$ population; $N$: number of measurements; $r$: correlation coefficient; $t$: Student's $t$-value; SIG: significance level of the linear regressions ***$P < 0.001$; **$P < 0.01$; *$P < 0.05$.

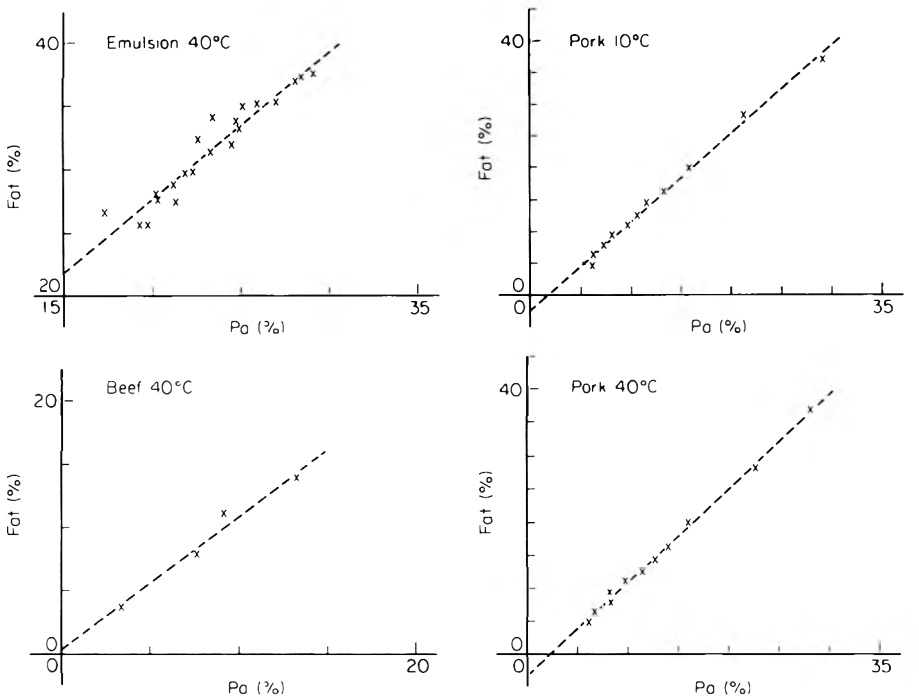


**Figure 2.** Relationship between fat content determined by chemical method and Pa percentage of the $T_{1a}$ population.

**Table 6.** Linear regression equations

| Sample | Method | $a \pm$ s.d. | $b \pm$ s.d. | $N$ | $r$ | $t$ | SIG |
|---|---|---|---|---|---|---|---|
| Emulsion | NMR (10°C) | −0.94 ± 0.07 | 77.0 ± 1.6 | 21 | 0.957 | 13.71 | *** |
| | Soxhlet | −0.83 ± 0.06 | 81.0 ± 2.0 | 21 | 0.985 | 25.07 | *** |
| Pork | NMR (10°C) | −1.02 ± 0.02 | 79.2 ± 0.3 | 21 | 0.995 | 41.99 | *** |
| | Folch | −0.76 ± 0.02 | 78.0 ± 0.5 | 11 | 0.999 | 62.76 | *** |
| Beef | NMR (40°C) | −0.92 ± 0.05 | 77.6 ± 0.5 | 16 | 0.979 | 17.96 | *** |
| | Folch | −0.83 ± 0.01 | 77.0 ± 1.0 | 4 | 0.999 | 153.8 | *** |

Water (%) = $a \times X$ (%) + $b$: $X$: percentage of fat determined by different methods (NMR, Soxhlet, Folch); $N$: number of measurements; $r$: correlation coefficient; $t$: Student's $t$-value; SIG: significance level for the linear regressions ***$P < 0.001$; **$P < 0.01$; *$P < 0.05$.

did not differ significantly in their slopes and intercepts. As opposed to the NIR method, the use of different calibration sets for each subgroup of meat products is not necessary with this procedure.

The sources of error in the measurement of fat content are the intrinsic uncertainties in NMR reading and sub-sampling. The increase in the number of signal accumulations improved the signal to noise ratio and reduced the inaccuracy on the NMR parameters. The reproductibility for a same sub-sample was correct and did not require an increase in the number of replicate readings.

The sample heterogeneity produced a scattering and between samples significant differences at the level $P < 0.001$ were found. When using the NMR method to determine fat content below 5% at least 4 sub-samples of the same sample must be analysed to allow correct confidence limits. The NMR method advantage comes from its rapidity. A large number of samples can be measured in a short while, the temperature and the sample weight are not necessary to be known.

With the equipment used, the method described is suitable for fat content determination in ground meat products at 10 or 40°C, the latter condition being more convenient from a practical standpoint, in a 3–40% lipid concentration range where the two components are well defined in the $T_1$ decay. The mean standard deviation of the difference between calculated (NMR measured population converted via regression data) and chemically assessed fat content is 0.8% for meat and 1.2% for emulsion samples.

## Acknowledgments

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# Viscosity and emulsifying ability of fish and chicken muscle protein

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## Summary

The influence of myofibrillar and sarcoplasmic proteins on the viscosity and emulsifying properties of bonito, cod, horse mackerel and chicken muscle homogenates was studied.

Variations in the emulsifying ability of the muscle homogenates and myofibrillar fractions at differing soluble protein concentrations were the same for the three fish species, which was lower than for chicken muscle. The sarcoplasmic proteins from all four species exhibited the same emulsifying properties. At low protein concentrations (5 mg/ml), the emulsifying capacity of the sarcoplasmic proteins from the fish species was higher than the emulsifying capacity of the myofibrillar proteins and the muscle homogenates; in the case of chicken muscle, however, the emulsifying capacity of the sarcoplasmic proteins was lower over the entire range of concentrations studied.

A linear relationship was found for apparent viscosity ($\eta_{app}$) *versus* protein concentration. The apparent viscosity of the chicken muscle extracts was higher than that of the fish muscle extracts. In all four species studied, the myofibrillar protein was the main factor responsible for the viscosity of the homogenates, though other factors caused it to increase.

There was a linear relationship between apparent viscosity and the amount of fat emulsified per 20 ml of protein extract over a concentration range between 14 and 40 mg of protein/ml in the four species studied, so it was possible to calculate one property from the other.

The lower emulsifying ability and apparent viscosity of the fish muscle extracts may be the result of greater lability of their myofibrillar proteins.

## Introduction

Emulsifying ability and viscosity, functional properties of proteins with a considerable effect on a variety of food processing techniques, have been widely studied for both vegetable and animal proteins, though references to fish proteins in the literature are limited.

In a previous paper (Jiménez-Colmenero & Borderías, 1983), the authors reported high correlations among emulsifying capacity, viscosity, and soluble protein in frozen fish muscle over the entire storage period. They also recorded appreciable differences among the functional properties of the species studied, but no attempt was made to explain the specific differences in behaviour.

It is generally accepted that salt soluble proteins possess superior emulsifying properties to those of water soluble proteins (Saffle, 1968; Schut, 1976). Nevertheless, Tsai, Cassens & Briskey (1972) found that, at low concentrations (less than 4 mg/ml),

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sarcoplasmic proteins exhibited greater emulsifying capacity than myosin, actin and tropomyosin-troponin. Gaska & Regenstein (1982a,b) observed that, whereas sarcoplasmic proteins did not take part in emulsion formation, high salt soluble proteins may play a very important role. Little information is available on how the salt soluble protein fractions affect the viscosity of muscle homogenates.

The object of the present study was to attempt to determine the role of proteins soluble in 5% NaCl solutions (myofibrillar proteins) and 0.05 M NaCl solutions (sarcoplasmic proteins) in the viscosity and emulsifying properties of muscle homogenates, in order to establish a range of suitable conditions for studying these functional properties in myosystems.

## Materials and methods

The samples studied were obtained at a local market and consisted of bonito (*Sarda sarda*, Bloch), cod (*Gadus morhua*, L.), horse mackerel (*Trachurus trachurus*, L.) and chicken breast muscle.

The samples were minced (orifice diameter: 5 mm), frozen at −30°C, vacuum packed, and stored at −25°C (maximum: 4 days) until analysis.

Several fractions were obtained from each of the myosystems, as indicated in Figs 1 and 2. It should be borne in mind that, in the H, Ha and SSP fractions, despite their being homogenized in 5% NaCl, part of the myofibrillar protein remained insoluble.

Total protein, fat binding capacity, and apparent viscosity were determined for each of the fractions shown. The amount of protein present was quantified by Kjeldahl's method ($N \times 6.25$) after precipitation with trichloroacetic acid; the emulsifying ability was determined using the method described by Jiménez-Colmenero & García Matamoros (1981) at four different protein concentrations, with three replicates for each dilution. The results are expressed in g of oil/100 mg of soluble protein (EC) and as the weight of oil in g emulsified per 20 ml of protein extract (WO).

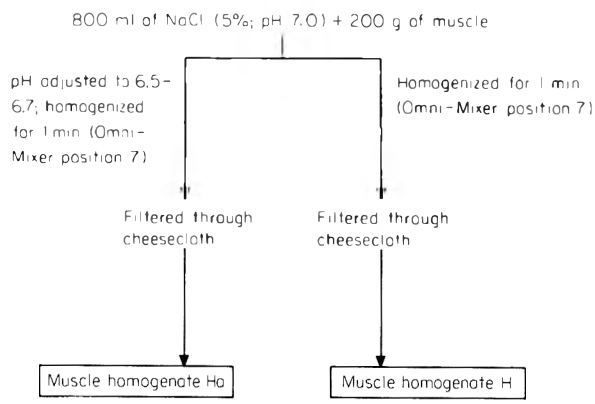


**Figure 1.** Method used to obtain the protein extracts Ha and H. The entire extraction process was carried out at a temperature of between 0 and 5°C.

The apparent viscosity ($\eta_{app}$) was measured with a Brookfield rotary viscometer using spindles Nos 3 (for fish) and 4 (for chicken) at a velocity of 20 rpm. As a certain degree of hysteresis had been observed in previous studies, mainly due to the air mixed in during homogenization, measurement readings were effected starting 20 min and

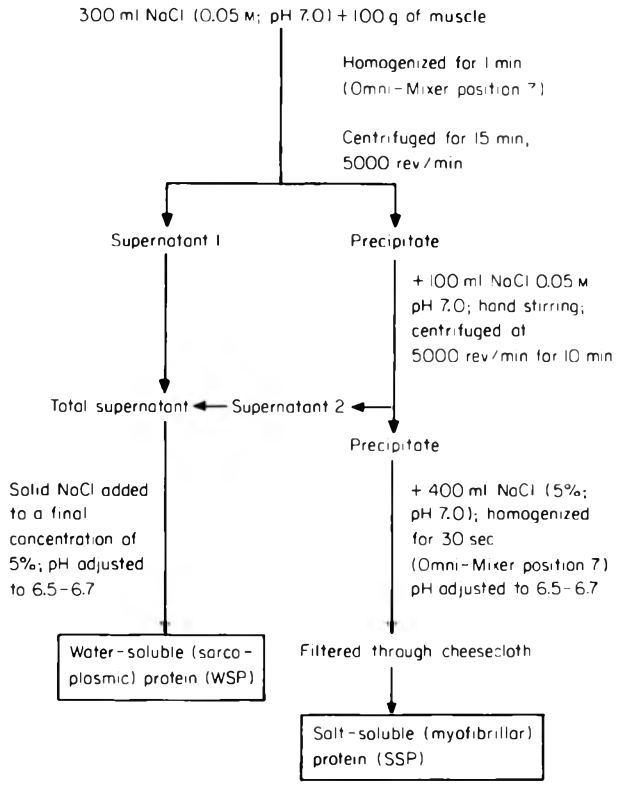


**Figure 2.** Method used to obtain the salt soluble protein (SSP) and water soluble protein (WSP) fractions. The entire extraction process was carried out at a temperature of between 0 and 5°C.

ending 40 min after homogenization. Four readings were taken. The rotor was operated for 3 min for each measurement; the protein extracts were placed in beakers in an ice-water bath, with the temperature of the homogenate being between 3 and 5°C. The apparent viscosity of the WSP fraction did not register on the measurement scale under the experimental conditions employed and was therefore not considered. Measurements were taken on at least four different concentrations of each fraction. Apparent viscosity is expressed in centipoises (cP).

The protein solubility in 5% NaCl (PS) was measured using the method of Ironside & Love (1958).

The regression curves were calculated using a microcomputer; the level of significance was established by applying an *F*-test and the goodness of fit by the index of determination, $r^2$.

## Results and discussion

*Emulsifying ability*

The emulsifying ability was plotted (Figs 3–5) against the soluble protein concentration for each fraction. The soluble protein concentration for H, Ha, and SSP was calculated using the values given in Tables 1 and 2.

In the species studied the relationship between EC and soluble protein

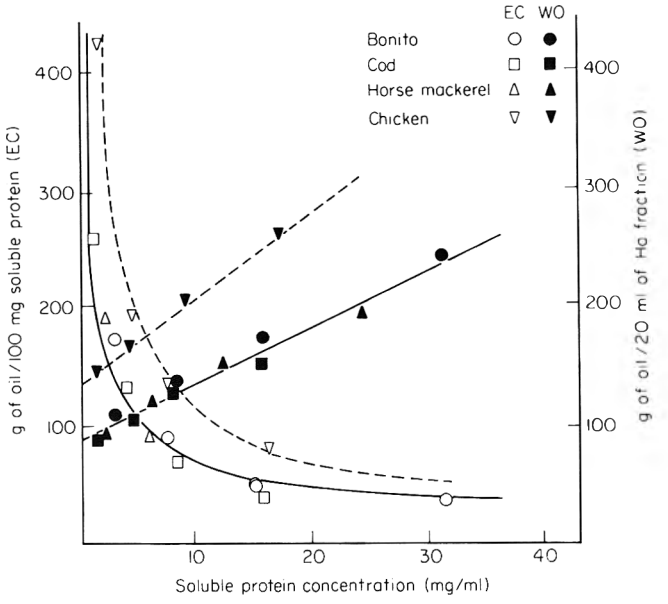


**Figure 3.** Emulsifying capacity (EC: g of oil/100 mg of soluble protein) and weight of oil (WO: g of oil/20 ml of Ha protein extract) *versus* Ha extract soluble protein concentration (mg/ml).

concentration (Figs 3–5) was found, for all the fractions, to fit power curves ($r^2 > 0.97$ and $P < 0.01$). This type of relationship has been reported in the case of livestock meat by various authors (Hegarty, Bratzler & Pearson, 1963; Tsai *et al.*, 1972; Gillet *et al.*, 1977) for both muscle homogenates and different protein fractions (actin, myosin, sarcoplasmic proteins, etc.).

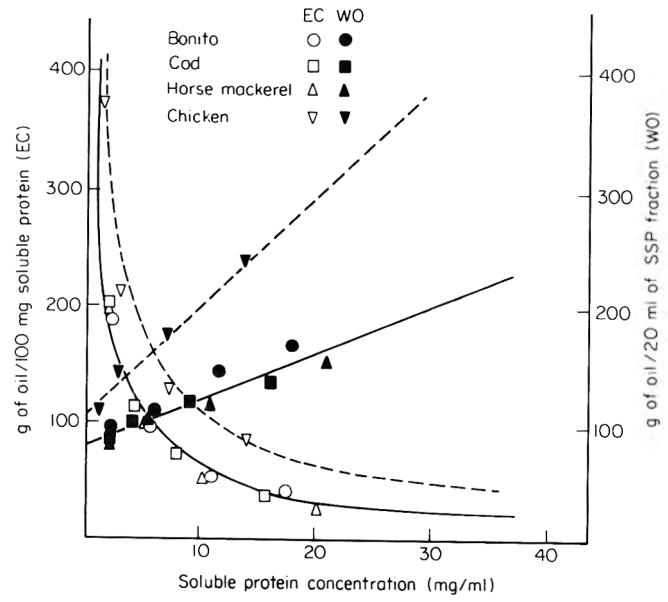


**Figure 4.** Emulsifying capacity (EC: g of oil/100 mg of soluble protein) and weight of oil (WO: g of oil/20 ml SSP fraction) *versus* soluble myofibrillar protein (SSP) concentration (mg/ml)

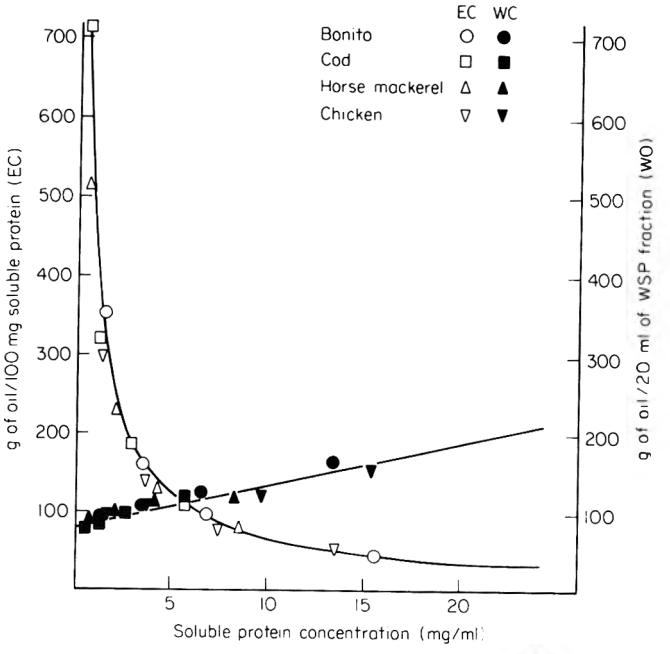


**Figure 5.** Emulsifying capacity (EC: g of oil/100 mg of soluble protein) and weight of oil (WO: g of oil/20 ml WSP fraction) *versus* water soluble protein (WSP) concentration (mg/ml).

The increase in EC as protein concentration decreases may be due to the fact that, as protein concentration drops, a greater degree of unfolding of polypeptides occurs during the shearing involved in the emulsifying process, and this is aided by

**Table 1.** Percent composition of the different protein fractions for the species studied.

| | Percent protein in muscle | | | |
|---|---|---|---|---|
| Fraction | Bonito | Cod | Horse mackerel | Chicken |
| H<br>Ha | 18.1 | 13.7 | 15.0 | 17.9 |
| SPP | 13.2 | 10.1 | 13.0 | 11.4 |
| WSP | 4.9 | 2.0 | 3.0 | 5.3 |

**Table 2.** Effect of pH on protein solubility (PS)

| | Percent protein solubility | | | |
|---|---|---|---|---|
| | Bonito | Cod | Horse mackerel | Chicken |
| H | 78.5 (5.87)* | 62.4 (6.34) | 73.1 (6.19) | 61.5 (5.77) |
| Ha | 85.6 | 62.4 | 81.3 | 61.4 |

*Figures in parentheses indicate the pH of the muscle homogenates.

hydrophobic association of the peptide chains with the liquid droplets, so that the net result is that a much greater volume/surface area of protein is made available and emulsifying efficiency is enhanced (Kinsella, 1976).

Plotting the results for the weight of emulsified oil per 20 ml of protein extract (WO) *versus* the soluble protein concentration in each fraction yielded linear relationships (Figs 3–5). Similar relationships have been described by Acton & Saffle (1972), Gillet *et al.* (1977), and others. These two ways of expressing emulsifying ability (EC and WO) are different and not directly comparable forms of expressing the same results, which on occasion has led to mistaken interpretations in the literature.

On analysing the various fractions studied, it can be observed that variations in the emulsifying ability at different soluble protein concentrations for the Ha and SSP fractions are similar for all three fish species, enabling the EC results for each fraction to be fitted by a single power curve ($r^2 = 0.98$; $P < 0.01$), while the WO results fit a single regression line ($r^2 > 0.85$; $P < 0.01$). The EC for the Ha and SSP frctions obtained from chicken muscle was higher than the EC for the fish fractions at all the concentrations studied. However, in the case of the WSP fraction, no differences were found which could be attributed to the muscle tissue source, hence the EC results for all four species were fitted by a single power curve ($r^2 = 0.99$; $P < 0.01$), and the WO results were fitted using a single regression line ($r^2 = 0.95$; $P < 0.01$) (Fig. 5).

The effect of protein concentration on the fat binding capacity differed according to the source fraction and type of muscle. Comparing the emulsifying capacities for the different fractions, it can be seen that in fish, at soluble protein concentrations less than or equal to 5 mg/ml, the WSP fractions exhibits a higher EC than do the Ha and SSP fractions. Similar results were obtained by Tsai *et al.* (1972) for pork muscle. At concentrations less than or equal to 5 mg/ml, the EC for chicken muscle extracts was highest for the Ha fraction. The WSP fraction had the lowest EC over the range of concentrations analysed, and, while such behaviour is commonly accepted as typical (Saffle, 1968; Schut, 1976; Kijowski & Niewiarowicz, 1978; and others), other authors (Gaska & Regenstein, 1982b) have pointed out that these sarcoplasmic proteins do not play a role in emulsion formation.

The superior emulsifying properties of the chicken muscle Ha extract compared to that from fish is due chiefly to the differing characteristics of the SSP fractions, since, as mentioned earlier, the emulsifying properties of the WSP fractions from all four species studied were identical (Figs 3–5).

It can thus be concluded that the weight of emulsified oil (WO) depends basically on two factors: the fraction used and the concentration of soluble protein. Variations among the fish species were due to varying soluble protein concentrations, whereas differences between the fish and the chicken muscle extracts were due to both factors.

Gillet *et al.* (1977) noted that the fat binding capacity of various protein extracts from livestock and poultry muscle depends on the soluble protein concentration rather than on the source. In the present experiment similar findings were recorded for the sarcoplasmic proteins from the four species considered, but in the case of the Ha and SSP fractions, this held true only for the fish. This behaviour would enable the soluble protein concentration to be calculated simply on the basis of the WO value. However, Carpenter & Saffle (1964) pointed out that soluble protein is not the only indicator for estimating the value of meat as an emulsifying agent; rather, there are factors inherent within the soluble protein which affect the emulsifying capacity.

The differing behaviour of the myofibrillar proteins from chicken and from fish muscle may be due to the fact that, because these latter are less stable (Matsumoto,

1980), they undergo certain interactions that reduce their emulsifying ability. In the case of chicken muscle, these interactions are probably not very intense, hence the effective protein concentration is higher than for fish muscle, thus giving it superior fat binding properties. Moreover, the interactions are minor in the case of the sarcoplasmic proteins (basically globular), which, coupled with the fact that at equal concentrations the number of molecules is greater, would in fish make the emulsifying properties of this fraction superior to those of the SSP.

As protein concentration increases, a point is reached when the system becomes overloaded, and the emulsifying capacity no longer depends on either the concentration or on the type of protein.

The factors affecting protein solubility also exert an influence on the emulsifying properties. The increase in pH brought about by adjusting it to 6.5–6.7 improved the fat binding capacity (WO) (Table 3). In the case of fish muscle, this effect can be attributed chiefly to the resulting protein solubilization (Table 2). There is a linear relationship between the amount of fat emulsified (WO) and the soluble protein concentration in

**Table 3.** Effect of pH on the amount of fat emulsified (in g) per 20 ml of protein extract (WO)

| mg total protein/ml | Bonito | | Cod | | Horse mackerel | | Chicken | |
|---|---|---|---|---|---|---|---|---|
| | 5 | 20 | 5 | 20 | 5 | 20 | 5 | 20 |
| H* | 105.3 | 155.2 | 94.9 | 135.3 | 97.3 | 145.5 | 137.6 | 205.5 |
| Ha (pH 6.5–6.7) | 123.2 | 180.0 | 99.4 | 137.2 | 106.2 | 159.6 | 159.1 | 225.2 |

*The pH for each species in given in Table 2.

mg/ml (S) ($WO = 4.54S + 85.86$; $r^2 = 0.94$; $P < 0.01$). In the conditions of the experiment, this equation appeared to be unrelated to the pH (two pH levels per species) and also unrelated to the source of the protein homogenates (three species).

In the case of fish muscle, the WO for the homogenate Ha can be calculated on the basis of the amount of soluble myofibrillar and sarcoplasmic protein present using the equation

$$WO_{Ha} = 89.6 + 4.01X_s + 5.91X_w,$$

where: $X_s$ = soluble myofibrillar protein concentration in the Ha extract, and $X_w$ = sarcoplasmic protein concentration in the Ha extract.

The coefficients for $X_s$ and $X_w$ are the slopes of the regression lines for WO *versus* soluble protein concentration for the SSP (Fig. 4) and WSP (Fig. 5) fractions, respectively. The above equation suggests that, in the case of the homogenate Ha, variations in the WO values according to the soluble protein concentration can be explained in terms of the combined effects of both fractions.

*Apparent viscosity*

Figures 6–9 show apparent viscosity *versus* protein concentration for the different fractions from each of the species. As can be observed in the figures, each of the plots conforms to a straight line for the range of concentrations studied, with a coefficient of determination of $r^2 > 0.90$ ($P < 0.01$).

In each case the regression lines cross the abscissa at a threshold concentration level, varying slightly for each species and fraction, below which no viscosity was observable

in the conditions of the experiment. The fact that viscosity for the WSP fraction did not register the measurement scale in the conditions employed might be due to a protein concentration close to the minimum threshold concentration referred to above or to the fact that the axial ratio for the sarcoplasmic protein molecules is lower than for the myofibrillar protein molecules.

Certain authors have found non-linear relationships between viscosity and protein concentration for proteins from different sources (Hamm, 1975; Hermansson, 1975; Kinsella, 1976; Pradipasena & Rha, 1977), though these findings were made under different test conditions than those in the present instance. Nonetheless, when working with low concentrations of myofibrillar fish proteins, other researchers have found linear relationships (Tomioka *et al.*, 1974).

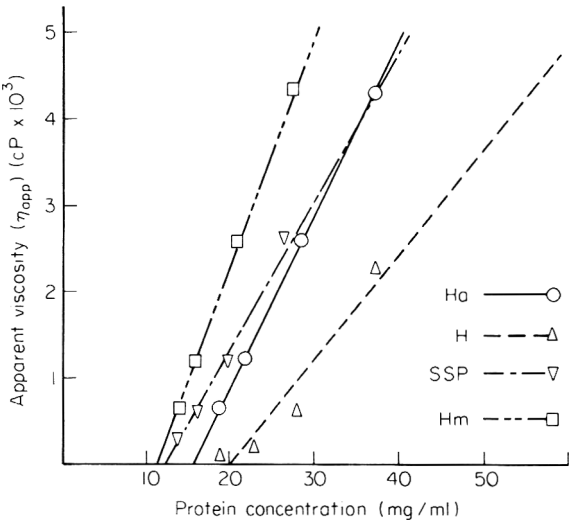


**Figure 6.** Bonito: apparent viscosity (cP) *versus* protein concentration (mg/ml); H and Ha extract protein concentration given in mg of total protein, Hm and SSP extract protein concentration given in mg of myofibrillar protein.

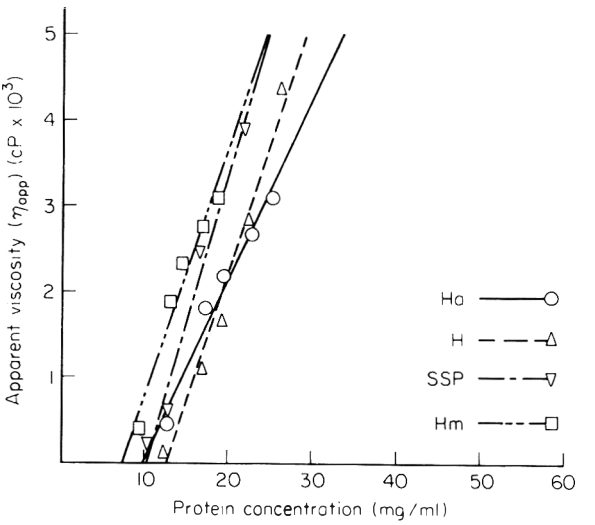


**Figure 7.** Cod: apparent viscosity (cP) *versus* protein concentration (mg/ml): H and Ha extract protein concentration given in mg of total protein, Hm and SSP extract protein concentration given in mg of myofibrillar protein.

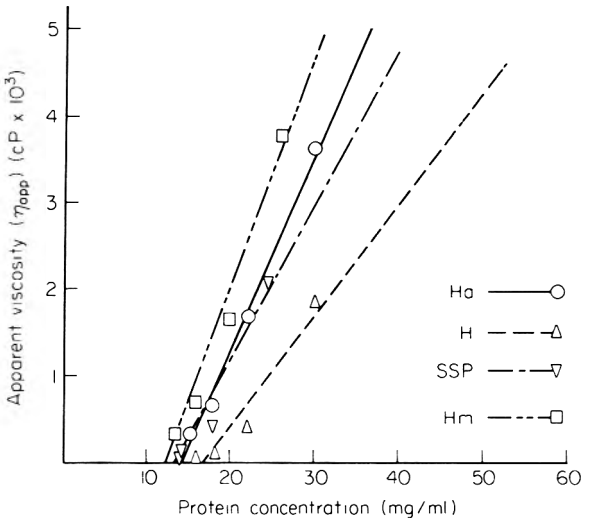


**Figure 8.** Horse mackerel: apparent viscosity (cP) *versus* protein concentration (mg/ml); H and Ha extract protein concentration given in mg of total protein, Hm and SSP extract protein concentration given in mg of myofibrillar protein.

Comparing the regression lines for $\eta_{app}$ *versus* protein concentration for the different species (Figs 6–9), it can be seen that, at equal concentrations, viscosity is highest in the chicken muscle protein fractions. Of the fish, cod muscle protein fractions exhibited higher viscosity values than did those from the other two species, for which the differences were not significant. Furthermore, when the pH of the homogenates (Ha fractions) was adjusted to 6.5–6.7, viscosity was higher at any given protein concentration level than when the pH was not adjusted (H fractions). Working with cod muscle, Nishimoto & Love (1974) achieved similar results. The increase in viscosity occurring as the protein moves away from its isoelectric point is caused by the state of

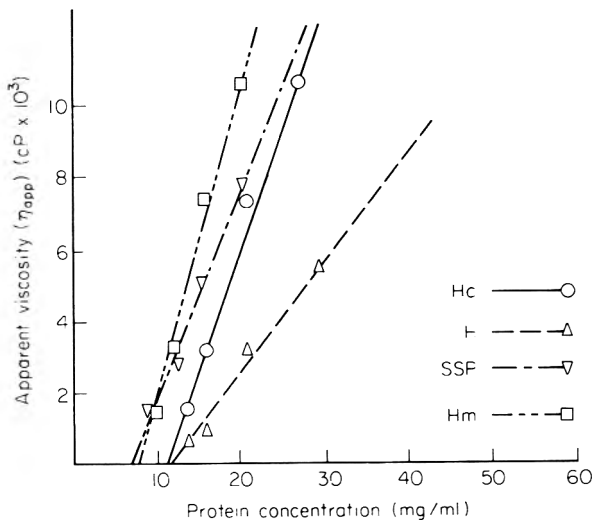


**Figure 9.** Chicken: apparent viscosity (cP) *versus* protein concentrat on (mg/ml); H and Ha extract protein concentration given in mg of total protein, Hm and SSP extract protein concentration given in mg of myofibrillar protein.

swelling of the protein molecule, which makes hydrophilic groups available (Toth & Hamm, 1968). This could be the reason why, in the three types of fish muscle, the regression lines tend to be similar when the pH of the muscle homogenates (Ha fractions) is adjusted. Differences still observable after the pH has been adjusted to 6.5–6.7 are the result of specific differences in the individual myosystems. The increase in viscosity might not take place in a different pH range, since it has been found that, for certain proteins, viscosity peaks at a given pH level, depending on the protein involved (Hermansson, 1975; Launay & Lisch, 1979; Lee & Rha, 1979).

Examination of protein solubility in 5% NaCl and viscosity values for the different species shows that PS is lower for chicken and cod muscle than for bonito and horse mackerel muscle (Table 2), and the viscosity of the Ha homogenates is higher (Figs 6–9). In view of the fact that the degree of protein solubility in 5% NaCl solutions provides an indication of the extent of protein aggregation, the higher viscosity levels exhibited by the latter species may in part be due to the larger size of the aggregates compared with molecules in the native state. However, in an earlier study (Jiménez-Colmenero & Borderías, 1983), it was noted that the viscosity of frozen fish muscle homogenates decreased with time, and the viscosity was correlated to a reduction in protein solubility and hence to greater protein aggregation. A possible explanation for these seemingly dissimilar findings may lie in the fact that protein aggregation is not a single state but instead corresponds to all types of protein–protein interaction (Hermansson, 1979); consequently, an initial stage might give rise to aggregates containing few cross-links, forming larger particles (Ohnishi, Tsuchiya & Matsumoto, 1978) still possessing a high water binding capacity; over time further protein–protein interaction might give rise to aggregates with only limited water binding capacity. Such a mechanism would be consistent with the changes taking place in the emulsifying properties during frozen storage, varying according to the species, concentration, and protein fraction.

In order to study the role of the SSP viscosity in the total viscosity of the Ha homogenates, the viscosity values for Ha were replotted in function of the SSP in Ha (from Table 1), to give the regression lines Hm in Figs 6–9. For all the species, the plots of these Hm regression lines indicate a shift to the left, which would imply that the myofibrillar protein is the majority component in the viscosity of the Ha homogenates, although other contributing factors increase the total homogenate viscosity somewhat.

*Relationship between emulsifying ability and apparent viscosity in the Ha homogenates*

An analysis was performed to determine the relationship between apparent viscosity and the amount of fat emulsified per 20 ml of protein extract (WO) at a given protein concentration level. The regression equations for each species are shown below:

Bonito $\quad WO = 0.017\eta_{app} + 166.531 \; (r^2 = 0.99; P < 0.01).$
Cod $\quad WO = 0.008\eta_{app} + 123.442 \; (r^2 = 0.99; P < 0.01).$
Horse mackerel $\quad WO = 0.014\eta_{app} + 140.937 \; (r^2 = 0.99; P < 0.01).$
Chicken $\quad WO = 0.006\eta_{app} + 203.63 \; (r^2 = 0.93; P < 0.01).$

The above equations apply to protein concentrations greater than 15 mg/ml. This limit is the minimum concentration at which viscosity readings can be obtained using the measurement scale under the conditions of the present experiment. The upper limit for protein concentration at which the equations were applicable in the experimental conditions described was between 30 and 40 mg/ml, depending moreover on the

conditions of extraction within each myosystem. Using the above relations the fat binding capacity (expressed as WO) can be calculated from the apparent viscosity ($\eta_{app}$), which is easier to measure.

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# A method of protein extraction from chicken bone residue and the chemical and electrophoretic characteristics of the extract

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## Summary

The study concerns the principle of the protein recovery from bone residue (BR) after mechanical deboning of chicken meat. The BR contained 36% of dry matter, 20% of total protein of which 35–40% constituted collagen, approximately 9% of fat, 7% of ash, and 0.2% of haem pigments. The BR was extracted with 6% water solution of NaCl with addition of 0.015% $NaNO_2$, and 0.05% ascorbate in relation to BR. The various solvent/BR ratios, additional homogenization, and addition of 0.3% sodium phyrophosphate were also investigated. The highest protein concentration in the extract was established at solvent/BR ratio 1.5 : 1, with additional homogenization of BR, and in the presence of $Na_4P_2O_7$. In the best conditions the extract contained 3.4% of protein which constituted almost 18% of total BR protein. It was shown that the deboning process involved the degradation of muscle proteins originating from BR. The SDS-PAAG electrophoresis showed that the protein with the mol. wt $55–48.10^3$ daltons predominated among the water soluble proteins while in the salt soluble proteins the highest content of proteins with the mol. wt of $47–42.10^3$ and $21–17.10^3$ daltons was found.

## Introduction

Production of mechanically deboned poultry meat (MDPM) is a long established common practice. The most frequently utilized material for deboning are the backs and necks of chicken, and the turkey frames, as well as the whole carcasses of out-of-lay hens. Sometimes the wings and the skin are also included in deboning material. After deboning two products (fractions) are usually obtained, the first main product (MDPM), and the second, called bone residue (BR). The latter fraction, because of high content of bone particles, is utilized for bone meal manufacture.

Generally, the yield of the first product, depending on the deboning machine and on the kind of material used for deboning, ranges from 50 to 80%. Thus, the BR fraction yielded from 50 to 20%, respectively. The BR, besides the bone particles, contains a notable amount of muscle, connective tissues and fat. The muscle tissue remaining in BR represents a valuable source of proteins which could be recovered and used in poultry meat products processing.

Young (1976) elaborated the method for preparation of protein isolate from BR of broiler necks and backs. This BR contained 40% solids of which 43% was protein, 32% fat, and 25% ash. The extraction of protein was performed with 0.1 M Na malate at pH 7

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and sufficient NaCl to make the ionic strength of the solvent 0.5. After blending the mixture was filtered and then diluted to ionic strength 0.2. The precipitate was washed with water and then freeze dried. A typical preparation contained 4.8% of BR. Protein solubility and emulsifying capacity of isolate were investigated. The author concluded that the protein isolate prepared from BR might have some use as a food ingredient, but more data are necessary on its functional characteristics.

Lawrence, Jelen & Fedec (1982) reported on the pilot plant extraction of protein from mechanically separated poultry residue (MSPR). The concept for alkali extraction (pH 10.5), and acid precipitation (pH 5.3) of protein was elaborated earlier (Jelen, Earle & Edwardson, 1979). The yield (kg) of precipitate, based on 100 kg of raw bone material was 24. Proximate composition of protein precipitate was as follows: moisture 77%, protein 8.5%, and fat 13%. The authors discussed some difficulties which occurred during the successive processing steps. The majority of the increased total solids content in both precipitate and the supernatant, in comparison with laboratory procedure, was due to the increased fat content.

Microbial analyses indicate that alkaline extraction of protein from MSPR using the suggested operating conditions does not produce any public health hazard (Jackson, Consolacion & Jelen, 1982).

Lawrence & Jelen (1982) reported also that the proposed alkali extraction procedure would not produce lysino-alanine in the protein extract under technologically optimal conditions.

The objective of this study was the elaboration of the method of protein extraction from chicken bone residue and chemical and electrophoretic characterization of the extract.

## Materials and methods

The backs, necks and wings, with the skin present, from broilers and/or from laying hens were mechanically deboned on a Paoli Model 21 A deboner. The BR fraction was immediately frozen and stored at −18°C. In this raw material the content of dry matter, fat (Soxhlet method), total protein (Kjeldahl), collagen (hydroxy-proline determination according to Möhler & Antonacopoulos, 1957), bone particles (Gerats & Terbijhe, 1975) and haem pigments using the procedure of Krzywicki (1982) were determined.

The BR was subjected to protein extraction with various amounts of 6% NaCl water solution as is shown in Fig. 1. In some experiments the BR was additionally comminuted (homogenized) on a colloid mill. The effect of the 0.3% $Na_4P_2O_7$ added to the solvent on the protein extractability was also investigated. During the mixing with the solvent the $NaNO_2$ (0.015%) and ascorbate (0.05%), in relation to BR weight, were added. In the extract the dry matter, protein, fat, bone particles, haem pigments, and non-protein nitrogen (Kijowski and Niewiarowicz, 1980) after precipitation of protein with TCA were determined.

The procedure for quantitative characteristics of protein in the extract was as follows: 50 ml of the extract in a 'Visking' dialysis tube 36/32 (Serva) was dialysed for 16 hr at 4°C against 1500 ml of redistilled water. The dialysate was centrifuged at 8000 rev/min for 30 min. After centrifugation the precipitate (proteins soluble in salt) and the supernatant (proteins soluble in water) were weighed and measured volumetrically. In both fractions the content of protein was determined by Kjeldhal method.

For electrophoretic protein separation and identification the extract in 6% NaCl was

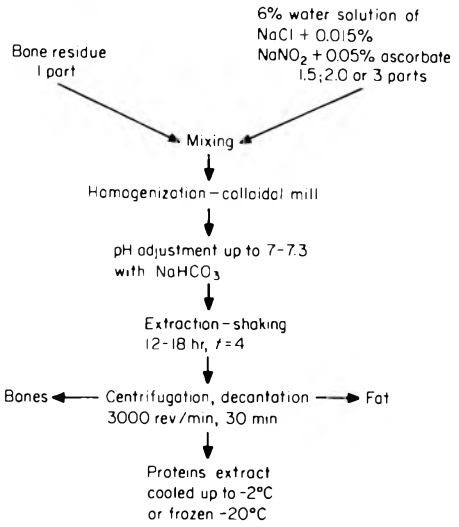


**Figure 1.** General procedure for protein extraction from poultry bone residue.

clarified with the centrifuge, type VAC 601 (Janetzki), for 40 min at 60000 g. The precipitate and the supernatant were dialysed as shown above. After dialysis the precipitate was twice washed with 1% solution of Triton-100. Then it was dissolved in the solution composed of 1 ml 4% water solution of SDS, 1 ml of buffer and 1 mg of ditiotreitol. The quantity of the precipitate was such adjusted that concentration of the protein was 20 mg/ml of solvent. The supernatant which contained the proteins soluble in water was condensed with the polyethylenglycol 20 000 and then the parallel samples from precipitate were prepared for further characteristics. The electrophoresis was carried out according to procedure reported by Kijowski (1984).

## Results and discussion

Composition of BR from broilers and hens is presented in Table 1. Proteins constitute the majority of dry matter and this corresponds to the data reported by MacNeil (1981). Among these proteins 35–30% belong to collagen originating from bone tissue, skin and tendons. At such a high level of collagen, the generally accepted coefficient 6.25 for calculation the content of crude protein is not adequate, because the coefficient for collagen is 5.55. Taking into consideration the above mentioned collagen content in BR, the coefficient 6.05 seems to be more suitable for the proper calculation of total protein amount in BR. In this case the content of total protein in BR is lower by about

**Table 1.** Composition of bone residue from broilers and hens (%)

| Type of meat | Dry matter | Total protein (N×6.25) | Total protein (N×6.05) | Collagen % of total protein | Fat | Ash | Haem pigments | Bone particles (raw bones) |
|---|---|---|---|---|---|---|---|---|
| Chicken broilers* | 36.6 | 20.0 | 19.2 | 35.3 | 10.1 | 6.6 | 0.19 | 33.0 |
| Hens, out-of-lay* | 36.1 | 20.9 | 19.9 | 40.2 | 7.8 | 7.2 | 0.22 | 34.1 |

*Every value represents the mean of three observations.

0.9% (Table 1). The level of fat is 8–10% constituting approximately one third of the total fat in the deboned raw material. The haem pigment content is about four times higher than that found in breast muscle of broilers and hens (Pikul, Niewiarowicz & Pospieszna, 1982). It is interesting that in the BR about one third of its weight is attributed to the bone particles of raw bones. Generally, the differences in chemical composition between the BR of broiler and hen are negligible.

Table 2 shows the effect of various extraction conditions on the chemical composition of the extract from broiler BR. At the lower ratio of solvent to BR the higher concentration of protein in the extract was achieved. Additional homogenization of BR before the extraction gave better protein recovery, ranging from 20 to 30%. Also the presence of pyrophosphate significantly increased the solubility of protein from BR. This can be partly attributed to the shift towards the higher pH value of the extract. The highest protein concentration in the extract was obtained using a solvent/BR ratio of 1.5:1, and additional homogenization in the presence of $Na_4P_2O_7$. At a lower solvent/BR ratio a little more fat remained in the protein after its separation from the extract, but no more than 0.5%. The content of bone particles in the extract is negligible.

**Table 2.** Effect of various extraction conditions on the chemical composition of extract from broiler residue

| Additional homogenization | Solvent/bone residue ratio (W/V) | Dry matter (%) | Total protein (%) | Fat (%) | Bones (%) | Haem pigments (%) |
|---|---|---|---|---|---|---|
| No | 3:1 | 4.1 | 1.3[a] | 0.2 | 0.011 | 0.13 |
| Yes | 3:1 | 4.5 | 1.5[b] | 0.2 | 0.012 | 0.11 |
| No | 2:1 | 4.9 | 1.8[c] | 0.3 | 0.016 | 0.17 |
| Yes | 2:1 | 5.1 | 2.4[d] | 0.3 | 0.019 | 0.22 |
| No | 1.5:1 | 7.3 | 2.5[d] | 0.4 | 0.020 | 0.23 |
| Yes | 1.5:1 | 7.9 | 3.0[e] | 0.5 | 0.025 | 0.27 |
| Yes* | 1.5:1 | 8.6 | 3.4[f] | 0.5 | 0.021 | 0.28 |

* With the addition of 0.3% $Na_4P_2O_7$ (pH = 7.03), while in other trials the pH was 6.8–6.9. The precipitate after centrifugation contained approximately 20.2% of dry matter, 9.1% of total protein and 4.7% of fat.

[a–f] Figures in the column marked with different letters are significantly different at $P = 0.05$.

The colour of the extract was intensive pink, especially at lower solvent/BR ratio, from higher haem pigment concentration. Depending on the method of extraction, 6.7–17.6% of total protein from BR may be recovered. According to Young (1976) the yield of protein isolate was 11–17% in relation to total protein content in chicken BR. However, Lawrence *et al.* (1982) reported that over 18% of the wasted protein in BR is alkali extractable. We consider that the yield of proteins depends, besides the method of extraction (Table 3), on the raw material used and the type of deboning machine.

The electrophoretic separation and identification of protein was performed on 8% polyacrylamide gel with SDS after protein fractionation into proteins soluble in water (sarcoplasmic) and proteins soluble in salt (miofibrillar). The composition of these two fractions is presented in Table 4. Some distinct differences between the electrophoretic patterns of proteins from broiler BR and from broiler breast meat aged for 24 hr were detected, as shown in the Fig. 2. Comparison of these densitograms indicates the differences in the number of particular peaks. This may be due to

**Table 3.** Effect of various extraction conditions on the percentage of proteins soluble in water and soluble in salt.

| | Solvent/bone residue ratio (W/V)* | | | Solvent/bone residue ratio (W/V)† | | |
|---|---|---|---|---|---|---|
| | 3:1 | 2:1 | 1.5:1 | 3:1 | 2:1 | 1.5:1 |
| Total protein | 1.25[a] | 1.80[c] | 2.52[d] | 1.48[b] | 2.36[d] | 2.98[e] |
| Protein soluble in water | 0.63[a] | 0.92[b] | 1.10[b] | 0.71[a] | 1.09[b] | 1.12[b] |
| Protein soluble in salt | 0.51[a] | 0.78[b] | 1.18[c] | 0.75[b] | 1.20[c] | 1.80[d] |

* Non-homogenized.

† Homogenized in colloidal mill.

Mean value from four parallel determinations.

[a–e] Figures in the rows marked with different letters are significantly different at $P = 0.05$.

the occurrence in BR of proteins originating not only from meat muscles but also from the skin, marrow and bones. Moreover, the possibility of enzymic protein breakdown should be taken into consideration.

The deboning process causes destruction of deboned material which results in the liberation of many lysosomal enzymes. During the protein extraction with salt solvent of pH 6.8–7.0, the neutral proteases may be particularly active, activated by $Ca^{++}$, $Mg^{++}$, $Fe^{++}$, $Fe^{+++}$ and $Na^{+}$ ions, which are present in high concentration in poultry meat residue. This suggestion is supported by the high level of non-protein nitrogen found in the extract from BR, amounting on average to 25%, while in the chicken pectoral muscle the amount of non-protein nitrogen is only about 10.5% of total nitrogen (Kijowski & Niewiarowicz, 1980).

Proteolysis may involve a noticeable change in myosin. On the densitogram scan from aged chicken breast muscle the peak of myosin occupied 30–36% of all the area,

**Table 4.** Electrophoretic characteristics of protein extracted from chicken bone residue*

| Solvent (6% NaCl)/bone residue ratio | | | Mol. wt | |
|---|---|---|---|---|
| 3:1 | 2:1 | 1.5:1 | (daltons × $10^3$) | Identified muscle proteins |
| Proteins soluble in water† | | | | |
| 20.1‡ | 19.1 | 21.0 | 70–60 | — |
| 57.2 | 60.1 | 59.0 | 55–48 | GAPDH, LDH, enolase, aldolase |
| 22.1 | 21.8 | 23.9 | 17–15 | myoglobin, haemoglobin |
| Proteins soluble in salt† | | | | |
| 10.6 | 9.9 | 11.0 | 220–200 | Myosin heavy chains |
| 10.8 | 11.3 | 9.8 | 160 | M protein |
| 7.1 | 8.2 | 6.0 | 140 | C protein |
| 31.8 | 26.1 | 32.1 | 47–42 | Actin |
| 11.8 | 10.4 | 10.5 | 38 | Tropomyosin |
| 27.1 | 31.5 | 30.8 | 21–17 | Light chains of myosin and troponin |

* Bone residue homogenized in colloid mill.

† Protein bands as percent of total densitometric scan area.

‡ Every value represents mean of three observations.

while in the case of densitograms of proteins extracted from chicken BR the adequate peak area amounted only to 10% in relation to all other peak areas. Probably the heavy chains of myosin were broken down to the subunits with the mol. wt $200-140\times10^3$ daltons (Fig. 2), found also in aged broiler breast muscle (Kijowski, 1984). Penny (1980) pointed out the pronounced breakdown of different myofibrillar proteins by the endogenic neutral proteases, especially CAF, in the conditioned meat.

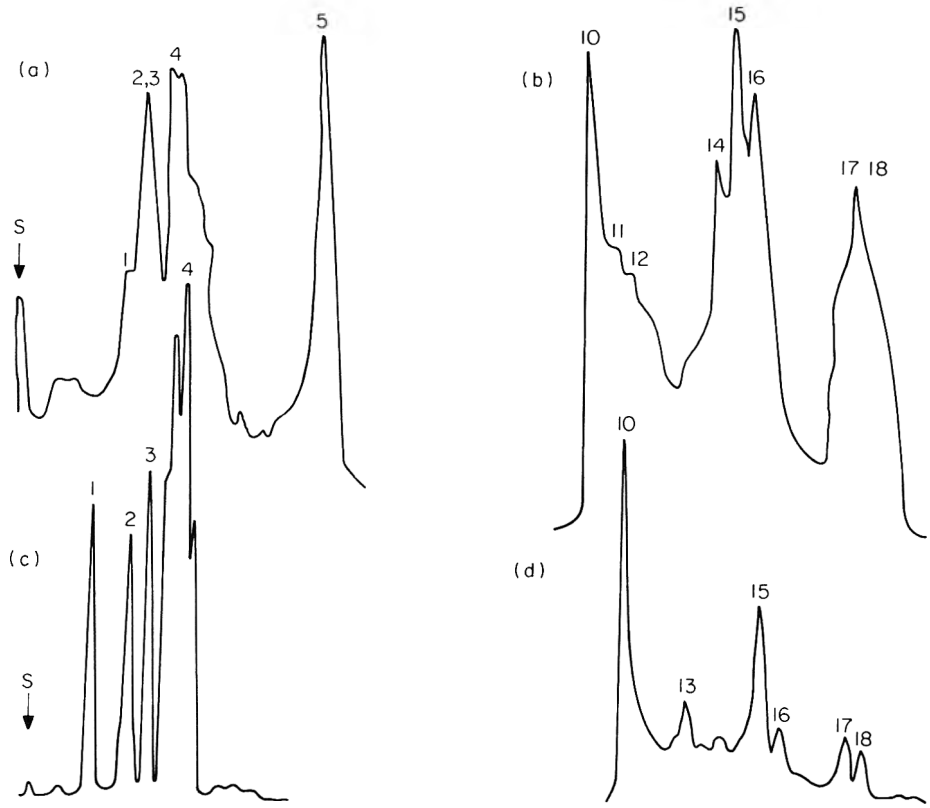


**Figure 2.** Densitometric tracing of (a) water soluble proteins from bone residue extract, (b) salt soluble proteins from bone residue extract, (c) water soluble proteins from 24 hr aged broiler breast meat, and (d) salt soluble proteins from 24 hr aged broiler meat. S = start, 1 = phosphorylase, 2 and 3 = enolase, aldolase, 4 = GAPDH, 5 = myoglobin, haemoglobin, 10 = myosin heavy chains, 11 = M protein, 12 = C protein, 13 = alpha actin, 14 = $55\cdot10^3$ daltons protein, 15 = actin, 16 = tropomyosin, 17 and 18 = light chains of myosin and troponin.

The visible coloured background (blue band) on the whole length of chromatogram also supports the suggestion that the high molecular proteins are degraded to the minor subunits in the chicken BR extract. A similar blue background was also observed by de Wreede, Stegemann & Heinert (1982) on electrophoretic patterns of proteins from strongly heated meat. Additionally, we found that the start line is not sharply separated from the band of the myosin heavy chains. This may be caused by the increased participation of the protein subunits in the densitometric scan.

The creation of degraded proteins in chicken BR was also present in the sarcoplasmic proteins soluble in water. The proteins with the mol. wt $55-48\cdot10^3$ daltons

amounted to more than half of the area in relation to all proteins soluble in water. This band was located at the same place of the chromatogram as the band of enzymic proteins from glycolytic cycle. Moreover, the significant increase of the peak related to the proteins of mol. wt $17 \cdot 10^3$ daltons was noted. This may be attributed to the higher content of the haem pigments originating from bone marrow.

The majority of proteins soluble in salt was recovered in the bands of mol. wt $47-38 \cdot 10^3$ and $21-17 \cdot 10^3$ daltons (Fig. 2). Among these proteins actin, tropomyosin and the light chains of myosin and troponin, respectively, should occur. Probably in this region the degraded products of the heavy chains of myosin are also located.

Further studies on the technical feasibility of the extraction method described for protein recovery from poultry bone residue are being conducted in a poultry processing plant.

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# Use of protein extract from bone residue in poultry meat processing

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## Summary

The use of protein extract from chicken bone residue (BR) in poultry meat processing was investigated. The protein was extracted with 6% of sodium chloride and some additives according to the procedure reported earlier. The extract contained 3.0–3.4% of protein and 0.5% of fat. In two types of meat mixture formulation (A and B) the added water was substituted with protein extract, and two basic types of comminuted chicken sausages were manufactured. The results showed the beneficial effect of protein extract from BR on the yield, the meat emulsion stability, the rheological properties, the colour, and the sensory characteristics of the experimental sausages. This effect can be attributed to the good solubility and to the high content of myofibrillar proteins and haem pigments in the extract. The bacterial count of freshly obtained extract is rather low. The frozen storage of the extract does not diminish its excellent functional properties. Some experiments carried out with fish sausage production were very promising. The authors concluded that utilization of the waste proteins from poultry BR for poultry, meat and fish products is important and worth industrial application.

## Introduction

Young (1976), MacNeil (1981), Lawrence, Jelen & Fedec (1982), Kijowski & Niewiarowicz (1985) reported that poultry bone residue (BR) originating from deboning machines still contains considerable amounts of meat proteins. Young (1976), Jelen, Earle & Edwardson (1982) and Kijowski & Niewiarowicz (1985) worked out the method for protein recovery from poultry BR. But only Jelen, Lawrence & Cerrone (1982) used alkali protein extract from BR in all-chicken luncheon meats as a replacement at 0–40% for industrial deboned chicken meat. They found that the differences in texture and flavour between the experimental products and a commercial all-chicken luncheon meat were rather small even at the highest replacement levels used.

Lawrence *et al.* (1982) reported on the pilot plant process for alkali extraction and acid precipitation of protein from poultry BR. The authors concluded that this process would not create any unusual problems from the point of view of health protection, but further research may be necessary to overcome the technological problems indicated by this pilot plant study.

The objective of our study was the evaluation of the technological effect of the protein extract from chicken bone residue on the model poultry sausages.

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## Materials and methods

Bone residue was obtained from deboning of chicken backs and necks on the Paoli, Model 21 A machine. The proteins were extracted according to the procedure of Kijowski & Niewiarowicz (1985). The BR was first additionally comminuted in a colloid mill, and then the proteins were extracted with 6% of water sodium chloride in the presence of 0.015% $NaNO_2$, 0.05% of sodium ascorbate, and 0.3% of sodium pyrophosphate. The solvent/BR ratio was 1.5 : 1. In the extract the dry matter, total protein, fat and pH were determined. The microbial quality of the extract was assessed by a standard plate count (SPC) and a coliform count. The tests were performed as described by Burbianka, Pliszka & Burzyńska (1983), using nutrient agar (Difco) for SPC and the 3 tube most probable number technique for coliforms.

*Preparation of experimental sausage*

There were two basic types of model sausage. The meat mixture of type A sausage contained 50% of chicken breast and leg meat with skin, 18% of pork lard, and 32% of added water. The formulation of type B sausage was 50% of chicken meat, 16% of pork jowl, and 34% of water. To both types of sausages 0.1% of seasoning was added. The meat was dry cured with 2% of NaCl and 0.015% of $NaNO_2$ at 6°C for 24 hr. All ingredients with or without the extract were chopped for 5 min, and then for an additional 5 min with fat added. At the end of chopping the temperature of the meat mixture was 13.5 ± 1.5°C.

From meat mixture type A four different kinds of sausage were produced: $A_1$—control (basic composition as above), $A_2$—half of the added water was replaced with tested extract, $A_3$—all added water was replaced with extract, and $A_4$—all water was replaced with extract, to which the 0.3% pyrophosphate was added before the extraction procedure.

Experimental sausages from the basic formulation type B were as follows: $B_1$—control, $B_2$—all added water was substituted with extract, $B_3$—all water was replaced with extract, frozen and stored at −18°C for 14 days, and $B_4$—all water replaced with extract, which contained the texturized soya protein Purina-50 produced by Ralston Purina Co. (U.S.A.). The addition of texturized soya protein amounted to 60% in relation to the weight of meat.

All experimental meat mixtures were analysed for dry matter, total protein, fat, sodium chloride content, dynamic viscosity (Kijowski & Niewiarowicz, 1978b), and thermal stability of the meat mixture emulsion (Kijowski & Niewiarowicz, 1978a).

The meat mixture was stuffed into 35 mm diameter synthetic protein casings and the product was hot smoked for 70 min. The batches were then heated in a water bath at 70–72°C for 20 min to internal temperature of 70°C, monitored with Thermistor thermometers connected to an 'Elab' recorder, and then chilled in tap water. After storage for 12 hr at 4°C the following examinations were carried out.

The yield of sausages was calculated in relation to the weight of cured meat used. The objective rheological analyses were performed on an Instron TM-1140 with accessories according to the procedure described by Kijowski, Pikul & Niewiarowicz (1982). The abbreviations used have the following meaning: $L_c$—shearing resistance of the sample, core 25 mm diameter, in Newton ×cm, $L_t$—total compression resistance of 60% of core height (20 mm), $L_s/L_p$—expresses the ratio of work (energy) needed for the elastic deformation, to the work involving plastic deformation of sausage core.

Reflectance spectrophotometric measurement of colour parameters at 540 and 640

nm was performed according to simplified method of Tyszkiewicz (1969). A 'Spekol' spectrophotometer Carl Zeiss Jena (G.D.R.) fitted with a reflectance chamber Rd/O was used. The instrument was zeroed with a Mg block and the sample thickness was 5 mm, diameter 25 mm. The dominant wavelength ($\lambda_d$) and the luminance of photometric brightness ($Y$) of the colour was calculated from reflectance percentage by formulae elaborated for cured and heated comminuted meat products.

Sensory testing was performed by six qualified judges using a 5-point scale ranging from 1 (worst quality) to 5 (best quality). The following organoleptic characteristics were considered: colour on the cut, binding, juiciness, flavour, and general preferences.

## Results and discussion

The chemical composition and pH value of the meat bone residue (BR) extract used for the production of various experimental sausages are presented in Table 1. The data show that the addition of 0.3% $Na_4P_2O_7$ to the sodium chloride solvent improved the efficiency of protein extraction and raises the pH value of the extract. Standard microbial analyses indicated that the SPC of the protein extract from BR was $5.6 \cdot 10^4$–$2.8 \cdot 10^5$ and coliforms < 3/g. It appears that the use of extract of the quality investigated here in poultry meat processing should not pose any great problems from the health protection standpoint. The various formulations of meat mixtures (Table 2) distinctly affect their chemical composition and physical characteristics. The higher content of dry matter and fat in A rather than in B type meat mixture results from the presence of

**Table 1.** Composition and pH value of bone residue extracts used in experimental sausage production

| Extract used in sausage | Dry matter (%) | Protein N×6.25 (%) | Fat (%) | pH |
|---|---|---|---|---|
| $A_{1-3}$ | 7.9 | 3.0 | 0.5 | 6.80 |
| $A_4$ | 8.6 | 3.4 | 0.5 | 7.02 |
| $B_{1-4}$ | 8.0 | 3.03 | 0.52 | 6.82 |

**Table 2.** Chemical and physical characteristics of experimental meat mixture

| Meat mixture | Dry matter (%) | Protein N×6.25 (%) | Fat (%) | NaCl (%) | Emulsion stability (ml drip/100 g) | Dynamic viscosity ($Ns/m^2$) |
|---|---|---|---|---|---|---|
| Control ($A_1$) | 33.2 | 11.6 | 19.3 | 2.1 | $32.2^a$ | $2.1^a$ |
| Half extract ($A_2$) | 33.6 | 12.0 | 18.9 | 1.8 | $17.8^b$ | $3.0^b$ |
| Extract ($A_3$) | 34.1 | 13.1 | 19.4 | 1.7 | $8.9^c$ | $4.1^c$ |
| Extract + $Na_4P_2O_7$ ($A_4$) | 36.4 | 13.4 | 20.7 | 1.7 | $10.8^c$ | $4.9^d$ |
| Control ($B_1$) | 25.1 | 11.5 | 11.7 | 1.8 | $26.5^e$ | $1.1^e$ |
| Extract ($B_2$) | 24.8 | 12.3 | 11.4 | 1.7 | $19.9^f$ | $1.6^f$ |
| Frozen Extract ($B_3$) | 24.4 | 12.7 | 11.4 | 1.7 | $21.7^f$ | $1.6^f$ |
| Extract + soya protein ($B_4$) | 25.8 | 13.5 | 10.9 | 1.8 | $25.6^{g,e}$ | $1.3^g$ |

[a–d] Figures in the columns with the same superscript letters are not significantly different at $P = 0.05$.

chicken meat with skin and pork lard in A, while in B formulation the meat without skin and pork jowl was used. For both types of meat mixtures the content of protein was higher in those formulations where the added water was partially or fully substituted with meat BR extract and with soya protein.

At the full replacement of water with extract the increase of protein in meat mixture averaged from 1.2 to 2.0% in comparison with the control. The content of NaCl was kept at approximately the same level by the appropriate addition of salt in the chopping operation. The addition of protein extract from BR significantly increased the dynamic viscosity of the meat mixture. Particularly high viscosity was found at full substitution of water with protein extract and in the presence of $Na_4P_2O_7$ ($A_4$). Freezing and freeze storage of extract do not influence the viscosity of meat mixture ($B_3$), while the addition of soya protein decreased the viscosity of meat mixture ($B_4$). The thermal stability of meat mixtures measured by the amount of drip after heating up to 68°C was very much improved in all experiments where the extract was added. It was especially effective at the higher content of fat in meat mixture ($A_{2\_4}$).

In Table 3 the effect of protein extract from BR on some technological characteristics of experimental sausages is presented. The substitution of added water with protein extract distinctly raises the yield of ready-to-eat products, especially in the sausages made from A type meat mixture. The highest yield was noted for the sausage with full replacement of water with the extract and in the presence of pyrophosphate. In this case the yield increased up to 7.4% in comparison to the control. Among the sausages based on the meat mixture B the best yield was found for the sausage $B_4$, where the texturized soya protein was used. The use of freeze stored extract only slightly diminished the yield of the sausage $B_3$ in comparison with sausages containing extract $B_2$, which had not been frozen. It is interesting that the sausages with the higher content of fat ($A_3$ and $A_4$) showed a slightly higher yield than those with the lower fat level ($B_{2-4}$).

**Table 3.** Yield, rheology and colour of experimental sausages

| Type of sausage | Yield (%) | Rheological properties | | | Colour | |
|---|---|---|---|---|---|---|
| | | $L_c$ (N×cm) | $L_t = L_s + L_p$ (N×cm) | $L_s/L_p$ | $\lambda_d$ | $Y$ |
| Control ($A_1$) | 134.9 | $23.1^a$ | $8.1^a$ | $0.41^a$ | $581.1^a$ | $44.5^a$ |
| Half extract ($A_2$) | 137.5 | $25.3^b$ | $8.5^a$ | $0.84^b$ | $581.6^a$ | $44.6^a$ |
| Extract ($A_3$) | 140.9 | $30.9^c$ | $13.8^b$ | $0.84^b$ | $583.7^b$ | $37.8^b$ |
| Extract + $Na_4P_2O_7$ $A_4$ | 142.3 | $31.2^c$ | $13.6^b$ | $0.89^b$ | $584.2^b$ | $35.7^c$ |
| Control ($B_1$) | 135.7 | $29.8^e$ | $8.9^e$ | $0.45^e$ | $578.7^e$ | $46.6^e$ |
| Full extract ($B_2$) | 138.0 | $38.8^f$ | $13.0^f$ | $0.87^f$ | $582.0^f$ | $41.6^f$ |
| Frozen extract ($B_3$) | 135.2 | $37.7^f$ | $12.3^f$ | $0.78^f$ | $581.9^f$ | $40.2^f$ |
| Extract + soya protein ($B_4$) | 139.4 | $40.7^f$ | $9.1^e$ | $0.61^g$ | $580.2^g$ | $44.6^g$ |

$^{a-g}$See Table 2.

The full substitution of added water with the protein extract significantly improves all investigated rheological properties of ready-to-eat sausages made from both types of meat mixtures. Only for the sausage made with the addition of soya protein ($B_4$) was the value of $L_t$ (i.e. total compressing resistance) the same as for the control. The rise of the rheological properties characterizing the improvement of sausage texture does not mean the creation of undesirable hardness but is rather desirable for poultry comminuted products.

The results of colour measurements showed that the replacement of water with protein extract from BR produced redder sausages. The rise of $\lambda_d$ values is interpreted as the change of colour tint towards the pink-red colour, and the fall of $Y$ value (photometric brightness) as the intensification of the observed colour. The most favourable changes in colour were found in the sausages at full substitution of water with protein extract to which the $Na_4P_2O_7$ was added. The more intensive (dark) colour for $B_2$ and $B_3$ type sausages probably results from the lower content of fat, and in $B_4$ the soya protein might influence this phenomenon.

The organoleptic evaluation (Table 4) showed that in all cases the experimental sausages manufactured with the addition of protein extract scored significantly higher than the controls ($A_1$ and $B_1$). The sausages produced from the meat mixture of type B, with the lower content of fat were preferred by the consumer to those of type A with the higher amount of fat. It is interesting to note that judges preferred the sausages with the lower content of solids—i.e. with more moisture. Among the sausages of type B, the trial $B_4$ containing soya protein, scored lower. From the comparison of the instrumental analysis with organoleptic evaluation of experimental sausages it may be concluded that the measurements of the shearing resistance ($L_c$) correlated best with the consumer scoring. The sensory examination of colour, separately for type A and B sausages, indicated greater preference in sausages with the protein extract.

**Table 4.** Sensory evaluation for model sausages

| Type of sausage | Colour of cut | Consistence (binding) | Juiciness | Flavour | General preferences |
|---|---|---|---|---|---|
| Control ($A_1$) | $3.2^{a}$ | $1.7^{a}$ | $2.2^{a}$ | $2.2^{a}$ | $2.4^{a}$ |
| Half extract ($A_2$) | $4.0^{a,b}$ | $2.8^{b}$ | $3.6^{b}$ | $3.3^{b}$ | $3.3^{b}$ |
| Extract ($A_3$) | $4.6^{b}$ | $4.1^{c}$ | $4.4^{b}$ | $4.0^{c}$ | $4.3^{c}$ |
| Extract + $Na_4P_2O_7$ ($A_4$) | $4.7^{b}$ | $4.4^{c}$ | $3.9^{b}$ | $3.8^{c}$ | $4.1^{c}$ |
| Control ($B_1$) | $2.6^{e}$ | $3.4^{e}$ | $3.6^{e}$ | $3.1^{e}$ | $3.0^{e}$ |
| Extract ($B_2$) | $4.9^{f}$ | $4.7^{f}$ | $4.6^{f}$ | $4.6^{f}$ | $4.7^{f}$ |
| Frozen extract ($B_3$) | $5.0^{f}$ | $4.7^{f}$ | $4.5^{f}$ | $4.6^{f}$ | $4.7^{f}$ |
| Extract + soya protein ($B_4$) | $4.3^{f}$ | $4.1^{f}$ | $4.5^{f}$ | $3.8^{e}$ | $4.1^{g}$ |

[a–g]See Table 2.

Besides the main experiment described above, a trial with fish meat was also performed. The mixture formulation was similar to that of type A in which, instead of chicken meat, Baltic cod fillets were used. The replacement of added water with protein extract from BR beneficially influenced the quality of ready-to-eat fish sausage. The colour was distinctly changed from white-grey towards pink-red. The consistency became more elastic, the shearing resistance increased, and the flavour was more meaty. The use of protein extract from poultry BR in fish sausage production seems to be very promising, and worth investigation.

The authors consider that the beneficial effect of the protein extract from chicken BR on the yield and quality of comminuted chicken sausages results from the increase in the meat mixture of easy soluble proteins. These proteins contained high amounts of myofibrillar proteins, and the products of their partial degradation, of good functional properties and a high level of haem pigments. The microbial contamination of the extract is reasonable. Moreover, the frozen storage of this protein extract does not diminish its functional properties. The utilization of the waste proteins from poultry

bone residue for poultry meat and for fish products seems to be important from the economical point of view, and worth practical application.

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# Effect of recipes on crust formation and mutagenicity in meat products during baking

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## Summary

A crust is formed on the surface of meat products during baking. Crust formation is accompanied by production of colour and of mutagenic substances. This study was undertaken in order to investigate how various recipes and how heat and mass transfer effect the formation of such mutagenic substances and colours. Mutagenicity did not increase with higher fat content. The correlation between mutagenicity and colour development was high, suggesting that the Maillard reaction plays an important part in the formation of mutagenic substances. With a recipe containing more fat and less water a higher surface temperature is reached more quickly. The fat might have an increasing effect on the heat transfer and less energy is needed to evaporate the water in the crust.

## Introduction

When meat products are roasted, fried or baked, a crust is formed on the surface as a result of high temperatures and drying out of the outer parts. It is a well known fact that the crust develops the brown colour and gives the meat flavour (Hertz & Chang, 1970) as the results of Maillard reactions. During recent years mutagenic substances have been reported as occurring in the meat crust and there is evidence that the Maillard reaction participates in their formation (Barnes *et al.*, 1983a; Jägerstad *et al.*, 1983). The crust formation, including the Maillard reactions and the production of mutagenic activity, is known to be influenced by heat and mass transport in the meat product during cooking. Thus it is of importance to find the connection between technical and physical conditions and the chemical reactions during cooking

The technical aspects, especially the dependency of air conditions in a convection oven, on crust formation and heat and mass transfer, have been studied for many years at our laboratory (Skjöldebrand, 1979; Holtz *et al.*, 1984). Dagerskog & Bengtsson (1974) examined crust formation during pan frying and oven baking. They stated that the fat content of the recipe did not influence the colour as measured by reflectance.

Conflicting opinions exist concerning the role of fat content on the formation of mutagenic activity during the cooking of meat products. Spingarn *et al.* (1981) demonstrated that mutagenicity of minced meat products reached a maximum of 10% added fat. They thought fat was a required material in the formation of the principal mutagens. On the other hand, studies by Bjeldanes *et al.* (1983) indicated that fat does not take part in the chemical reactions. Instead, along with Barnes, Maher & Weisburger

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(1983b), they suggested that the role of fat could be due to a more efficient heat transfer.

This study was undertaken in order to investigate the effects of various recipes on chemical reactions in the crust and on the heat and mass transport in the product during baking. Minced meat loaves containing various amounts of added fat were baked in a convection oven. The heat transfer, the temperature profile, the amount of energy supplied and the crust thickness were all studied as well as the brown colour and mutagenic activity of the crust.

*Theory of heat and mass transfer in meat products*

In this investigation heat was transferred to the meat loaf via heated air at a defined air velocity—i.e. by means of convection.

Heat is used for raising the temperature of the surface and the product and for evaporation of water. The rate of the heat transfer to the centre of the product depends on the thermal properties of the meat product. These properties may be altered by the recipe—i.e. by the fat and water content.

When the surface has reached the evaporation temperature water will vaporize and the evaporation zone will gradually move towards the centre with time. The outer parts will become dry and the temperature of the crust will rise towards that of the air buik. More details of the mechanisms for the heat and mass transport have been published previously (Hallström & Skjöldebrand, 1983; Holtz *et al.*, 1984; Skjöldebrand, 1979; Skjöldebrand & Olsson, 1980).

To be able to relate the Maillard reaction and the mutagenic substances to the heat and mass transfer the kinetics of these reactions should be known. They are probably of a high order—i.e. depending particularly on water activity and temperature. By combining, for example, time/temperature for an element in the meat product during a certain interval of time with the reaction rate equation, the degree of change may be calculated. When describing a time/temperature influence on a chemical reaction it is often convenient to translate it into terms of equivalent time at a reference temperature of 100°C. This is called the cook value. In a separate investigation the kinetics of the brown colour and the mutagenic substances have been studied in order to relate more precisely the heat and mass transfer to the Maillard reaction and mutagenicity (Holtz *et al.*, unpublished).

In the present investigation different experiments have been compared by calculating the total amount of energy transferred to the crust during each procedure. A concept called 'time–temperature dose' (TTD) has also been used. The TTD is directly proportional to the time and temperature differences of the crust. This may not be directly correlated to any chemical reaction but is used here to compare the different procedures and recipes with each other.

## Materials and methods

*Recipe of meat loaves*

A mixture of minced beef (*M. biceps femoris*), backfat, water and a water binding agent of locust bean gum (Frimulsion 10, Hercules, Sweden) was used. The beef and backfat were ground passing a screen of 4.5 mm. The meat loaves were prepared with various amounts of fat up to 16.6%. The ratio between protein and water content was held constant at 0.25 and the water binding agent was added at a level of 0.25%. The composition of the different recipes is shown in Table 1. The minced meat mixtures were formed into loaves of 200 × 50 × 50 mm, each weighing 600 g.

**Table 1.** Experimental conditions for baking of meat loaves

| | Experimental No. | Recipe | Content of fat (%) | Content of water (%) | Final surface temperature (°C) | Baking time (min) |
|---|---|---|---|---|---|---|
| I | 1 | A | 2.8 | 74.8 | 140 | 56 |
| | 2 | B | 8.1 | 72.3 | 140 | 50 |
| | 3 | C | 13.9 | 67.8 | 140 | 47 |
| | 4 | D | 16.6 | 63.8 | 140 | 39 |
| II | 5 | C | 13.9 | 67.8 | 129 | 39 |
| | 3 | C | 13.9 | 67.8 | 140 | 47 |
| | 6 | C | 13.9 | 67.8 | 143 | 50 |
| | 7 | C | 13.9 | 67.8 | 150 | 59 |
| | 8 | C | 13.9 | 67.8 | 170 | 97 |

*Experimental design*

Meat loaves of four different recipes defined as A, B, C and D (Table 1) were all baked until a final surface temperature of 140°C was reached and designated as experiment I: Nos 1–4 (Table 1).

Thereafter meat loaves with a fixed recipe (C) were baked for different lengths of time, to a final surface temperature between 129 and 170°C, designated as experiment II: Nos 3, 5–8 (Table 1).

*Baking procedure*

The baking was performed in a convection oven developed for well controlled baking experiments as previously described (Skjöldebrand, 1979). The temperature and the velocity of the air were kept constant at 200°C and 9 m/sec respectively and the air was not humidified.

During the baking process the loaves were placed in a teflon coated net cage in order to maintain shape (Skjöldebrand, 1979). Thermocouples were inserted in the centre of the loaves and at various distances from the surface. The temperature of the surface was measured using a pyrometer. The product was weighed continuously during processing. Fifty grams of the crust were peeled away from each meat loaf using a scalpel and kept at −20°C until analysis.

*Calculation of supplied amount of energy*

The energy supplied to the crust is used for heating the product, evaporating the water in the evaporation zone and superheating the vapour in the crust. The amount of energy supplied was calculated according to the equation:

$$\frac{dQ}{d\tau} = A \cdot \alpha \, (t_a - t_s) = C_{po} \cdot \rho_o \cdot \nu \cdot \frac{dm_1}{d\tau} - r \cdot \frac{dG}{d\tau} - (t_s - 100) \cdot C_{pv} \cdot \frac{dG}{d\tau},$$

where

$A$ = heat transfer area ($m^2$)
$C_{po}$ = specific heat of product (J/kg°C)
$C_{pv}$ = specific heat of vapour (J/kg°C)
$G$ = weight of product (kg)

$Q$ = energy (J)
$r$ = heat of evaporation (J/kg)
$t_a$ = temperature of the surrounding air (°C)
$tm$ = mean temperature of the product (°C)
$t_s$ = temperature of the surface (°C)
$V$ = volume ($m^3$)
$\alpha$ = heat transfer coefficient ($W/m^{2\circ}C$)
$\rho_o$ = product density ($kg/m^3$)
$\tau$ = time (sec)
(Skjöldebrand, 1979).

From this equation the heat transfer coefficient may be calculated. The thermal properties were taken from the literature (Sörenfors & Dagerskog, 1978).

*Calculation of time/temperature/dose*

Temperatures were measured on the surface and in the crust at different distances from the surface. The total time/temperature/dose (TTD) for the crust was calculated in the following way: the estimated temperatures were used, and the temperatures through the whole crust were calculated at intervals of 0.1 mm *versus* time. At each step the temperature difference for temperatures above 100°C was multiplied by the actual time step. The TTD is defined as the sum of every time/temperature contribution from each step of distance and time in the total crust and integrated over the total baking time.

*Analyses*

*Chemical composition.* Fat by the SBR method, protein according to Kjeldahl and water content, with drying at 104°C overnight, were all analysed according to standard methods (Nordic Committee on Food Analysis, 1955, 1976 and 1974, respectively).

*Colour of the crust.* Duplicates of samples were extracted in 0.6 N perchloric acid and the proteins were precipitated. The supernatant was neutralized and the colour was measured spectrophotometrically at 375 nm, expressed as absorbance per g dry matter of crust.

*Mutagenic activity.* Thirty-five grams of lyophilized powder of crust from each loaf was used. Mutagens were extracted according to Felton *et al.* (1980).

Mutagenicity was analysed according to Ames, McCann & Yamasaki (1975) using TA98 as the test organism with and without activation by rat liver homogenate (S9). The analyses were performed using S9 mix containing 5% liver homogenate as described by Jägerstad *et al.* (1983).

The samples dissolved in 2 ml of DMSO were analysed in duplicates at two different amounts, 10 and 20 ml per plate. Each meat loaf was analysed on two different occasions and appropriate controls were included in the assay.

*Statistics*

The analyses of the variation in mutagenicity and its dependence on other variables were done using multiple regression analyses and, in part, multiway analyses of variance. Standard tests in these situations were performed at a significance level of 5% (Draper & Smith, 1981).

## Results and discussion

*Effects of varying fat content*

In the first experimental series, the meat loaves contained varying amounts of fat up to 16.6% (Table 1). As a result, the water content was decreased in order to keep a constant ratio between protein and water. In order to obtain the same heat treatment for the meat loaves, the baking procedure was stopped when the surface temperature reached 140°C. The amount of energy supplied showed similar values for all four recipes (Fig. 1), confirming that all the products had received the same heat treatment independent of recipe. By increasing the amount of additional fat the baking was decreased from 56 to 39 min (Table 1).

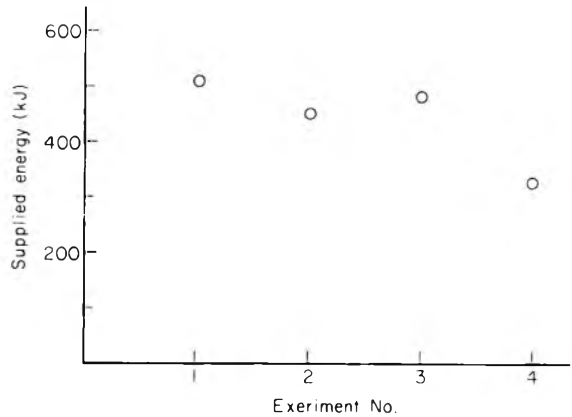


**Figure 1.** The amount of supplied energy for experiment Nos 1–4.

A higher surface temperature was reached more quickly with the meat loaf containing more fat and less water. This can be explained by the fact that there was less water in the crust to evaporate and therefore a larger amount of the supplied energy was used for raising the temperature of the surface and the crust. In this type of product with a high weight loss, the main part of the energy is used to evaporate the water and to superheat the vapour. As a result the weight losses were highly correlated with the amount of energy supplied, in all eight experiments (Fig. 2).

The system used, consisting of various amounts of fat, water and protein is very complex and it is difficult therefore to distinguish the factors that are of greatest importance for the formation of mutagenic substances.

The fat might have an increasing effect on the heat transfer between air and the product. When the product is heated the fat will melt and form a thin layer of melted fat flowing around the product causing a more efficient heat transfer.

The production of mutagenic activity in the crust of the meat loaves of different composition, is shown in Fig. 3a,b. The mutagenicity is expressed in revertants per g dry matter of the crust, (a) uncorrected and (b) corrected for the fat content of the crust. The amount of mutagenic activity was found to decrease with increasing fat content (Fig. 3a), but after correction for fat content only meat loaf No. 4, was significantly lower than all the other loaves (Fig. 3b). The reason for this is not known, but as seen in Fig. 4 meat loaf No. 4 also showed the lowest colour value. Although the heat supplied

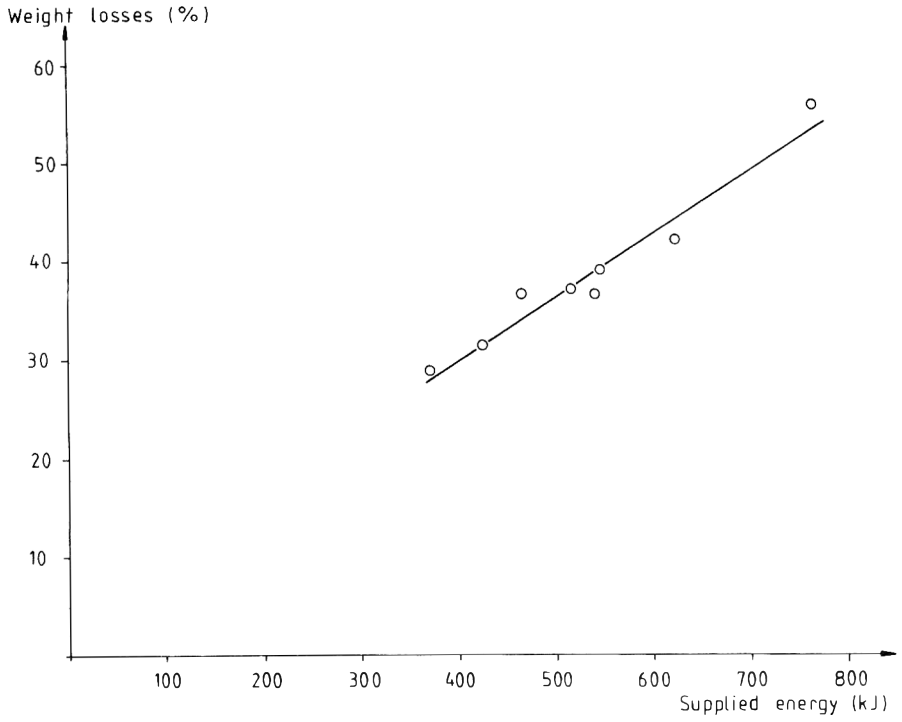


**Figure 2.** Weight losses *versus* the amount of supplied energy for all experiments.

to the surface of the four different recipes was almost identical, the development of colour decreased with increasing fat content and seemed to vary in parallel with the mutagenicity produced (Fig. 4).

The fact that mutagenicity in the crust did not increase with increasing fat content does not speak in favour of fat as a chemical reactant in the formation of mutagenicity. Instead the high correlation between mutagenicity and colour development suggested that the Maillard reaction plays an important role in the formation of mutagenic substances.

*Effects of varying baking times using a fixed recipe*

In order to further clarify the role of heat transfer on crust formation and mutagenicity meat loaves having the same fat and water content were baked four times, ranging from 39 to 97 min. With increasing baking time the final surface temperature rose from 129 to 170°C. In parallel the mutagenicity increased up to a surface temperature of 150°C but at 150°C the mutagenicity did not increase further (Fig. 3). Such levelling effects on mutagenicity as the baking times increased have also been reported by Bjeldanes *et al.* (1983).

*Statistical results*

In order to single out which variables were responsible for variations in mutagenicity, data produced in both experimental series were subjected to statistical evaluation.

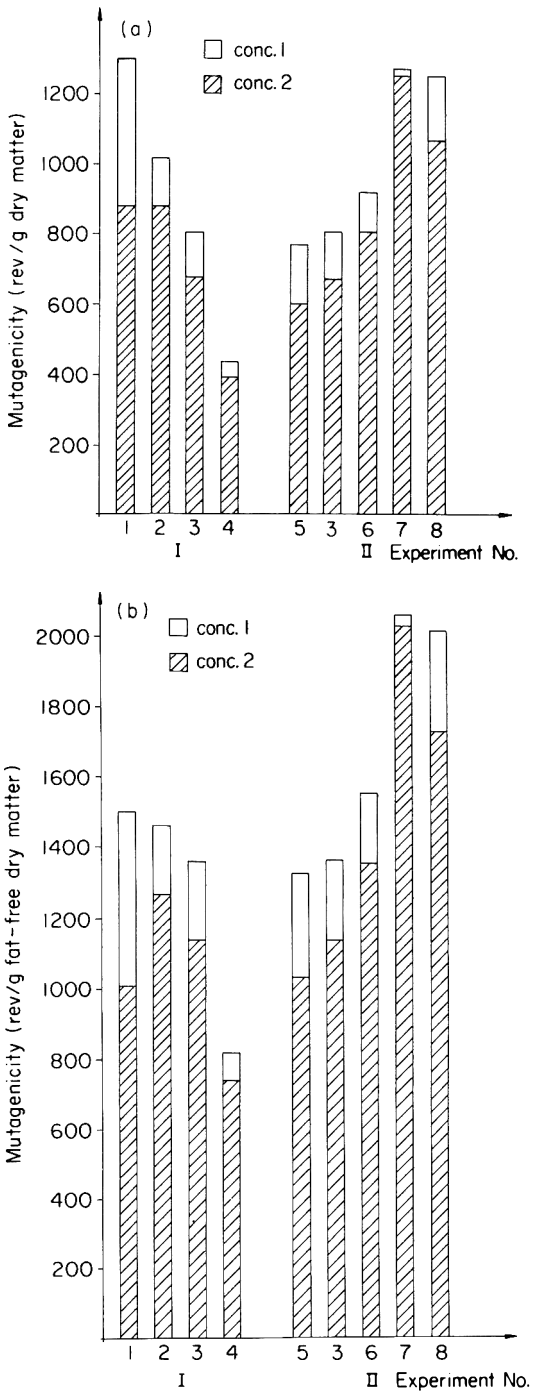


**Figure 3.** Mutagenic activity in revertants per g crust, calculation on dry material, (a) uncorrected and (b) corrected for fat content. Unfilled bars represent 10 $\mu$l of sample extract/plate and filled bars, 20 $\mu$l. (For details of the design of experiments I and II, see Table 1.)

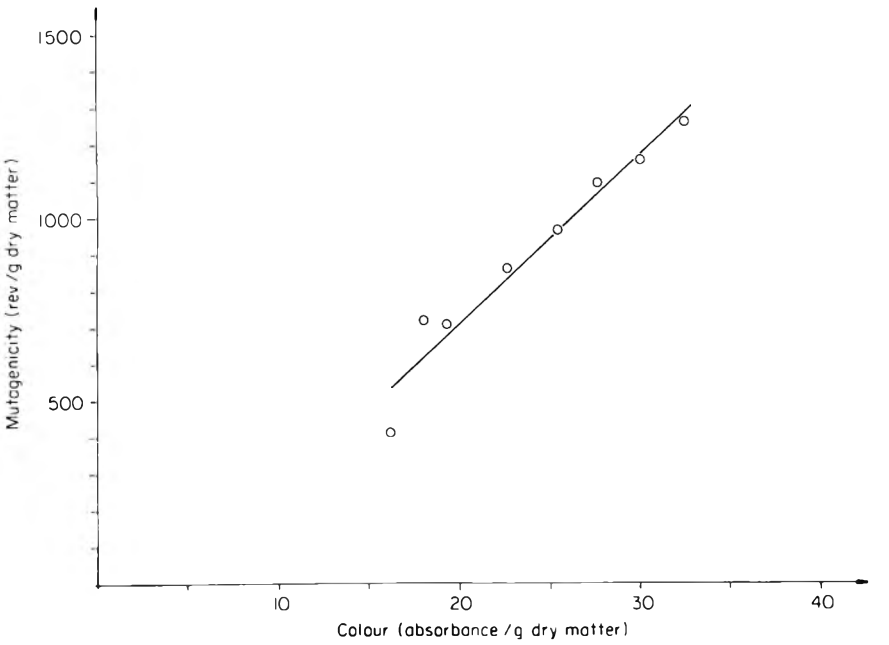


**Figure 4.** Mutagenicity *versus* colour. Colour expressed as absorbance at 375 nm/g dry matter of crust.

First, statistical analyses of mutagenicity were made. The two quantities used in the assay resulted in means which differed significantly, suggesting methodological error (Fig. 3). The most likely explanation for these differences, are that the amount of S9 was optimal for only one of the amounts assayed. Another possibility is that the crust extracts assayed for mutagenicity contained traces of oleic and/or linoeic acid, which are known inhibitors of the Ames test (Hayatsu *et al.*, 1981). This, however, seemed unlikely because the difference between the two doses did not follow the fat content of the meat loaves (Fig. 3a,b).

Further statistical evaluations of the two experimental series showed variations in colour development of the crust to be mainly responsible for the variation in mutagenicity. Fifty-seven percent of the variation could be explained by variation in colour. Looking at each experimental series separately, the baking time was mainly responsible for variation in mutagenicity with varying recipes. In the second series, using meat loaves of a fixed recipe, the development of a brown colour explained most of the mutagenic variation.

Since colour development explained the main part of the variation in mutagenicity it is interesting to identify the variables that affected colour. Baking time was found to explain 50% of the variation in crust colour in the statistical evaluation. Baking time was found to explain 50% of the variation in the crust colour. Together with the TTD/g dry matter of the crust 80% were explained in the statistical evaluation.

## Conclusions

The present study shows that the total amount of heat supplied to the meat loaves was greatly influenced by recipe as well as baking time. With an increasing ratio of fat to

water the baking time decreased for the same amount of heat transferred to the surface. According to a study by Spingarn *et al.* (1981) fat was reported to participate as a reactant in the formation of mutagens. This does not agree with our results, but in their study the possibility of an increased heat transfer with an increasing fat/water ratio was not monitored or discussed. Our conclusions regarding the role of fat for mutagenicity are similar to those recently reported by Bjeldanes *et al.* (1983). They also stressed the importance of the ratio between fat and water for the amount of heat transferred into the product.

The present study also showed that the colour development, as an expression for Maillard reactions, was the main cause for variation in mutagenic activity of baked meat loaves. This is in agreement with two previous reports (Jägerstad *et al.*, 1983, 1984).

In order to clarify the role of fat *versus* water for mass and heat transfer it is necessary for more work to be done. A meat mixture is very complex and the different components act in different ways. One way to proceed might be to use mathematical models in order to simulate the temperature and water content or activity profile in the product with various recipes and with different heating conditions. Such work is in progress (Holtz *et al.*, unpublished). The present study indicated that colour development might be a useful marker of the formation of mutagenicity. Work is also in progress to further define the link between heating time, temperature, colour development and mutagenicity using kinetic models, the predicting value of which will be compared with results of realistic cooking experiments.

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# Thin layer air drying of French fried potatoes

WIN-CHIN CHIANG AND JAMES N. PETERSEN*

## Summary

A drying system was designed and constructed to investigate the thin layer air drying of French fried Russet Burbank potatoes. This system permitted the weight loss of the sample to be monitored accurately and continuously without removal of the sample from the drying chamber. Constant drying conditions over a practical range were maintained by continuous control using an on-line computer. The drying conditions investigated in this study included dry bulb temperature ranging from 43 to 93°C, relative humidity ranging from 10 to 60%, and air velocity ranging from 0.79 to 1.77 m/sec. A logarithmic model was used to describe the drying curve. The factors affecting drying rate are discussed. The dry bulb temperature had a dominant effect at low relative humidity, whereas at high relative humidity other factors also affected the drying rate. Relative humidity affected the drying rate significantly, while air velocity had the least effect on the drying rate. The drying rate constant in the logarithmic model was correlated with relative humidity, dry bulb temperature and air flow rate and a good fit was obtained.

## Introduction

In the French fry process raw potatoes are washed, peeled, sorted and cut into the desired shape. They are blanched in water at 65–82°C, and then dehydrated to a moisture content of approximately 60% on a wet basis with air to achieve a crisp texture in the finished French fry. The potato strips are fried in hot oil at 170–193°C, cooled in ambient air and frozen in electrically operated freezers. Finally, frozen French fries are packaged (Blackstock & Skiver, 1974). Therefore, the drying step plays a very important role in controlling the quality of the product.

The mechanisms governing thin layer drying have been studied extensively. Lewis (1921) divided thin layer drying into constant and falling rate periods. During the constant rate period of drying, the surface temperature of the material will be at or close to the wet bulb temperature of drying air, and a quasi-thermodynamic equilibrium prevails at the surface. The rate of drying for this period is therefore controlled by external conditions (Berger & Pei, 1973). Once the drying process has entered the falling rate period, the external parameters such as internal vapour diffusion and internal heat conduction. The drying behaviour during the falling rate period is then controlled by the sorptional isotherm of the drying system (Berger & Pei, 1973).

Lewis (1921) suggested that the rate of drying during the falling rate period is directly proportional to the difference between the actual moisture content of the material and the equilibrium moisture content which is determined by the sorptional isotherm of the material, i.e.:

$$\frac{dM}{dt} = -k\,(M - Me), \tag{1}$$

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where $M$ is moisture content in the material, $Me$ equilibrium moisture content, $t$ drying time and k drying constant. After separating variables, eqn (1) can be integrated to:

$$MR = \frac{M-Me}{Mo-Me} = \exp(-kt), \quad (1)$$

where $Mo$ is initial moisture content. The eqn (2) is called the exponential or logarithmic model (Sabbah, Keener & Meyer, 1977).

Becker & Sallans (1956) and Ross & White (1972) found that during grain drying k is a function of the absolute temperature of the drying grain. Henderson & Pabis (1961) have shown the theoretical justification for using an Arrhenius type equation to relate the thin layer drying constant to temperature. Their resulting equation is:

$$k = p \exp(-q/T), \quad (3)$$

where p and q are constants determined by the material. Chittenden & Hustrulid (1966) and Van Rast & Isaacs (1968) found that the value of k for shelled corn and other grains is dependent on the moisture content as well as the drying temperature. They found that the effect of moisture content, however, seemed to be less than that of drying temperature.

## Materials and methods

This section is divided into three parts which describe the apparatus, procedure and data analyses.

*Apparatus*

The dryer design is shown schematically in Fig. 1. Air is recirculated through the dryer by a centrifugal fan located at the bottom of dryer. Air moves through a recirculation valve where the humid air can be discharged and the fresh air introduced to dilute the humid air. The air passes through two 1600 W heating elements to be heated up to the desired temperature. Power to the heating elements was controlled by a WICAT (150/6 WS) computer in order to maintain the dry bulb temperature within the dryer. Flow straighteners were placed on the elbow above the material being dried to develop a uniform air flow. The sample track rack (0.3×0.3 m) was hung on a Super-mini 50 lb capacity load cell (model SM-50-40, Interface Co., Inc., Scottsdale, AZ, U.S.A.). A strain gauge amplifier, Action Pak AP 4501 (Action Instruments Co., Inc., San Diego, CA, U.S.A.) was used to sense the stress in the load cell. The location of the wire-mesh sample tray was approximately in the middle of the drying chamber. Two thermocouples were placed above the sample to measure the dry bulb and wet bulb temperatures. The one to measure the wet bulb temperature was wrapped with wet cotton wick dipped in a bottle of distilled water. High air velocity is required for the determination of humidity using wet bulb temperature to minimize the resistance of mass transfer existing at the interface of a gas film and a liquid film. Keey (1972) indicated that as long as turbulent flow prevails past the wetted bulb, the resistance of mass transfer could be neglected. The air flow used in this study was in the turbulent flow region; the lowest Reynolds' number among the drying conditions was 11000. Steam as introduced into a surge tank and then injected into the dryer to maintain the humidity of the drying system at the desired condition. The introduction of steam depended on whether a solenoid valve was open or closed, which was controlled by the message signal from the computer.

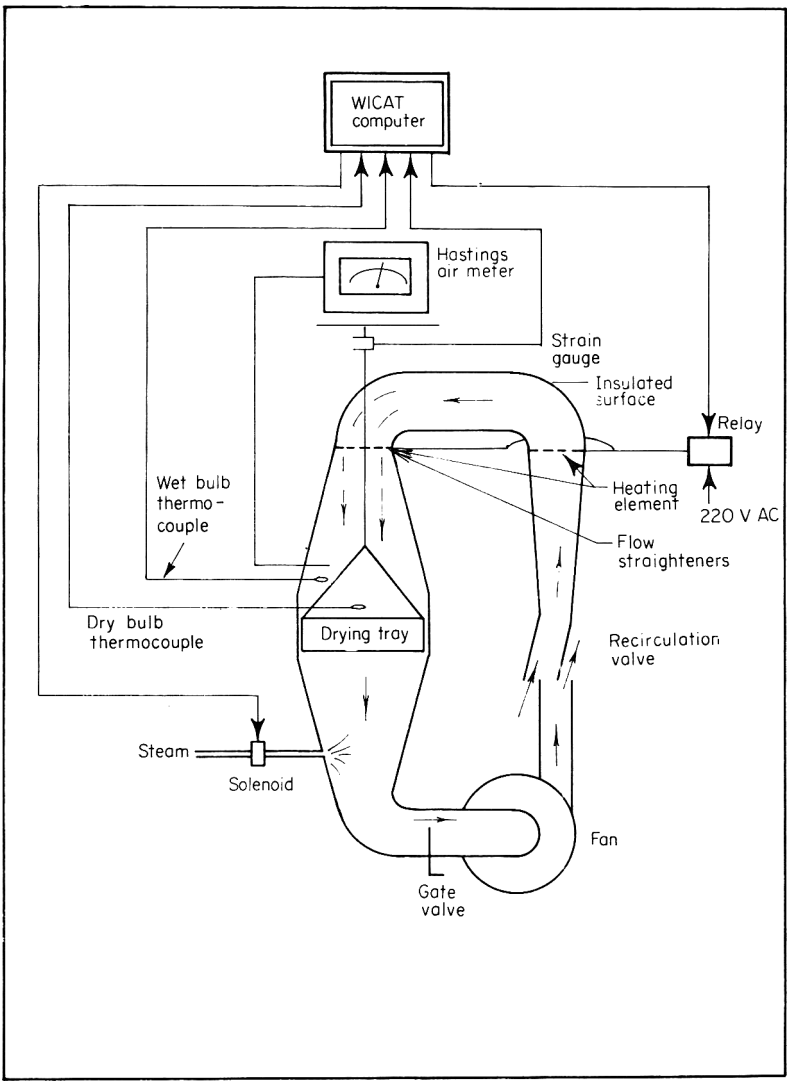


**Figure 1.** Schematic diagram of thin layer dryer.

A manually operated gate valve ahead of the fan was used to adjust the air flow rate in the dryer. The average air flow rate was measured using a Hastings air meter (Model B-22, Hastings-Raydist Co., Hampton, VA, U.S.A.) at nine positions, two inches above the sample tray. The velocity distribution of air varied within 20% of the average value. The Hastings air meter was calibrated in the centrifugal fan wind tunnel with $0.9 \times 0.9$ m test area and the test position was 8 m from the fan so that the turbulence of the wind stream was less than 1%.

The drying condition of the air blowing through the sample tray was maintained at constant dry bulb and wet bulb temperatures using an on-line digital computer. The weight of material being dried was also recorded by the computer every second throughout the experiment. On–off control was used to maintain the wet and dry bulb temperatures by switching the heating elements and the solenoid valve in the steam

line. The drying conditions that were investigated included dry bulb temperatures at 43, 60, 77 and 93°C, and air velocities at 0.79, 1.06 and 1.77 m/sec, while relative humidities were adjusted to be 10 and 19% for 93°C, 10, 30 and 36% for 77°C, and 10, 30 and 60% for 60 and 43°C. The drying conditions were maintained within 1°C for dry bulb temperature, 3% of relative humidity and 20% of air velocity.

*Procedure*

Russet Burbank potatoes (*Solanum tuberosum*) were purchased from a local supermarket and stored in a cold storage room at 4°C for about 1 month until used. Stored potatoes are normally more uniform in physical properties than freshly harvested potatoes (Matthews & Hall, 1961). Two ends of the potatoes were cut off and the middle region was cut into 7.9×7.9×51.0 mm strips using a French fry cutter (EKCO Housewares Co., Franklin Park, IL, U.S.A.). The potato strips were then blanched in a water bath at 80°C for 3 min to inactivate the enzymes, mainly polyphenol oxidase (E.C. 1.10.3.1) and peroxidase (E.C. 1.11.1.7) (Collins & McCarty, 1969) because polyphenol oxidase produces a darkening of tissue and peroxidase produces a brown coloration if allowed to react with the substrates occurring in the potato tissues after cutting. The blanching temperature was set to match conditions in a production processing plant, 80°C. The blanching time was determined by extrapolating the curve of the residual enzyme activity at 80°C to be 0.2% which was presented by Dimick, Ponting & Makower (1951). After draining for a short period of time, the potato strips were placed on the tray and arranged in one layer about 5 mm apart from one another. The sample was then put into the dryer after the drying conditions in the dryer reached steady state. The potato strips being dried were not removed from the dryer until constant weight was reached. Moisture content of potato strips at the beginning and the end of drying was determined by drying the sample for 8–10 hr at 105–110°C (AOAC, 1980). Moisture determination was performed in duplicate.

*Data analyses*

No a priori assumptions of the drying mechanism were used in the data analysis. The technique used to analyse the data allowed both the constant rate period and the falling rate period to be taken into consideration. The following development describes how this was done. The drying rate can be expressed as:

$$R = -\frac{Ms}{Ax}\frac{dX}{dt}, \tag{4}$$

where R: drying rate, $Ms$: weight of dry solid, $X$: bulk moisture content of the solid (g of liquid/g of solid), and $Ax$: surface area of material.

For the constant rate period of drying, R is a constant. Let $Mt$ be total mass of the material any time, then

$$X = (Mt - Ms)/Ms. \tag{5}$$

Substituting this expression into eqn (4), produces

$$R = -\frac{1}{Ax}\frac{dMt}{dt} = c. \tag{6}$$

Therefore, integrating this equation yields:

$$Mt = a_1 t + a_2, \tag{7}$$

where $a_1 = -c\,Ax$ and $a_2$ = initial weight of the sample.

During falling rate period of drying, the drying rate is assumed to be linearly proportional to the moisture content of the sample (McCabe & Smith, 1976), so

$$R = b_1X+b_2$$

$$= \frac{1}{Ax}\frac{dMt}{dt} \tag{8}$$

After manipulating the above equation, one obtains

$$\frac{dMt}{dt} = a_3Mt+a_4, \tag{9}$$

where

$a_3 = -b_1\,Ax/Ws$
$a_4 = Ax(b_1-b_2)$.

The above differential equation was solved and became

$$Mt = a_6+a_5\exp(a_3t), \tag{10}$$

in which $a_6 = -a_4/a_3$.
This reduced to:

$$MR = \frac{X-Xe}{Xo-Xe} = \exp(a_3t), \tag{11}$$

where $Xo$ is the moisture content of the solid at the start of the falling rate period or at the beginning of the experiment if there is no constant rate period during drying and $Xe = (a_6-Ws)/Ws$ equals the equilibrium moisture content, while $a_6$ is the predicted mass of the sample when time is equal to infinity. Therefore, the thin layer drying rate constant, k, in eqn (2) equals $-a_3$. A non-linear SAS (statistical analysis system) program (SAS, 1982) was used to calculate the coefficients of the thin layer equation and also plot the drying curve of each experiment. Another SAS procedure (SAS, 1982) was used to perform the non-linear regression of the thin layer drying constant with relative humidity, dry bulb temperature and air velocity.

## Results and discussion

It was visually observed that the free water on the surface of blanched potato strips disappeared within the first 10 sec after drying started. The data analyses indicated that the entire drying process took place during the falling rate period with no constant rate period existing. This is in contrast with the results of Saravacos & Charm (1962) and Husain *et al.* (1972), but in agreement with Ede (1958), Gorling (1958) and Igbeka *et al.* (1976). Thirty-three thin layer drying curves, which were plotted as the measured weight of French fried potatoes *versus* drying time, under different drying conditions showed that the logarithmic model fitted the drying curves very well. In the drying curves, exemplified by Figs 2 and 3, the weight of the sample decreased rapidly during the first drying period and then levelled off thereafter to reach a constant value. It took about 2 hr to reach a constant weight for high dry bulb temperature and low relative humidities. Longer time was needed to reach a constant weight when the dry bulb temperature decreased or relative humidity increased.

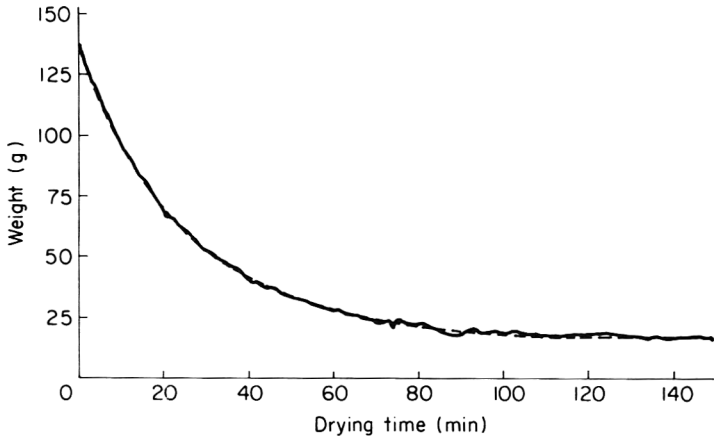


**Figure 2.** The drying curve at drying conditions of 93°C, 10% relative humidity and an air velocity of 1.06 m/sec.

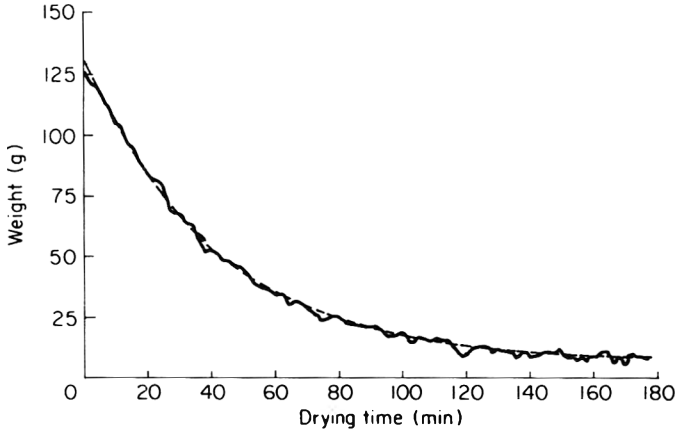


**Figure 3.** The drying curve at drying conditions of 77°C, 36% relative humidity and an air velocity of 1.77 m/sec.

The values of the drying rate constant, k, were determined by fitting data developed in the experiments to logarithmic model eqn (2). The k values for different dry bulb temperatures with different air velocities—1.77, 1.06 and 0.79 m/sec—are shown in Tables 1–3, respectively. Theoretically, the faster the drying rate is, the greater the drying constant, k, will be. From Tables 1–3, it is apparent that thin layer drying rates increased with increasing dry bulb temperature and with decreasing relative humidity, as was expected. It also shows that the velocity of drying air has little effect on the drying rate constant. Henderson & Pabis (1962) studied the thin layer drying of wheat at 43°C, relative humidity of 21% with air velocities of 0.015, 0.1, 0.45 and 0.7 m/sec. They also found that air flow rate had little effect upon the drying rate. Ede (1958), Saravacos & Charm (1962) and Hutchinson & Otten (1983) indicated that air velocity did not affect the drying rate during the falling rate period.

The values of the drying constants as shown in Tables 1–3 are relatively close at the same dry bulb temperature and relative humidity for the three different air velocities. Compared to the same drying temperature, drying rates were generally found to be higher at lower relative humidities. The driving force for vaporizing

**Table 1.** The drying rate constant at air velocity of 1.77 m/sec with different drying conditions

| Dry bulb temperature (°C) | Relative humidity (%) | Absolute humidity (g water/g dry air) | Drying rate constant (1/min) |
|---|---|---|---|
| 93 | 10 | 0.052 | 0.0400 |
| 93 | 19 | 0.110 | 0.03633 |
| 77 | 10 | 0.026 | 0.03281 |
| 77 | 30 | 0.084 | 0.02438 |
| 77 | 36 | 0.110 | 0.02428 |
| 60 | 10 | 0.0125 | 0.02890 |
| 60 | 30 | 0.039 | 0.01703 |
| 60 | 60 | 0.081 | 0.00887 |
| 43 | 10 | 0.0052 | 0.01936 |
| 43 | 30 | 0.017 | 0.01655 |
| 43 | 60 | 0.033 | 0.00441 |

**Table 2.** The drying rate constant at air velocity of 1.06 m/sec with different drying conditions

| Dry bulb temperature (°C) | Relative humidity (%) | Absolute humidity (g water/g dry air) | Drying rate constant (1/min) |
|---|---|---|---|
| 93 | 10 | 0.052 | 0.03999 |
| 93 | 20 | 0.110 | 0.04111 |
| 77 | 10 | 0.026 | 0.04001 |
| 77 | 30 | 0.084 | 0.02288 |
| 77 | 36 | 0.110 | 0.01888 |
| 60 | 10 | 0.0125 | 0.02510 |
| 60 | 30 | 0.039 | 0.02131 |
| 60 | 60 | 0.081 | 0.01226 |
| 43 | 10 | 0.0052 | 0.01635 |
| 43 | 30 | 0.017 | 0.01357 |
| 43 | 60 | 0.033 | 0.00192 |

**Table 3.** The drying rate constant at air velocity of 0.75 m/sec with different drying conditions

| Dry bulb temperature (°C) | Relative humidity (%) | Absolute humidity (g water/g dry air) | Drying rate constant (1/min) |
|---|---|---|---|
| 93 | 10 | 0.052 | 0.03919 |
| 93 | 19 | 0.110 | 0.03369 |
| 77 | 10 | 0.026 | 0.03424 |
| 77 | 30 | 0.084 | 0.01966 |
| 77 | 36 | 0.110 | 0.02117 |
| 60 | 10 | 0.0125 | 0.01835 |
| 60 | 30 | 0.039 | 0.01891 |
| 60 | 60 | 0.081 | 0.00434 |
| 43 | 10 | 0.0052 | 0.01777 |
| 43 | 30 | 0.017 | 0.01471 |
| 43 | 60 | 0.033 | 0.00464 |

water from the surface of material is the difference between the vapour pressure of water at the temperature of the surface and the partial pressure of the water in the air. The driving force becomes larger when relative humidity decreases. It is clear that at each temperature a substantial decrease in drying rate accompanies the humidification of the drying air, especially for low temperature and high relative humidity at an air velocity of 1.77 m/sec and the drying temperatures of 43, 60 and 77°C is shown in Fig. 4. It shows that the drying rate decreases as relative humidity increases.

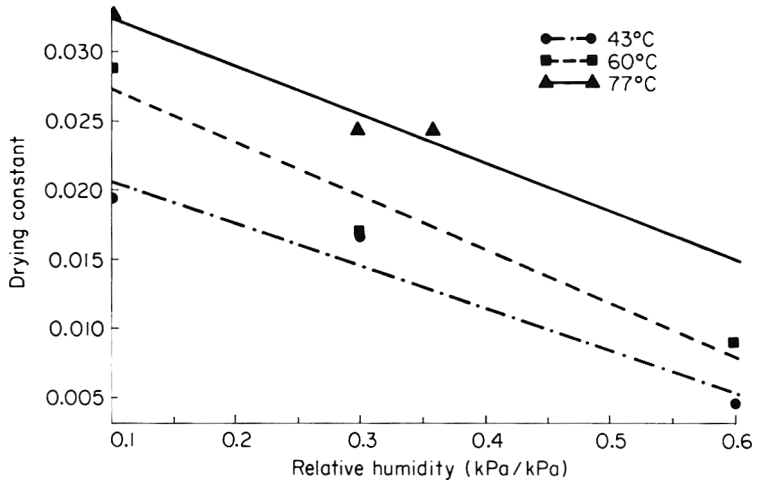


**Figure 4.** The effect of relative humidity on the drying constant at an air velocity of 1.77 m/sec and different drying temperatures.

From Tables 1–3 it can be seen that dry bulb temperature has a great effect on the drying constant. Allen (1960) pointed out that the effect of increasing the temperature of the drying air at constant relative humidity was to increase the drying rate considerably. The results in this study are in agreement with Allen. Stone (1982) investigated the thin layer drying of hops and described the drying constant as a linear function of drying temperature. But as shown by Henderson & Pabis (1961), the temperature effect on the drying rate constant follows the Arrhenius type equation. Pabis & Henderson (1961) expressed the drying rate constant for thin layer drying of shelled corn *versus* temperature as an Arrhenius type of equation. The relationship between logarithmic values of drying constant and the reciprocal of temperature at relative humidities of 10, 30 and 60% are shown in Figs 5–7, respectively. The correlation coefficient for drying constant *versus* temperature, expressed as the Arrhenius type relationship is high (0.929) at a relative humidity of 10%, but the correlation coefficient decreases when relative humidity value increases. This indicates that at low relative humidity, temperature is a predominant factor on the drying constant, whereas at higher relative humidity other factors also affect the drying rate constant.

On the basis of the above discussion and analysis, it is concluded that the drying rate constant is not a function of only one factor of drying conditions, but the combination of relative humidity, dry bulb temperature and velocity of drying air. Therefore, a non-linear regression analysis was used to predict the drying rate constant. Since the correlation coefficient of a linear regression between the drying

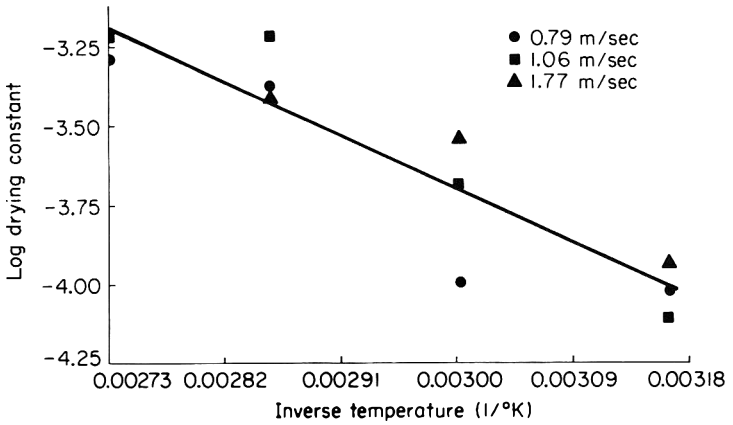


**Figure 5.** Relationship between the drying constant and the reciprocal of drying temperature at 10% relative humidity and different air velocities. The correlation coefficient for the straight line is 0.929.

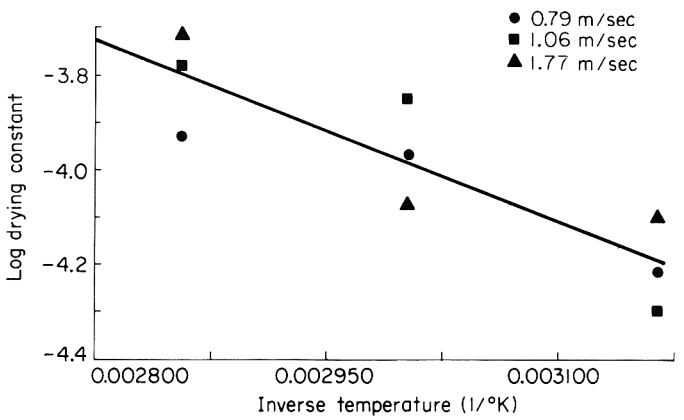


**Figure 6.** Relationship between the drying constant and the reciprocal of drying temperature at 30% relative humidity and different air velocities. The correlation coefficient for the straight line is 0.878.

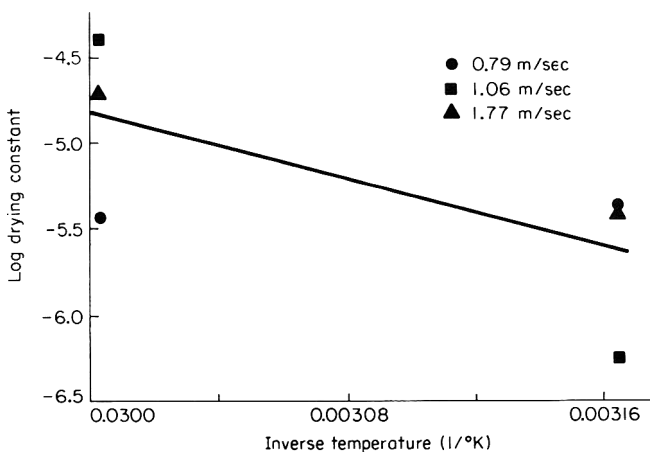


**Figure 7.** Relationship between the drying constant and the reciprocal of drying temperature at 60% relative humidity and different air velocities. The correlation coefficient for the straight line is 0.703.

constant and relative humidity was relatively low (0.771) as shown in Fig. 4, and Agrawal & Singh (1977) and Wang (1978) showed the drying constant related with relative humidity to some power, it was decided that the empirical form shown below would be used.

$$k = c_1 + c_2 (RH)^{c_5} + c_3 \exp(c_4 T) + c_6 V. \quad (12)$$

A SAS program using non-linear regression procedure was used to calculate the parameters in this equation. The final expression relating the drying rate constant to relative humidity, dry bulb temperature and velocity of the drying air is given by:

$$k = 0.01138 - 0.03299 (RH)^{0.859} + 21.9514 \exp(-2407.98/T) + 0.002077 V, \quad (13)$$

where RH is relative humidity in decimal value, $T$ dry bulb temperature (°K), and $V$ air velocity (m/sec). As was expected, dry bulb temperature has the greatest effect on the drying rate. Equation (13) indicated that the drying rate constant was not linearly dependent upon relative humidity. Rather, the drying rate constant is proportional to relative humidity raised to the 0.859 power. In addition, the effect of drying air velocity is very small as indicated by the small coefficient on this term. As discussed previously, the external conditions are relatively unimportant as compared with the internal conditions during the falling rate period. The small effect of air velocity on the drying rate proves this conclusion. The dry bulb temperature and relative humidity are coupled effects on internal heat and moisture transfers and have greater influences on the drying rate. A linear regression with an intercept of zero and confidence level of 95% for experimental and predicted values of the drying constant is shown in Fig. 8. A correlation coefficient of 0.965 and $F$-value of 420 in the $F$-test were computed using the fitted equation.

## Conclusions

A well operating thin layer drying experimental system was constructed. The drying conditions were maintained within 1°C for dry bulb temperature, 3% of relative humidity and 20% of air velocity. The drag force due to the air blowing through the

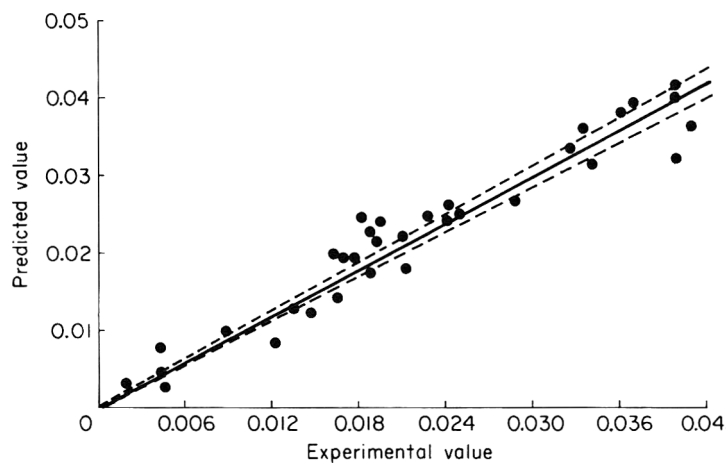


**Figure 8.** Relationship between the predicted drying constant from regression and experimental value. The correlation coefficient for the solid line is 0.965. Two dotted lines show the confidence level of 95%.

sample tray was measured to be constant throughout the experiment. Therefore, the weight loss could be monitored accurately and continuously without removal of the sample from the drying chamber. A manually operated gate valve was used to adjust the flow rate of air so that the drying system could be operated at different air velocities between one experiment and another.

The thin layer air drying of French fried potatoes was found to take place completely in the falling rate period. At low relative humidity, temperature is a dominant factor on the drying rate, whereas other factors also affect the drying rate at high relative humidity. Relative humidity has a significant effect upon the drying rate while air flow rate has little effect. A non-linear equation related the drying constant to relative humidity, dry bulb temperature and air velocity has also been developed.

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# Inactivation of cocoa seeds after treatment by heat, acetic acid and ethanol

R. S. MORETON

## Summary

The death rates of cocoa seeds (beans) were measured after treatment with wet and dry heat, 1% ethanol and 1% acetic acid. Death rate was logarithmic over the range 40°C–60°C. The relevance of this information to the fermentation of cocoa beans is discussed.

## Introduction

The seeds of the cocoa tree, *Theobroma cacao*, undergo a complex post-harvest fermentation producing flavour precursors which, on roasting, develop into the compounds associated with the aroma and flavour of cocoa and chocolate products. The accepted sequence of events during the fermentation has been reviewed by Forsyth & Quesnel (1963). Briefly, after harvesting, the seeds (beans) are removed from their pods and placed either on the ground, sometimes on and covered by leaves, or in specially constructed fermentation boxes. The object of the fermentation is to remove the mucilaginous pulp surrounding each seed, which is difficult to remove mechanically, enabling the seeds to be dried for transport to the manufacturer. Flavour precursor development was presumably originally a fortuitous byproduct of this drying process.

Yeasts and bacteria begin to grow on the pulp, producing ethanol and acetic acid and raising the temperature of the fermenting mass to ≅50°C. It is claimed that the chemical and biochemical changes which lead to production of these flavour precursors are initiated by death of the seeds caused by a combination of heat, ethanol and acetic acid.

There is little published information to show which of these factors is most important and under what conditions seeds are rendered non-viable in the presence of these three factors. Quesnel (1965) described experiments to elucidate these points, but his interpretation of his results is somewhat unclear.

## Materials and methods

*Cocoa beans*

Cocoa beans were amelonado type, grown in the Chundale area of Karnataka State, India. Ripe pods were harvested in May/June and air freighted to Bombay. Experiments were carried out at the Thane (Maharashtra) factory of Hindustan Cocoa Products.

Pods were split with a knife, beans extracted manually and placed in water in a Hobart mixer with wire whisks to remove the pulp. Whisking for 15–20 min with several changes of water completely removed the pulp without damaging the testa.

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*Assessment of bean 'death'*

Cocoa beans were assumed to be non-viable, ('dead') if they failed to germinate when left at ambient temperature (≅30°C) for 7 days wrapped in wet absorbent cotton wool and sealed in foil pouches to prevent dehydration. Germination was scored as emergence of the radicle from the seed. There were wide variations in the length of the radicle at this time, from a few millimetres up to several centimetres with secondary roots, many with signs of tip necrosis. All were scored as positive.

Germination rates for untreated controls, de-pulped as above, were > 95%.

*Chemicals*

Ethanol and acetic acid were obtained locally, and were of reagent grade.

*Bean treatment*

Solutions of 1% (v/v) ethanol and acetic acid and a 1% mixture of each, were pre-heated to the required temperature in a water bath and 150 g (wet weight) of de-pulped beans added to 250 ml of the solution in screw capped plastic containers incubated for intervals of from 0–1000 min. Air heated samples were incubated in a fan circulated incubator.

After the required period, beans were removed from the solution, washed in tap water and sealed in foil pouches to germinate.

## Results

Figure 1 shows times to 100% non-viability, plotted on a logarithmic scale, versus temperature, at 5°C intervals from 40 to 60°C.

It can be seen that 1% ethanol plus 1% acetic acid is the most effective combination, followed by 1% acetic acid, 1% ethanol, water and air heating being the least efficient.

The data points converge on the temperature axis at 70 ± 3°C, except for air heating, which meets the axis at 76°C. Because of this convergence, the ratio between the times taken by the most and least efficient treatments, reduces as the temperature increases. At 45°C heating in air takes 830 min to 100% non-viability, 36 min in 1% ethanol plus 1% acetic acid. At 50°C the corresponding values are 340 and 19 min, and 55°C, 92 and 9.5 min. The ratios between these values being 23.00, 17.89 and 9.68, respectively.

## Discussion

During cocoa fermentations, ethanol concentrations in the pulp reach ≅ 4% (v/v) after ≅ 30 hr and then decline as the acetic acid bacteria oxidize ethanol to acetic acid. Acetic acid gradually increases to ≅ 1.5% (v/v) after 150 hr (Forsyth & Quesnel, 1963). The concentrations of both ethanol and acetic acid used in these experiments represent conditions found in the fermentation after ≅ 120 hr where concentrations of ethanol and acetic acid are both ≅ 1%, and the corresponding temperature would be ≅ 50°C. Under these conditions, the beans should be 100% non-viable in 19.0 min, 36 min at 45°C or 9.5 min at 55°C. Even at 40°C, the times to complete non-viability are much lower than 120 hr, about 50 hr for air heating, 16 hr for water heating, reducing to 1.16 hr for 1% ethanol plus 1% acetic acid.

With minimal heat evolution and production of ethanol and acetic acid, all beans should be killed well before the end of the fermentation, allowing flavour precursor formation to proceed.

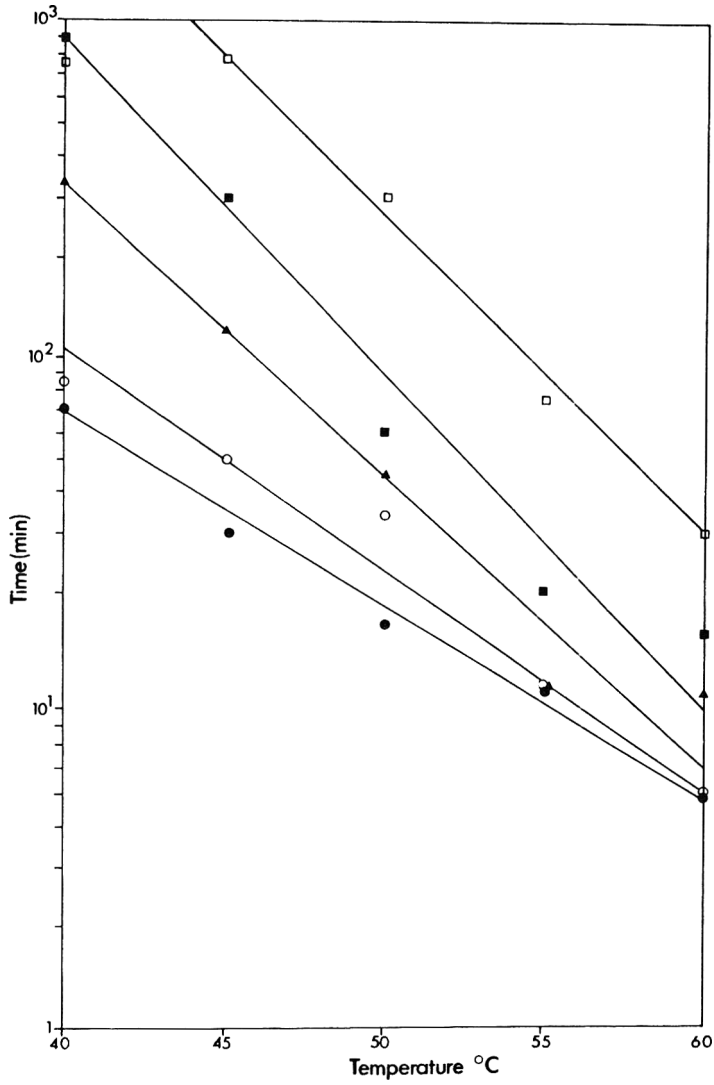


**Figure 1.** Heat and chemical inactivation of cocoa beans. Times to 100% non-viability after treatment with dry heat (□), wet heat (■), 1% ethanol (▲), 1% acetic acid (O), and 1% ethanol + 1% acetic acid (●).

These simple experiments make no allowance for the fact that temperature, ethanol and acetic acid increase gradually during the fermentation. The beans will never be exposed suddenly to concentrations as high as those used here. To some extent, therefore, these experiments are unrealistic, but they do give an idea of the time scale required to inactivate beans in a fermentation. It is clear from the relatively short times found for 100% non-viability under these conditions that there should be no viable beans at the end of a well managed fermentation.

Quesnel (1965) used time to 50% viability as his criterion of bean death. This value was more erratic and unreproducible than the times to 100% non-viability shown in Fig. 1.

The relationships shown in Fig. 1 are the normal logarithmic relationships expected

in a thermally induced killing process, characteristic of a first order chemical reaction. At temperatures between 40°C and ambient (30–32°C), death rates were so slow that the data was erratic. Above 60°C, death rates were so fast that sampling became difficult, and again the data was not reproducible. There seems little reason to suppose, however, that the same logarithmic relationship does not hold down to ambient.

From these data, and re-interpreting those of Quesnel (1965), it is clear that the three agents have a synergistic effect at all temperatures between 40 and 60°C and probably down to ambient as well.

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# Composition and storage stability of commercial anhydrous milk fat and hydrogenated oils

M. H. ISKANDER, S. E. BAYOUMI AND S. I. SHALABI*

## Summary

Two brands of ghee, three brands of butter oil and two brands of hydrogenated oils were examined for compositional properties and keeping quality. Analysis indicated that the samples were within the usual requirements, except one ghee sample which had a very high initial rate of free fatty acids (FFA).

During storage the FFA and peroxide values (PV) increased. The increase in FFA was more obvious at 20°C than at 35°C, but in general the increase was not large. The PV showed a faster increase at 35°C than at 20°C. The increase at 20°C was mostly below 20 mEq peroxide/kg, which had no practical effect on flavour changes. This suggested that oxidative rancidity could be a major problem if the fat is stored at temperatures above 20°C.

Among the fat samples examined, ghee samples (Egyptian products) showed the least storage stability, while butter oil Frico brand (Netherlands product) showed the highest storage stability.

## Introduction

Butter oil (from cow milk) is the anhydrous form of milk fat produced in Europe and the U.S.A. Ghee is the equivalent product produced in Egypt, India and other Middle East and Asian countries. It differs from butter oil in that it is prepared by heating butter at 140°C, whereas butter oil is prepared by melting butter at 80°C (Ganguli & Jain, 1973; Lambert, 1975). However, butter oil is produced in large quantities and is much in demand in developing countries. In Egypt most of the butter oil comes from the EEC countries.

Radema (1974) has shown that transportation of butter oil to and from tropical countries had little effect on the organoleptic and chemical quality. In this investigation it was considered worthwhile to determine the extent to which the chemical quality of butter oil is affected by long storage at high temperatures compared with ghee. Because of the vast increase in the consumption of hydrogenated oils, samples were included for comparison.

## Materials and methods

Butter oil samples were: Cream of Paris (French product); Frico (Netherlands product) and Excellenta (Belgian product). Ghee samples (from cow milk) were: Baladi and Royal-Pack (Egyptian products). Hydrogenated oils samples were: Crisco (from soybean and palm oils—American product) and Nefertiti (from cotton seed oil—Egyptian product).

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The samples in 5 lb capacity tins were brought from the market. The label information indicated that all samples were of same month and year of production. The tins were immediately opened for analysis and then divided into batches consisting of three tins of each variety and stored opened at 20 or 35°C. Samples were taken after 1, 2, 4 and 6 months for analysis.

*Analytical methods*

*Colour.* This was determined by the colour Wesson method using Lovibond glasses and calibrated according to Cocks & Van Rede (1966).

*Refractive index and melting point.* These were determined as described in AOAC (1975).

*Moisture content, free fatty acids, peroxide value, iodine value and saponification value.* These were determined as described in AOCS (1974).

*Vitamin contents.* Vitamin E was determined complexometrically and vitamin A colorimetrically as described by Freed (1966).

*Protein content.* Digestion by sulphuric acid followed by Kjeldahl distillation.

*Fatty acids content.* This was performed by gas liquid chromatography. The methyl esters of fatty acids were prepared as described in AOAC (1975). The gas chromatographic analysis was carried out using a GCD Pye Unicam gas chromatograph equipped with a dual flame ionization detector. The coiled glass column (1.5 m × 4 mm) was packed with Diatomite (100–120 mesh) and coated with 10% polyethylene glycol adipate. The column was operated isothermally at 190°C, detector and injection temperature was 220°C. Flow rate was nitrogen 30 ml/min, hydrogen 30 ml/min, air 330 ml/min and sample size was 2 $\mu$l.

*Spectral analysis.* This was carried out as described by Chapman (1965) using a Unicum SP 1100 infrared spectrophotomter.

## Results and discussion

*Physico-chemical characteristics of the samples*

Results in Table 1 illustrate the physical properties and chemical composition of two brands of ghee, three brands of butter oil and two brands of hydrogenated oils. The data demonstrate variation in red colour intensity between the samples, ghee Royal-Pack had the highest value (6.4) and butter oil Cream of Paris brand had the lowest value (4.5). The hydrogenated oils Nefertiti brand had a colour intensity of 10 while the colour of Crisco brand (1.0) was almost white.

Milk fat samples showed little differences in the refractive index, melting point and saponification number values. The iodine value was somewhat higher in the butter oil Excellanta brand. The two hydrogenated oil brands differed markedly in the melting point and iodine number values but were almost similar in the refractive index and saponification number values.

The peroxide values of the fat samples were in the range of 1.1 to 4.2 mEq peroxide/kg well below the 10 mEq limit (Davis, 1968; Egan, Kirk & Sawyer, 1981). In

**Table 1.** Physico-chemical characteristics of milk fat and hydrogenated oils samples

| Analysis | Ghee brands | | Butter oil brands | | | Hydrogenated oils brands | |
|---|---|---|---|---|---|---|---|
| | Baladi | Royal-Pack | Cream of Paris | Excellanta | Frico | Nefertiti | Crisco |
| Colour, Lovibond R/Y 5.25 in. cell | 5.6/35 | 6.4/35 | 4.5/35 | 5.0/35 | 5.0/35 | 10/35 | 1/10 |
| Refractive index (40°C) | 1.451 | 1.450 | 1.451 | 1.452 | 1.451 | 1.456 | 1.457 |
| Melting point (°C) | 32 | 30 | 30 | 30 | 31 | 36 | 43 |
| FFA % (oleic acid) | 0.8 | 0.3 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 |
| Peroxide value | 3.7 | 1.1 | 1.5 | 2.0 | 1.8 | 4.2 | 1.2 |
| Iodine value | 36.1 | 35.6 | 35.6 | 40.2 | 35.8 | 74.6 | 89.4 |
| Saponification value | 215.3 | 214.7 | 214.0 | 213.0 | 215.5 | 190.4 | 190.9 |
| Moisture (%) | 0.34 | 0.26 | 0.18 | 0.23 | 0.12 | 0.26 | 0.19 |
| Vitamin A (μg/100 g) | 343 | 432 | 523 | 714 | 717 | — | — |
| Vitamin E (mg/100 g) | 43 | 86 | 104 | 117 | 122 | — | — |

general, the peroxide value was much higher in the ghee Baladi brand and in the hydrogenated oil Nefertiti brand than other fat samples. Free fatty acids (FFA) as oleic acid were below the permissible limit of 0.3% (EOS, 1976; IDF, 1977; Egan *et al.* 1981) except in the ghee Baladi brand which had a very high percentage (0.8%).

Analysis showed that all samples were protein free and moisture contents were below the limit of 0.3–0.4%. Vitamin A content was in the range of 343–717 μg/100 g, and vitamin E content ranged from 43 to 112 mg/100 g with higher values in butter oil samples than in ghee samples. This could be attributed to the inherent compositional differences between milks and/or animal feeding and climate (Hartman & Dryden, 1972).

**Table 2.** Fatty acid composition of milk fat and hydrogenated oils samples

| Fatty acids | | Ghee brands | | Butter oil brands | | | | Hydrogenated oils brands | |
|---|---|---|---|---|---|---|---|---|---|
| | | Baladi | Royal Pack | Cream of Paris | Excellanta | Frico | | Nefertiti | Crisco |
| Saturated | C8 | 1.6 | 0.9 | 1.2 | 1.3 | 1.9 | | — | — |
| | C10 | 3.1 | 2.1 | 2.8 | 2.7 | 4.0 | | — | — |
| | C12 | 3.7 | 3.7 | 4.6 | 3.8 | 4.6 | | — | — |
| | C14 | 13.2 | 14.1 | 14.4 | 10.8 | 12.3 | | 0.5 | 0.2 |
| | C16 | 32.4 | 30.7 | 30.7 | 30.4 | 25.0 | | 19.9 | 15.0 |
| | C18 | 14.4 | 15.9 | 13.7 | 15.4 | 17.3 | | 5.5 | 13.8 |
| Total | | 68.4 | 67.4 | 67.4 | 64.4 | 65.1 | | 25.9 | 29.0 |
| Unsaturated | C14–1 | 3.0 | 1.6 | 1.7 | 2.3 | 2.1 | C16–1 | 0.3 | — |
| | C16–1 | 2.2 | 1.6 | 1.4 | 1.5 | 1.9 | C18–1 | 61.9 | 43.0 |
| | C18–1 | 26.4 | 29.4 | 29.5 | 31.8 | 30.9 | C18–2 | 11.9 | 26.9 |
| | | | | | | | C18–3 | — | 1.2 |
| Total | | 31.6 | 32.6 | 32.6 | 35.6 | 34.9 | | 74.1 | 71.1 |
| Saturated/unsaturated | | 2.17 | 2.07 | 2.07 | 1.81 | 1.87 | | 0.35 | 0.41 |

*Fatty acid composition*

Gas chromatography analysis (Table 2) showed that milk fat samples contained C8–C18 saturated fatty acids and C14:1, C16:1 and C18:1 unsaturated fatty acids. In hydrogenated oil, the saturated fatty acids were C14, C16 and C18; the unsaturated fatty acids were principally C18:1 and C18:2, with small amounts of C16:1 in Nefertiti brand and C18:3 in Crisco brand.

The major saturated fatty acids (C14, C16 and C18) varied considerably between the samples. The highest values were C14 in butter oil Cream of Paris brand, C16 in ghee Baladi brand and C18 in butter oil Frico brand. Hydrogenated oils Nefertiti brand contained the higher amounts of C14 and C16 and the lesser amount of C18 than the Crisco brand.

Similarly oleic acid (C18–1) the predominant unsaturated fatty acid varied between the samples, being higher in the Excellanta brand for milk fat samples and in the Nefertiti brand for hydrogenated oils samples. Moreover, the saturated/unsaturated fatty acid ratios ranged from 1.81 to 2.17 for milk fat samples and were higher in the ghee samples than in the butter oil samples. The value for hydrogenated oils was 0.35 for Nefertiti brand and 0.41 for Crisco brand. These apparent variations in fatty acid content are expected as there are many factors which affect fatty acid composition (see Kurtz, 1972).

*Spectral analysis*

Examination of the samples by infrared spectroscopy showed similar overal spectral patterns and similar absorbance maxima. Since various wavelengths represent certain functional groups (O'Connor, 1956; Chapman, 1965), the results indicate that similar functional groups are present in all samples (see Table 3).

*Storage stability*

The storage stability tests were carried at 20 and 35°C corresponding to mild and tropical conditions. Results in Table 4 show that free fatty acids developed in all samples during storage. But the rate of increase was higher in samples stored at 20°C than those stored at 35°C. Ghee samples showed the greatest rate of increase in the FFA, especially Baladi brand which had very high initial rate reaching the values of 1.2 and 1.0 after 6 months of storage at 20 and 35°C, respectively. In the other fat samples

**Table 3.** Infrared spectra of milk fat and hydrogenated oil samples

| Functional group | Wavelength λ (per cm) |
|---|---|
| R—C—$H_3$ ($\lambda_{as}$ $CH_3$, $\lambda_s$ $CH_3$) | 2940 |
| $R_2$—C—$H_2$ ($\lambda_{as}$ $CH_2$, $\lambda_s$ $CH_2$) | 2850 |
| C = O (—CO.OR) | 1745 |
| C—C—$H_3$ ($S_{as}$ $CH_3$) | 1465 |
| C—C—$H_3$ ($S_s$ $CH_3$) | 1375 |
| S—OH, λC—O (—CO.OH) | 1240 |
| λC—O (—CO.OR) | 1170 |
| SOH | 1110 |
| γCH | 965 |
| γOH | 945 |
| S $CH_2$ | 725 |

**Table 4.** Development of free fatty acids (FFA) in anhydrous milk fat and hydrogenated oils after storage for 6 months at 20 and 35°C

| Storage period (months) | | Ghee brands | | Butter oil brands | | | Hydrogenated oils brands | |
|---|---|---|---|---|---|---|---|---|
| | | Baladi | Royal-Pack | Cream of Paris | Excellanta | Frico | Nefertiti | Crisco |
| 20°C | 0 | 0.8 | 0.3 | 0.2 | 0.2 | 0.2 | 0.2 | 0.2 |
| | 1 | 1.0 | 0.5 | 0.4 | 0.4 | 0.3 | 0.3 | 0.3 |
| | 2 | 1.0 | 0.5 | 0.4 | 0.4 | 0.3 | 0.3 | 0.3 |
| | 4 | 1.0 | 0.7 | 0.4 | 0.4 | 0.3 | 0.3 | 0.4 |
| | 6 | 1.2 | 0.7 | 0.5 | 0.5 | 0.3 | 0.3 | 0.4 |
| 35°C | 1 | 0.8 | 0.3 | 0.3 | 0.3 | 0.3 | 0.3 | 0.3 |
| | 2 | 0.9 | 0.4 | 0.3 | 0.3 | 0.3 | 0.3 | 0.3 |
| | 4 | 0.9 | 0.4 | 0.3 | 0.3 | 0.3 | 0.3 | 0.4 |
| | 6 | 1.0 | 0.5 | 0.3 | 0.4 | 0.3 | 0.3 | 0.4 |

Free fatty acids (FFA) as % oleic acid.

the extent of increase was not large and after 6 months of storage at 35°C the FFA content were still within the permissible limit.

Table 4 suggests that ghee samples were much more prone to hydrolytic rancidity development than butter oil and hydrogenated oils samples. This is rather surprising since it was expected that the involvement of a high temperature (140°C) in the processing of this type of anhydrous milk fat would inhibit the lipolytic activity. There is a possibility of the presence of heat stable enzymes produced by the raw milk microflora and/or bacterial contamination during canning.

In contrast to FFA development, the rate of increase in the peroxide values (PV) was much higher during storage at 35°C than at 20°C. Data in Table 5 illustrate that the PV increased after 6 months of storage at 20°C from about 1.1–2.0 to 4.5–5.5 in most fat samples. Higher values were recorded in ghee Baladi brand (27.5) and in hydrogenated oils Nefertiti brand (23.9) for reasons which are obscure. Since the maximum PV

**Table 5.** Peroxide value changes in anhydrous milk fat and hydrogenated oils during storage at 20 and 35°C for 6 months

| Stored period (months) | | Ghee brands | | Butter oil brands | | | Hydrogenated oils brands | |
|---|---|---|---|---|---|---|---|---|
| | | Baladi | Royal-Pack | Cream of Paris | Excellanta | Frico | Nefertiti | Crisco |
| 20°C | 0 | 3.7 | 1.1 | 1.5 | 2.0 | 1.8 | 4.2 | 1.2 |
| | 1 | 6.7 | 3.7 | 2.6 | 3.5 | 2.4 | 9.7 | 2.6 |
| | 2 | 10.8 | 5.2 | 3.8 | 5.7 | 3.7 | 12.4 | 4.3 |
| | 4 | 16.8 | 5.6 | 4.2 | 5.8 | 3.2 | 19.5 | 4.3 |
| | 6 | 27.5 | 5.5 | 4.5 | 5.4 | 4.5 | 23.9 | 4.6 |
| 35°C | 1 | 20.3 | 6.3 | 3.0 | 4.2 | 2.4 | 13.1 | 5.5 |
| | 2 | 60.4 | 53.0 | 8.5 | 13.0 | 7.2 | 24.0 | 23.6 |
| | 4 | 76.0 | 68.5 | 22.8 | 25.9 | 12.3 | 33.2 | 29.9 |
| | 6 | 86.8 | 74.9 | 32.7 | 35.2 | 20.8 | 35.9 | 30.7 |

attained were mostly less than 20, this has no practical significance for the oxidative rancidity development (Egan *et al.*, 1981).

However, at 35°C all samples showed a considerable increase in PV as the storage time proceeded. The rate of increase was, however, very fast in the two ghee brands to the extent that after only 2 months of storage the PV were as high as 60.4 and 53.0 and increased further after 6 months of storage. The other fat samples showed a slower rate of increase reaching the values between 20.8 and 35.9 after 6 months of storage.

Unfavourable flavour was detected in the ghee samples after 2 months of storage, while the rest of the samples hardly showed any changes. The variations in the rate of oxidative rancidity development between fat samples in general and between ghee and butter oil samples in particular could be attributed to the differences in natural antioxidant content (vitamins A and E content). Results in Table 1 support this view. Variation in methods of processing and canning conditions could be other factors.

## Conclusion

The results of this study show remarkable variations between market distributed fats, but in general the compositional quality was satisfactory.

Free fatty acid development results demonstrated that hydrolytic rancidity is not a problem in anhydrous fats when stored under tropical conditions. However, oxidative rancidity is the principal problem in deterioration of the fat.

The stability of butter oil Frico brand to oxidative rancidity demonstrated the importance of natural antioxidant content (vitamins A and E, Table 1). The failure of ghee samples to show good keeping quality suggest that greater attention should be paid to the manufacture of such fat.

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# Effects of the retting of cassava on product yield and cyanide detoxication

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## Summary

Cassava toxicity is a factor that limits the utilization of cassava. A traditional processing technique, referred to as retting, that effects cyanide detoxication in cassava products was investigated. Cassava retting is a technique of long soaking of cassava roots in water to effect the breakdown of tissues and extraction of the starchy mass. A simulation of the technique followed by sun drying, showed that loss of starch and soluble components caused an apparent rise in percent crude fibre in the products. The rise in percent fibre and root peel/pulp ratio showed an inverse relationship with product yield. The technique of retting followed by sun drying removed up to 98.6% of initial cyanide in the roots, the average cyanide content of the product being 6.9 ppm; this is over 20-fold more efficient than simple sun drying.

## Introduction

Cassava (*Manihot esculenta* Crantz) constitutes one of the major staple foods for an estimated 300 million people in the tropical world (FAO, 1968). Africa, Latin America and Asia produce about 42, 30 and 28%, respectively, of the developing world total production of about 105 million t/year (FAO, 1978).

Fresh cassava, among other roots and piths, has a very short storage life, and it must be used or processed into durable forms soon after harvest. However, one of the main reasons for cassava processing is the removal of toxic cyanide from the products. Among the cassava cultivars and wild species no acyanogenic plants have been found (Sadik, Okereke & Hahn, 1974), although the levels of the cyanoglucoside varies among the species. It is known that some traditional methods of cassava processing effect the removal of cyanide with varying degrees of efficiency (Coursey, 1973; Ayernor, 1981).

The problem of cassava toxicity has received increased attention in the last two decades (Nestel & McIntyre, 1973). Coursey (1973) and Ayernor (1976, 1981) have reviewed the various traditional food processes for cyanide detoxication in cassava; but it appeared that the retting technique had not been fully examined. However, attempts have been made in recent times to upgrade the cassava retting process by improving facilities for material handling and by raising production capacities (Nzieffe, 1983). Perhaps the most important suggestion by Coursey (1973) for investigation is direct field observations of traditional detoxication techniques for cassava and their critical evaluation using reliable analytical procedures.

Retting inevitably involves fermentation; it is one of the traditional techniques for cassava detoxication (Ayernor, 1981). The process is carried out, in most cases, by immersing fresh cassava roots in stagnant or slow running ponds or in large household

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pots until the cassava pulp becomes soft. This may take 5 days or more. The pulpy mass is then sun dried and pulverized into flour as an end-product.

The objectives of this study are to simulate as closely as possible this major mode of traditional cassava processing, and to evaluate product yield and the extent of cyanide detoxication.

## Materials and methods

*Cassava varieties used*

Three cassava types according to the classification of cooked tissue texture (Ayernor, 1977): (a) vitreous type (60444), (b) mealy type (ISU-2), and (c) texturally intermediate type (Calabar), were selected. These three major cultivars also represent the sweet (ISU-2), bitter (60444), and intermediate (Calabar) types of cassava in traditional terms of classification (Ayernor, 1977). All the cassava varieties were planted in the same field and harvested 12 month after of planting.

*Retting of cassava roots*

About 4 kg of eight to ten randomly selected cassava roots of similar sizes of about 30 cm long and 8 cm in diameter, unpeeled or peeled (but not cut into pieces) were immersed in water in the proportion of 6 l of stagnant tap water per kg of fresh roots in large earthen pots (of 50 l capacity) placed in the open for 5 days. There was no change or addition of fresh water during the retting period. The average day and night ambient temperatures were 30 and 28°C, respectively.

*Storage of cassava roots*

Cassava roots were stored in the shade in the same environment and for the same time as the retting experiment, in order to study the changes of sugar levels in the stored roots concomitant with the retting experiment.

*Sun drying of test samples*

Sun drying of fresh cassava roots was done on thin (5 mm) cross-sectional slices. The samples were put in desiccators, containing concentrated sulphuric acid, overnight, followed by sun drying until the materials were dried to about 8% moisture. The fresh sun dried samples were used as control in this experiment. Retted portions of cassava pulp and peel were similarly dried.

*Chemical analyses*

*Dry matter/moisture content.* Dry matter of fresh root samples was determined on thin slices (2 mm) as for moisture content by drying to constant weight in a forced air oven at 105°C.

For determinations of starch, total sugars, crude fibre and amylose in fresh cassava roots, 5 mm slices of the roots were dried in the oven at 50°C; the dried samples were ground to flour.

*Starch.* For the determination of starch, the AOAC (1980) Method No. 14.032 was used.

*Total sugars.* The AOAC (1980) Method No. 31.077 was used for the quantitative determination of total sugars.

*Crude fibre.* Crude fibre determination was done using the AOAC (1980) Method No. 7.065.

*Amylose.* The determination of amylose in the three cassava varieties was done on cassava flours prepared from batch samples of ten roots according to the method of Juliano (1971).

*Cyanide.* In the preparation of samples for cyanide determination, grated and mixed materials were taken from proximal (head), middle and distal sections of ten fresh unretted roots; but for retted and sun dried materials (4 kg of fresh roots), the pulpy mass or flour was mixed thoroughly to ensure homogeneity. Cyanide was determined, using the enzymic assay method described by Cooke (1978).

*Treatment of results*

(a) Texture index of Cassava root tissues was calculated as the ratio of starch content of fresh roots to the moisture content.
(b) Peel/pulp ratio of roots was calculated as the ratio of the weight (on dry weight basis) of root peel to the weight of root pulp.
(c) Product yield was calculated as the percentage of the ratio of the dry weight of the retted product to the dry weight of unretted material.

Analytical results were reported as averages of three determinations from the batch samples, except fibre and amylose contents where the means with standard deviations from ten replicates from same batch samples were reported.

## Results and discussion

*Raw material and the retting process*

The retting process causes the breakdown of starch storage tissues and the release of cyanide. In order to understand the disintegration of the starchy tissues and the release of the toxic principle from the starchy mass, three different textural types of cassava tissues were selected. These represent two extremes with a floury or mealy type (ISU-2), and a vitreous type (60444), and an intermediate type (Calabar variety). The textural types of cooked cassava tissues (Ayernor, 1977) and the starch/water ratio or texture index of the test materials are shown in Table 1.

It was observed that the rate of tissue disintegration by the retting process did not follow a definite pattern with respect to texture index or hardness of cooked tissues. The texturally intermediate type (Calabar variety) was the most resistant to tissue

**Table 1.** Texture profile of cooked tissues and texture index of raw tissues of cassava roots used in the experiment

| Cassava variety | Texture profile* | | | | Texture index |
|---|---|---|---|---|---|
| | Mealy | Soggy | Gummy | Vitreous | |
| ISU-2 | +++ | – | – | – | 0.446 |
| Calabar | + | – | + | + | 0.385 |
| CA60444 | – | – | – | +++ | 0.293 |

*+++ most, ++ less, + least, – absent textural characteristics.

breakdown by the retting process. It is known that the textural characteristics of starchy tissues are not dependent on starch content alone but also on the nature of the cell wall and intercellular or middle lamellae (Mohsenin, 1970; Ayernor, Brennan & Rolfe, 1974); and that the rate of tissue breakdown through retting could not be attributed only to starch content.

The carbohydrate profile (starch–amylose–total sugars–crude fibre) of the test samples which was used as a factor for understanding the major differences among the raw test samples are shown in Table 2. Cassava ISU-2 contained 28.4% starch and 21% amylose, similar to the amylose level in other roots and piths such as the yams and potato (Rasper, 1967). The Calabar variety contained about 26% starch and 17.7% amylose which represents the average amylose level for cassava in the literature (Jones. 1959). The vitreous variety (60444) showed 21.5% starch and about 15% amylose, a level below the average amylose content for roots and tubers (Rasper, 1967). The sugar content for roots did not show a definite pattern with respect to starch and amylose contents (Ketiku & Oyenuga, 1972), but starch and amylose contents had been shown to determine the texture of cooked cassava tissues (Ayernor, 1977).

**Table 2.** Dry matter, starch, amylose, and total sugars and crude fibre contents of fresh cassava root samples used in the retting fermentation

| Cassava variety | Dry matter | Starch content (% wet wt) | Amylose content (% dry wt) | Total sugars content (% dry wt) | Crude fibre (% dry wt) |
|---|---|---|---|---|---|
| ISU-2 | 36.4 | 28.4 | 21.0±0.22 | 2.6 | 1.8 |
| Calabar | 33.0 | 25.8 | 17.7±0.23 | 1.7 | 2.7 |
| CA60444 | 26.6 | 21.5 | 15.6±0.65 | 3.1 | 2.1 |

*Retting and product yield*

Cassava roots are known to demonstrate increase in sugar content during post-harvest storage (Ketiku & Oyenuga, 1972; Booth *et al.*, 1976). Since sugar is readily fermentable, an attempt was made to evaluate the changes in sugar content in cassava roots stored alongside the retting experiment. The changes in total sugars in roots stored at ambient conditions similar to the retting experiment are shown in Fig. 1. The level of total sugars in Calabar variety rose from 1.7 to 7.2% (dry weight basis) in 3 days; ISU-2 variety demonstrated a rise from 2.6 to 6.1%, and 60444 variety showed an increase in total sugars content from 3.1 to 4.5% in the same period. The percent rise in sugars in the stored roots did not correlate to total product loss in the retted products. This may be attributed to the high loss of starch, especially in those samples retted without the covering peel, or due to different conditions of sugar formation when the roots are soaked.

The rise in percentage of fibrous material and the ratio of peel to pulp, at the end of the retting period, were used to monitor the loss of fermentable components in relation to the yield of the endproduct (Table 3). In ISU-2 variety, the fibre content rose from 1.8 to 2.1% in the product (pulp only) retted with the peel, and to 2.5% in that retted without the peel. Similar trends were demonstrated by the other two varieties (Table 3). The peel of the cassava root was relatively resistant to tissue breakdown within the time limit of the experiment, but the root flesh or pulp yielded readily to tissue

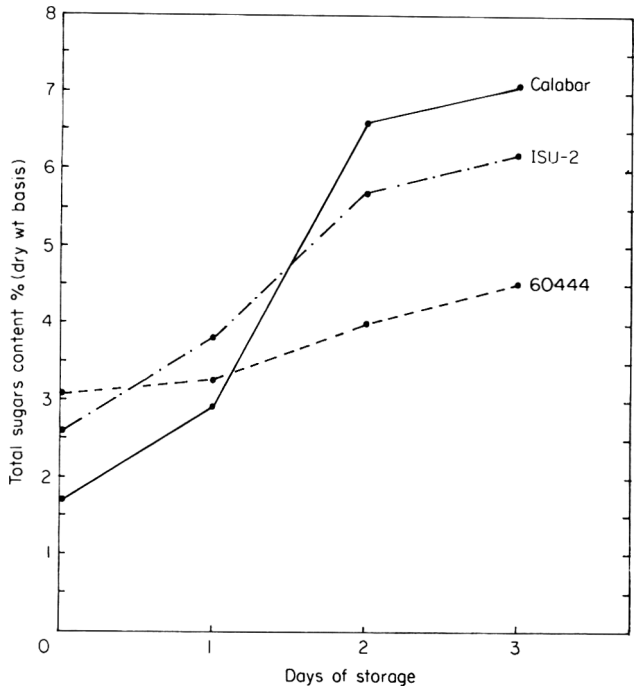


**Figure 1.** Changes in total sugars content in cassava roots durir g storage.

disintegration. The peel/pulp ratio clearly demonstrated a reliable index for relating loss of fermentable components in the samples during the retting process. The lower the the peel/pulp ratio, the higher the endproduct yield. In ISU-2 sample, the unretted material had peel/pulp ratio of 0.091 and 0.123 in the retted product. The retted samples showed significant weight losses and much more so in roots retted without the peel. In ISU-2 sample, the product yield (pulp, on dry weight) of the roots retted with and without the peel were 88 and 79%, respectively. Similar trends prevailed in the other two batches with cassava 60444 showing as low as 65% product yield in the peeled retted material (Table 3); this is indicative of loss of starch during the retting process.

**Table 3.** Fibre content, peel–pulp ratio and product yield of retted and unretted cassava products

| | Fibre content (%) (dry wt basis) | | | Peel/pulp ratio (dry wt basis) | | Product yield (dry wt basis) (% of unretted) | |
|---|---|---|---|---|---|---|---|
| Cassava variety | Peeled unretted root | Unpeeled (whole retted root) | Peeled retted root | Unretted root | Retted root | Unpeeled retted root | Peeled retted root |
| ISU-2 | 1.8±0.02 | 2.1±0.09 | 2.5±0.11 | 0.091 | 0.123 | 88 | 79 |
| Calabar | 2.7±0.9 | 3.2±0.05 | 3.8±0.08 | 0.120 | 0.173 | 86 | 74 |
| CA60444 | 2.1±0.01 | 3.5±0.08 | 3.8±0.12 | 0.101 | 0.162 | 74 | 65 |

*Cyanide detoxication*

The principle underlying the removal of cyanide in cassava is based on the rupture of the tissue to cause the enzyme, linamarase, to come in contact with the substrate, the glycocyanide (Butler, Reay & Tapper, 1973; Coursey, 1973; Ayernor, 1976, 1981) which may lead to the release of cyanide ion ($CN^-$) (Cooke, 1978). The hydrocyanic acid which may be formed volatilizes readily, especially under drying or cooking conditions (Ayernor, 1976). This principle is the basis for traditional cassava processing and the detoxication of cassava products. However, the retting process appears to be more complex than direct enzyme hydrolysis and weak acid volatilization; it involves also slow tissue breakdown, fermentation, and leaching of soluble materials into the soaking water (Coursey, 1973; Ayernor, 1981).

The results on the efficiency of cyanide reduction by the retting process are shown in Table 4. The unretted ISU-2 cassava peel contained 91.66 mg NCN/100 g sample, however, this concentration was reduced to 10.55 in the retted, and to 3.8 in the retted and subsequently sun dried samples. The estimated reductions in cyanide content in the retted and retted–dried peels were 88.5 and 95.9%, respectively. Roots retted with the peel (but with the peel removed after retting) showed reductions in cyanide in the pulp by 96.2 and 98.6% in the retted pulp and retted–dried pulp, respectively. Similar patterns and magnitudes of cyanide removal by retting and subsequent drying are demonstrated by the other two varietal samples (Table 4). It is evident that retting only, without subsequent drying can effect a considerable detoxication, and that with subsequent drying, the total cyanide content in the ultimate product can be reduced to trace levels such as the estimated 5.4 ppm cyanide in an end product (Table 4).

**Table 4.** Total cyanide content in unretted and retted cassava products

| Cassava variety | Unretted root cyanide: mg/100 g root (wet* and dry wt basis) | | | Retted root cyanide: mg/100 g root (dry wt basis) | | | Sun dried root cyanide: mg/100 g root (dry wt basis) |
|---|---|---|---|---|---|---|---|
| | Peel only | Whole root (with peel) | Root without peel | Peel only | Whole root retted with peel/pulp values only | Root without peel | Root without peel |
| ISU-2 | (33.20) | (28.60) | (23.41) | 3.80† | 1.10† | 0.80† | 15.70 |
| | 91.66 | 79.44 | 65.02 | 10.55 | 3.05 | 2.22 | |
| Calabar | (18.80) | (15.60) | (11.70) | 3.51† | 1.20† | 0.54† | |
| | 56.96 | 47.27 | 35.45 | 10.63 | 3.63 | 1.63 | 12.30 |
| CA60444 | (20.70) | (18.40) | (15.15) | 4.90† | 1.10† | 0.74† | |
| | 79.61 | 70.76 | 58.27 | 18.84 | 4.23 | 2.84 | 17.90 |

*Wet weight values in parentheses.

†Retted sun dried values.

For comparison, the sun dried three different varietal root samples, which were not subjected to the retting process, showed reductions in cyanide content of 75.8, 65.3 and 69.2%, corresponding to 157, 123 and 179 ppm cyanide, respectively. It is obvious that the apparent relatively efficient cyanide reduction in the unretted but simply sun dried

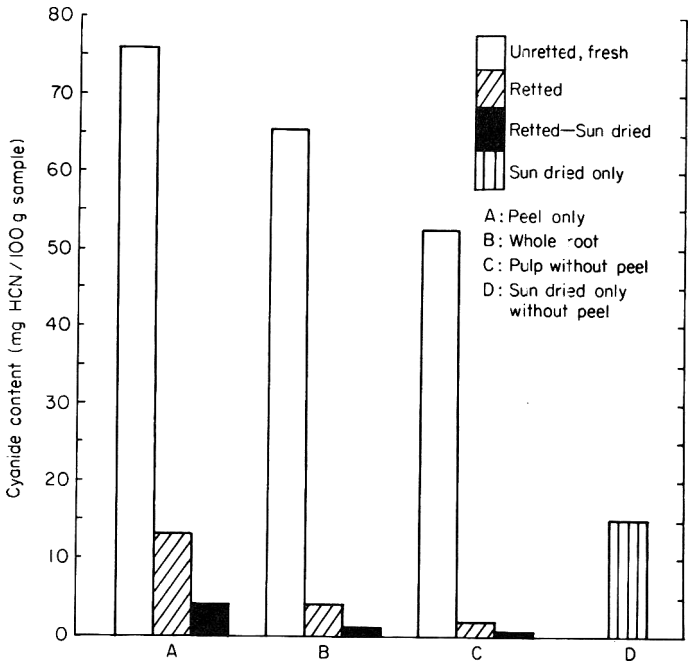


**Figure 2.** Summary of the efficiency of cyanide detoxication in cassava products by the retting process.

products is due to the thin (5 mm) slices which had undergone some tissue rupture and increase in total surface area for cyanide loss; and that cyanide loss might be less if larger tissue pieces were exposed to sun drying.

The average values for the three different cassava batches with respect to the efficiency of the retting process is illustrated and summarized in Fig. 2. Though the peels were relatively resistant to tissue breakdown, the retting process followed by sun drying was able to effect cyanide reduction from 761 to 41 ppm in the treated peels. The average value for cyanide in the end product of roots retted with the peel was 11 ppm; and for the roots treated without the peel, the ultimate product contained 6.93 ppm cyanide compared to 152 ppm cyanide in the unretted but simply sun dried product.

## Conclusion

The significance of this study is that it has been able to ascertain that the traditional retting process is efficient in cyanide detoxication; and that it is possible to produce, low cyanide cassava flours suitable for food utilization. However, in applying such a process, attempts should be made to minimize product losses.

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# Heat stability of concentrated milk: enhancement of initial heat stability by incorporation of food grade lecithin

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## Summary

Food grade lecithin derived from soya beans can promote large increases in the initial heat stability of full cream evaporated milk. By means of lecithin incorporation, heat stable concentrate can be manufactured without addition of inorganic phosphate and can be processed at higher than usual homogenization pressures. As a result, the efficiency of reduction of fat globule size is enhanced. Limited storage trials have indicated no deleterious effects associated with the use of lecithin.

## Introduction

Homogenization of concentrated milk usually results in a marked decrease in initial heat stability. The effect is of commercial significance, for without adequate homogenization, creaming of milk fat occurs during storage. The extent of creaming is time dependent and whilst product may be acceptable after 6 months storage, when the same concentrate is examined 1 year later fat separation becomes unacceptable. The problem can be minimized by increasing the severity of homogenization but, at certain seasons, this results in inadequate stability for subsequent sterilization. The problem has been exacerbated by the changing market for full cream evaporated milk. Whilst domestic sales have declined, a growing export market for the product has developed and, as a result, the shelf life requirement of concentrate has been extended from less than 1 year to over 2 years. Comparatively little attention has been focussed on the heat stability of evaporated milk in recent times (cf. Rose, 1963) because empirical solutions to most processing problems have been developed. Nevertheless, with the growing importance of export markets for U.K. milk some new studies have been initiated.

There is significant seasonal variation in the heat stability of homogenized concentrated milk (Sweetsur & Muir, 1982a) which is in part the result of changes in the soluble calcium concentration. The concentration of soluble calcium is closely related to the citrate level (Holt & Muir, 1979) and this is amenable to dietary manipulation (Holt *et al.*, 1979). Methods of manipulation of heat stability have also been considered and Sweetsur & Muir (1982b) defined optimal conditions for salt addition, pH, homogenization temperature and processing order. Nevertheless, even with optimal processing conditions it is difficult in early spring and late autumn to produce adequately homogenized evaporated milk which will withstand sterilization.

Sweetsur & Muir (1983) showed that interactions involving sulphydryl group containing fractions are particularly important in determining the heat stability of

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homogenized concentrated milk. When sulphydryl group interactions are inhibited by addition of copper or *N*-ethylmaleimide, or when low levels of iodate are added, the effect of homogenization on heat stability was suppressed. Unfortunately, none of the successful additives were appropriate for use in foodstuff. Nevertheless, it is known that the family of related phosphatides, commonly known as 'lecithin', interact with a number of milk fractions, including those bearing sulphydryl groups. Barratt & Rayner (1972), Patrick *et al.* (1972) and Barratt, Austin & Whitehurst (1974) have demonstrated interactions of lysolecithin with acid, $\alpha_{s1}$, $\beta$ and $\kappa$ caseins whilst Korver & Meder (1974) and Hanssens & van Cauwelaert (1978) have found evidence for lysolecithin interaction with $\beta$ lactoglobulin and $\alpha$ lactalbumin, respectively.

However, Green (1971) found that lecithin did not stabilize either $\alpha_s$ or $\beta$ casein against precipitation by ionic calcium. In addition, Shalabi & Fox (1982) stated that, whilst addition of lysolecithin to skim milk displaced the heat coagulation time (HCT)–pH profile of skim milk to lower pH values, the shape of the HCT-pH profile and the overall level of heat stability were unchanged. On the other hand, Maxcy & Sommer (1954) found that lecithin addition promoted a 4-fold increase in heat stability of recombined evaporated milk and Leviton & Pallansch (1962) reported a similar improvement in both the heat and storage stability of conventional and recombined evaporated milk. Against this apparently divergent background, the potential use of lecithin to enhance the heat stability of full cream evaporated milk has been investigated.

## Methods and materials

Full cream evaporated milk was prepared as described in Hardy *et al.* (1984). Lecithin (Emulfluid A, a strongly polar soya lecithin which is water dispersible, Lucas Meyer (U.K.) Ltd, Chester, U.K.) was added to the concentrate and dispersed with a high speed mixer (Silverson Machines Ltd, Chesham, U.K.). Subsequently, the concentrate was homogenized, canned and sterilized as in Hardy *et al.* (1984). With the exception of one series of experiments, sodium phosphate ($Na_2HPO_4$) was added at a level of 0.15% w/w to all concentrates.

The HCT was measured as described in Sweetsur & White (1975) and grain point defined as in Hardy *et al.* (1984).

Milk and ultrafiltrate for salt analyses were prepared as described in Holt *et al.* (1978), except that protein in the samples prepared for calcium analyses was precipitated with tungstic acid (sodium tungstate, 1% w/v in sulphuric acid 0.1 N containing orthophosphoric acid, 1 drop/l).

The concentration of total and soluble calcium in milk and ultrafiltrate as estimated with a Corning 940 calcium analyser (Corning Ltd, Halstead, U.K.) and that of inorganic phosphate by the method of Allen (1940) as modified by White & Davies (1958).

The level of chloride in milk and ultrafiltrate was estimated according to the appropriate British Standard (BSI, 1963).

When samples of ultrafiltrate were required from concentrate, the material was subjected to a preliminary centrifugation (MSE Hi-Spin 21 centrifuge, MSE Scientific Instruments, Crawley, U.K.) for 15 min at 33 000 *g* (using an 8×50 ml sample, angle rotor, 20°C). The fluid middle portion of each sample was then siphoned off leaving a solid—predominantly fat—layer at the surface and a sediment comprised mostly of protein. This 'serum' was then further separated by ultrafiltration to yield a colloid free

serum. (A TCF-10 ultrafiltration unit, Amicon Ltd, Stonehouse, U.K., fitted with a 90 mm, type PM 30 membrane was used.) The preliminary clean-up procedure results in slightly variable removal of fat and protein from concentrate with associated changes in the extent of volume reduction. This problem was partially overcome by assay of chloride ion in the concentrate and in the colloid free serum. Since the chloride is not partitioned the apparent increase in chloride concentrate is a measure of solids removed. Hence the actual concentrations of calcium and phosphate in the colloid free serum can be corrected to yield the concentration of soluble (but partitioned) salts in the original concentrate, e.g.:

$$\text{Soluble calcium} = \frac{\text{measured serum calcium} \times \text{total chloride in conc.}}{\text{serum chloride}}$$

It should be noted that the Donnan effects are not compensated for in this method, and although small, this renders the values comparative rather than absolute.

## Results

*Effect of lecithin addition on heat stability of evaporated milk*

Lecithin addition (0.2% w/w) significantly increased the HCT of full cream evaporated milk between pH 6.5 and 7.0 (Fig. 1). There was no change in either the shape of the HCT–pH profile or in the natural pH of the respective concentrates. These results contrast with those of Shalabi & Fox (1982) who found that, for skim milk, HCT was not increased, but that a pH shift of the entire HCT-pH profile occurred. Since no significant shift in pH occurred as a result of lecithin addition to concentrate the single point estimation of grain point also yields a true measure of the entire HCT-pH profile. The results of experiments carried out over a year are shown in Table 1. As expected, there was considerable variation in the grain point of full cream evaporated milk and this was particularly marked in the months when the diet of the cow undergoes its greatest change. For example, there was a wide variation in heat stability of milk samples processed in April and in October. Neverthless, in every case, incorporation of lecithin (0.2% w/w) resulted in a significant increase in heat stability. A wide range of

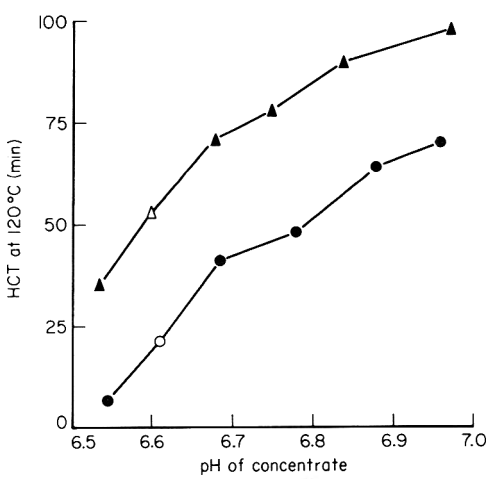


**Figure 1.** The effect of lecithin addition (0.2% w/w) on the HCT–pH profile of homogenized evaporated milk. ●, control; ▲, control + added lecithin. Open symbols represent natural pH.

**Table 1.** Effect of lecithin addition (0.2% w/w) on the grain point of full cream evaporated milk

| Month | Grain point (min at 120°C) | |
|---|---|---|
| | Control | Control + lecithin |
| April (a) | 20 | > 50 |
| May | 50 | > 50 |
| June | 40 | > 50 |
| September | 30 | > 50 |
| October (a) | 11 | > 50 |
| October (b) | 27 | 37 |
| October (c) | 4 | 24 |
| April (b) | 9 | 45 |
| Mean | 24 | 44 |

a–c = separate trials carried out in months where greatest changes in diet occur.

'lecithin' is available commercially, reputedly optimized for different types of food use. Therefore, the effect of changing the type of lecithin on its ability to stabilize full cream evaporated milk was considered. As shown in Table 2, stabilization ocurred with all products evaluated and the differences in HCT and grain point between the different products was small and of comparatively little practical significance. This observation is in agreement with the data—albeit limited—presented by Leviton & Pallansch (1962). They found that little difference was apparent when HTST sterilized concentrated milk was stabilized by a range of phosphatides.

**Table 2.** Effect of addition of various types of food grade lecithin (0.2% w/w) on the grain point of full cream evaporated milk

| Additive | Description* | Coagulation time (min, 120°C) | Grain point (min, 120°C) |
|---|---|---|---|
| Control | — | 10 | 9 |
| Emulfluid A | Strongly polar, rich in *N*-acyl-phosphatidyl ethanolamine | 49 | 45 |
| Emulfluid E | Hydrophilic, increased lysophospholipid content | 46 | 45 |
| Metarin P2 | Fractionated phospholipid, very surface active | 51 | 45 |
| Metarin F | Fractionated phospholipid, very surface active | 35 | 40 |

* All samples were obtained from Lucas Meyer Ltd and are approved for use in food.

*Effect of homogenization pressure on heat stability*

Samples of full cream evaporated milk were prepared—with and without lecithin addition—in which the homogenization pressure was varied from 24.2 to 41.4 MPa. As the homogenization pressure was increased, the efficiency of homogenization improved (Table 3). (A good practical measure of homogenization efficiency is given by the proportion of particles < 0.84 $\mu$m for this feature is related to extent of creaming—

**Table 3.** Effect of lecithin addition on homogenization efficiency of full cream evaporated milk

| Total homogenization pressure (MPa) | Particles < 0.84 μm (%) | |
|---|---|---|
| | Control | Control + Lecithin (0.2%) |
| 24.2 | 83.4 | 80.0 |
| 27.6 | 86.3 | 90.1 |
| 31.1 | 87.8 | 90.3 |
| 34.5 | 91.0 | 93.5 |
| 38.0 | 92.8 | 94.3 |
| 41.4 | 93.0 | 95.0 |

Banks, Muir & Wilson, 1982.) At all but the lowest applied pressure, lecithin incorporation resulted in a small (but statistically insignificant) increase in efficiency, although this effect was less than that of increasing pressure *per se* (which was statistically highly significant). Using initially stable concentrates it was found that as homogenization pressure increased, heat stability fell sharply (Fig. 2). It is unusual to treat concentrate at pressures greater than 31.1 MPa total pressure and to retain enough stability for the product to withstand sterilization. In the winter period when the heat stability of concentrate is particularly low (Sweetsur & Muir, 1982a), it is often difficult to retain stability at a pressure of 24.2 MPa (cf. Table 1). However, addition of lecithin (0.2% w/w) resulted in marked stabilization and, even at the highest pressure tested (41.4 MPa), initial heat stability was very high. Thus lecithin addition allows high homogenization pressures to be used in the manufacture of evaporated milk and offer a potential solution to the problems of creaming on long term storage.

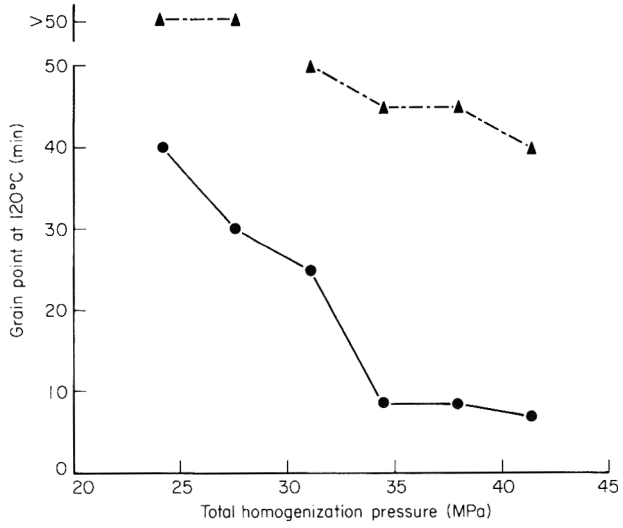


**Figure 2.** Effect of lecithin addition (0.2% w/w) on the grain point of homogenized evaporated milk. ●, control; ▲, control + added lecithin.

*Lecithin addition in the absence of phosphate stabilizer*

It is impractical to manufacture evaporated milk without the use of mineral salt addition and, of the salts permitted as additives, phosphates have the most significant effect (Sweetsur & Muir, 1980, 1982b). However, phosphate addition imparts a salty flavour to evaporated milk and, if used in excess, can result in the formation of mineral deposits from the milk during storage. Attempts were made to prepare evaporated milk with no added phosphate. The dose–response curve relating grain point to level of added lecithin for evaporated milk with no phosphate is shown in Fig. 3. At the previously used level of lecithin (0.2% w/w), the heat stability attained was inadequate for sterilization, but by increasing the level of lecithin to 0.4% w/w safe heat treatment could be assured. At higher levels of lecithin addition, proportionately higher stabilities were attained.

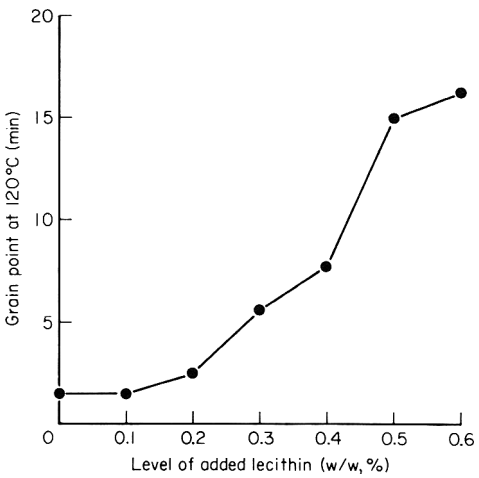


**Figure 3.** The effect of lecithin addition at various levels on the grain point of homogenized evaporated milk—no $Na_2HPO_4$ stabilizer was added.

*Lecithin addition and salt partition*

The phosphatides in food grade lecithin are charged and have the potential to bind calcium ions. Experiments were thus carried out to investigate the effect of lecithin addition on the mineral equilibrium of evaporated milk. Mean values for samples prepared in seven separate trials are shown in Table 4. Although there was slight

**Table 4.** The effect of lecithin addition on the partition of soluble calcium and inorganic phosphate in full cream evaporated milk

| | Control* | Control + 0.2% lecithin* |
|---|---|---|
| Total calcium (mM) | 65.2 (3.15) | 65.3 (2.83) |
| Soluble calcium (%) | 18.9 (1.42) | 19.1 (2.14) |
| Total phosphate (mM) | 59.7 (5.91) | 60.0 (7.16) |
| Soluble phosphate (%) | 35.9 (2.29) | 35.6 (2.61) |

*Results are mean of data from seven trials; s.d. in parentheses.

variability in results between trials, the mean values for soluble calcium and inorganic phosphate in evaporated milk were unchanged by incorporation of lecithin. This result is wholly consistent with the findings of Green (1971), for she observed that lecithin addition did not influence the precipitability of $\alpha_s$ or $\beta$ casein by calcium ion.

*Effect of lecithin on heat stability of skim milk*

Since lecithin did not appear to enhance heat stability by perturbations of mineral equilibrium, it seemed likely that the effect was associated with the homogenization process. Skim milk was concentrated to 20% total solids and homogenized as for evaporated milk (24.2 MPa total pressure), either with or without lecithin addition. Results from two separate trials are shown in Table 5. Irrespective of whether heat stability was assayed as HCT or grain point, the effect of lecithin addition to concentrated skim milk was slight and of no practical significance. A stabilization of slightly higher magnitude was found when the heat stabilities were compared before and after homogenization. All concentrates contained some residual fat, but this did not exceed 0.5%.

**Table 5.** Effect of lecithin addition and homogenization on heat stability of concentrated skim milk (20% TS)

| | Trial 1* | | Trial 2* | |
|---|---|---|---|---|
| Sample | HCT (min) | GP (min) | HCT (min) | GP (min) |
| Unhomogenized control | 13 | 9 | 14 | 12 |
| Homogenized control† | 16 | 12 | 22 | 17 |
| Unhomogenized + lecithin‡ | 13 | 9 | 16 | 14 |
| Homogenized + lecithin†‡ | 18 | 12 | 23 | 18 |

*HCT = heat coagulation time; GP = grain point; at 120°C.
†Concentrate was homogenized at a total pressure of 24.2 MPa.
‡Lecithin was added at 0.2% w/w.

*Order of lecithin addition*

In earlier work it has been noted that the order in which processing steps are carried out has a marked influence on the heat stability of concentrate (Leviton *et al.*, 1963; Sweetsur & Muir, 1982b). The position of lecithin addition was investigated by incorporating lecithin either before or after the homogenization step (Table 6). In trial A, in

**Table 6.** The effect on heat stability of the position of lecithin addition during the manufacture of evaporated milk

| | Heat coagulation time* (120°C), min | | Grain point* (120°C), min | |
|---|---|---|---|---|
| Sample | A | B | A | B |
| Control, no lecithin | 23 | 5 | 26 | 3 |
| Lecithin before homogenization | 45 | 12 | 38 | 23 |
| Lecithin after homogenization | 41 | 10 | 38 | 23 |

*Results from Trial A, added $Na_2HPO_4$ = 0.15%; Trial B, added $Na_2HPO_4$ = 0.075%.

which the normal level of phosphate stabilizer was added (0.15% w/w) no significant difference in heat stability was observed irrespective of the position of lecithin addition. This result was confirmed in another trial in which less phosphate (0.075% w/w) was added and in which the control sample was so unstable that sterilization could not be achieved.

*Storage stability*

A limited number of samples with added lecithin (0.2% w/w) have been stored at ambient temperature (15°C) for periods up to 1 year. No fat separation, gelation or sediment formation has been observed. More extensive trials are now under way.

## Discussion and conclusion

Lecithin addition has a significant effect on the heat stability of evaporated milk irrespective of the season in which the concentrate is manufactured. Its use allows complete or partial elimination of the need for phosphate inclusion to ensure stability during sterilization. In addition, incorporation of lecithin allows higher than usual homogenization pressures to be applied to evaporated milk with a consequent improvement in emulsion fineness and inhibition of creaming. Limited storage trials have also shown that lecithin incorporation does not result in deterioration of product quality and, it is anticipated, from work with UHT sterilized concentrate (Leviton & Pallansch, 1962), that improvements in stability may be attained. Lecithin is a permitted additive in U.K. for incorporation in dried milk but its use in evaporated milk for the domestic market is not sanctioned. Few limitations on its use for export markets are, however, extant and in this role it should find ready application.

The mechanism by which lecithin stabilizes full cream evaporated milk is not clear. Results are presented which suggest that the effect of lecithin is not mediated by changes in mineral equilibrium. The principal effect requires the presence of fat. However, lecithin does not simply displace protein during the homogenization process to stabilize the newly formed fat surface, for it is equally effective when added after homogenization. Lecithin may displace protein already on the fat surface or be adsorbed at interstices if the protection coating is incomplete. It has not been found practical to test these possibilities because of inherent problems of separation of the fat from the serum phase. Methods applicable to UHT cream (Anderson *et al.*, 1977) do not yield a clean phase separation because in evaporated milk a very wide range of particles of differing size and density exist. Further research to solve this problem is in hand. Notwithstanding the mechanisms suggested above, it is also possible that the stabilizing effect of lecithin is associated with its interaction with $\kappa$ casein or $\beta$ lactoglobulin (Korver & Meder, 1974) since such interactions play an important part in the destabilization of homogenized concentrated milk (Sweetsur & Muir, 1983). Irrespective of the manner in which lecithin stabilizes evaporated milk its use appears to offer a solution to several outstanding technological problems.

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# Book Reviews

**Microbiological Methods,** 5th ed. By C. H. Collins and Patricia M. Lyne.
London: Butterworth, 1984. Pp. x + 448. ISBN 0 408 70957 X. £16.00.

Twenty years ago the first edition of *Microbiological Methods* was published. The fifth edition bears out the authors' statement that '"Manual" microbiology still has a long way to go before automation takes over'; their compilation of methods for the examination of clinical material, food, drink and pharmaceuticals continues to emphasize practical manual techniques and observations.

Over the years the design of equipment has improved, disposables are widely used and some procedures can be speeded by better methods or rapid kits; but most microbiologists still rely on the same classical techniques and skills in their daily work. This volume, based mainly on techniques in use in the PHLS, is a catalogue of information on everyday equipment, laboratory safety, cultural and identification methods, with useful lists of suppliers of reagents, media and equipment.

The contents are directed primarily at workers in the Public Health sector, with emphasis on clinical methods, but the information on basic laboratory skills would be invaluable to all benchworkers and supervisors and the chapters on non-clinical material are a useful introduction for those requiring a simple listing of appropriate cultural methods for a wide variety of products.

In this edition there are many new references, reflecting the large body of information published on all aspects of microbiology in recent years; the text has been updated but the basic format is unchanged.

Although the typeface and clarity have been increased, making headings clearer and easier to read, the rather unstructured nature of the text causes problems in locating information within a chapter. Without using the index many nuggets of information become lost in the weight of listed material.

The book is a good source of practical detail, drawing on many years experience, and remains a useful bench guide for any laboratory worker.

*Kay H. Gordon*

**Basic Food Chemistry,** 2nd Ed. By Frank A. Lee.
Westport, Conn.: AVI, 1983. Pp. xi + 564. ISBN 0 87055 416 6. U.S.$35.00.

The initial impression of this book from thumbing through the pages is that it is similar in style and content to many other food science books, such as Fox and Cameron, and at $35 would seem to be considerably more expensive. However, a closer study of the contents reveals a somewhat different approach with chapters being devoted to the major food components: carbohydrates, lipids, proteins, vitamins, minerals, etc.; followed by a series of chapters describing the major food commodities: baked products, milk and milk products, coffee, tea, cocoa, meat and meat products and finally fruit and vegetables. This approach must inevitably lead to a certain amount of

overlap and although there is considerable cross-referencing between chapters—for example between the chapters on enzymes and alcoholic fermentation—some duplication has still crept in; for instance we are treated to two figures showing the structure of $\alpha$-lactose in chapters 4 and 16. This dual approach is interesting, but individual readers will have to decide whether the necessary increase in book length, and hence cost, is worthwhile.

The book is aimed at food science undergraduates and will undoubtedly provide a good basis in food chemistry. In addition to the chapters mentioned above it is pleasing to see more general chapters on diverse topics such as photosynthesis, water and solutions, colloids, flavour, colours and the browning reaction. Some of the chapters on the major food components, and indeed some of those on the commodities, do include outlines of analytical methods but there is no consistency in the depths of the descriptions given. However, this is not a food analysis book but rather a book showing the chemical composition of foods and the reactions of food components. The standard of presentation is generally good with clearly written text, but the diagrams are of very variable standard with several erroneous structures (e.g. three valent carbon on p. 77, double bond missing on p. 51, trigonelline and nicotinic acid structures wrong on p. 402, pyridine with a saturated ring on p. 411 and many more instances of ambiguous or unclear structures, in particular whether ring structures are aromatic or not). Each chapter concludes with a brief summary which is useful as it helps fix the main points. Also each chapter has an extensive bibliography but with only passing reference to the recent literature—for example the vitamin chapter contains very few references beyond 1970.

In conclusion, this book will provide a useful basic undergraduate text which could have been greatly improved by more detailed attention to figures in production. It is a relatively expensive volume but it does provide two separate approaches to food chemistry via major components or commodities. The prospective reader must decide whether this dual approach is helpful or not.

*R. Macrae*

**Developments in Food Analysis Techniques,** Vol. 3. Ed. by R. D. King. Barking, Essex: Elsevier Applied Science, 1984. Pp. x+217. ISBN 0 85334 262 8. £26.00.

To quote Euripides, 'the same man cannot well be skilled in everything; each has his special excellence'; this series of reviews of selected topics in food analysis gives a concise appreciation of the present knowledge and experience in specialized areas which are of current interest or concern.

All the authors and co-authors originate from government or educational establishments and so the reviews are a nmixture of theoretical specialization and practical experience.

The topics covered are the problems associated with the analysis of dietary fibre, trace element analysis, mycotoxins, pesticides and immunochemical methods in food analysis.

Dietary fibre has more definitions, in my opinion, than the variations of the Kjeldahl method for the determination of organic nitrogen—all based primarily on the chemistry of the components of dietary fibre. Here, that of Trowell in 1972 is quoted

and then qualified—i.e. we are not quite sure what we are determining in any one foodstuff. There then follows an exceedingly good review complete with flow diagrams which covers both the advantages and disadvantages of the methods at present available for the various constituents. Personally, I feel that a standardized empirical enzymic method will eventually be the answer, both on specificity and on economic grounds but, in saying that, everyone wishing to claim the quantitative presence of dietary fibre on the label of a prepacked food should read the concluding remarks.

The chapter entitled 'Trace element analysis' should really be qualified 'by atomic spectrometry' and was, to the reviewer, disappointing in that no mention was made of recent electrochemical or ion chromatographic methods of analysis; the one part which could be quoted many times is that on quality assurance, sampling and validation of analytical data.

The account of the determination of mycotoxins should be read by everyone concerned with the examination of food, the discussion on sampling and sample preparation is essential reading and, in the case of aflatoxins in peanuts, the subject of a 'code of practice'. There is a slight overlap with the next chapter on immunoassay techniques, but this is of little importance. The accounts of both mycotoxin and pesticide determinations are scattered with helpful tips and gives the impression of practical experience. The account on pesticides is particularly helpful, especially the multi-residue procedures, but I do like the warning that 'only by years of laboratory experience may one obtain a working knowledge of pesticide analysis'.

Some years ago there appeared in certain newspapers cartoons of kangaroos jumping into hamburger bars; as one who had to apply immunochemical methods to many hundreds of samples in a short time, the rapidity and elegance of the techniques are most outstanding, although it should be mentioned that interferences must be borne in mind. The review is well worth reading by students and all concerned with qualitative and quantative methods of detection of toxins, anabolic sex hormones, enzymes and many other materials, including 'foreign' proteins in meat products, such as soya protein and casein, and the detection of the meat of various origins.

Generally, anyone who is interested in food analysis should read this book, certainly students, both pre- and postgraduate; those in nutrition and enforcement laboratories will find much of interest. The references are adequate and up to 1982; it has a very clear and easily readable type making for easy reading on rainy Sundays and altogether will be of assistance to working analysts in the laboratory; as this is the aim of the editor, it is indeed successful.

*S. Landsman*

**Control of Food Quality and Food Analysis.** Ed. by G. G. Birch and K. J. Parker. Barking, Essex: Elsevier Applied Science, 1984. Pp. xi+332. ISBN 0 85334 239 3. £35.00.

This volume contains the papers presented at the 14th Annual Symposium of this series, held in March 1983 in Reading. A total of eighteen papers were read, and apart from the keynote address, each is intended to concentrate on a particular aspect of food quality, its assessment and control.

The papers may be divided into two broad categories. In the first group are those which are primarily concerned with methods of assessment, both chemical and

microbiological. They include reviews of recent developments in rapid instrumental methods of analysis such as near infrared spectrophotometry, light microscopy, immunological methods, rapid microbiological methods, and also a historical review of developments in sensory methods. There are particularly interesting contributions concerned with the problems associated with obtaining reproducible results in the analysis of food components, the possible influence on future legislation of improved knowledge of risk–benefit considerations regarding additives, and also fundamentals of control of microbiological quality. The second group of papers deals with factors influencing quality in certain commodities such as dairy products and meat, and also control of processing operations by means of models.

With a couple of unfortunate exceptions, the papers presented contain much useful information. It must be said, however, that in only relatively few cases do the authors address themselves to the problems of control of food quality. The contributions concerned with methodology will undoubtedly be of value to readers seeking up-to-date information regarding the various techniques, but the applications of the techniques in quality control tend to be overlooked. Similarly, the contributions dealing with commodities, again containing much useful information, are devoted mainly to the factors influencing quality rather than their control. All the papers except one describe the most recent work available at the time of the symposium; judging from the references cited, the work described in the remaining paper was carried out fully 10 years or more before the first symposium of this series.

The somewhat nebulous nature of quality in foods does to some extent preclude its discussion in scientific terms. As Sawyer points out, the assessment of quality by analytical means is a largely ephemeral concept. The performance of some test and the comparison of the result obtained, which itself is likely to vary between laboratories, with a predefined standard, yields information which is of value to the manufacturer or control authority, but of little direct significance to the consumer. Quality is ultimately determined by the consumer, and the acceptance of standards of quality in a particular item is manifested by the consumer's willingness repeatedly to purchase that item. The manufacturer and retailer, while operating within the normal constraints of legislation and sound practice, should be happy to rely on the consumer's unbiased judgment of quality. Professor Hawthorn's keynote address touches on the increased subjection of the consumer to the not wholly impartial and frequently misinformed pronouncements of those who attempt to impose their own views of quality in foods. The industry must of course continue, through more efficient control of quality, to improve its products and its service since this is the surest way to retain the confidence of the consumer; however, it cannot afford to neglect the often specious claims of those who believe they have all the answers. It is perhaps a pity that this important influence on the perception of quality could not be explored further.

The presentation of the contents of this volume is identical to that of earlier volumes in the series. The papers are well laid out, and the tables and figures are very clear and easy to follow. This reader found only one serious error (the end determination of cellulose hydrolysis products as sucrose) in what is a collection of very readable papers. The high price of £35 will probably result in the main purchasers being libraries rather than individuals, but since it is a book intended primarily as a source of reference this is to be expected. The book contains much that will be appreciated by those working in the industry and also students seeking to broaden their knowledge of food quality and food analysis.

*P. A. Burns*

**Carbohydrate Metabolism in Plants.** By C. M. Duffus and J. H. Duffus. London: Longman, 1984. Pp. xii + 183. ISBN 0 582 44642 2. £7.50.

The stated objective of this book is to integrate the physiological processes with biochemical events in carbohydrate metabolism in higher plants. The authors must be satisfied with their efforts since this objective has been admirably achieved. The introductory chapters discuss cell structure, location and function. This is followed by chapters discussing the various metabolic pathways. An individual chapter is devoted to metabolism of sucrose, one of our most common sweeteners. The following chapters discuss polysaccharide synthesis, including starch and cell wall biosynthesis, and the biosynthesis of complex carbohydrates such as glycoproteins and glycolipids. The following chapter on polysaccharide degradation describes the various enzymic systems involved in hydrolysis. Secondary products are described next, including, for example, the cyclitols, gums and glycosides. The book concludes with chapters on the regulation of carbohydrate metabolism and the techniques of carbohyhdrate analysis. The final chapter on analytical techniques is mainly devoted to wet methods of analysis. I would have liked to have seen more emphasis placed on physical methods—e.g. carbon nuclear magnetic resonance and hard sphere calculation. Furthermore, a section on the analysis of secondary products would not have been out of place since an earlier chapter was devoted to a description of these compounds. Nonetheless an interesting overview is presented.

This is an introductory text and much of the information in the book can be obtained from other sources, yet its layout, clarity of presentation and easy-to-read style will make it popular with students in this area. Each chapter includes a list of the most important references which may be followed up for an in-depth study into a particular topic. Many helpful diagrams and figures are included in the book.

I thoroughly enjoyed reading this book and, at its reasonable price, have no hesitation in recommending it to all students working in this field.

*S. Z. Dziedzic*

**Developments in Food Colours,** Vol. 2. Ed. by John Walford. Barking, Essex: Elsevier Applied Science, 1984. Pp. x + 257. ISBN 0 85334 244 X. £28.00.

The publication consists of seven chapters by selected authors supplementing the nine in Vol. 1 (published 1980), the two books now affording a wide coverage of the subject. Together these books not only provide up-to-date general information on the composition, sources and production of food colours, but also put their usage as food additives into perspective by reviewing regulatory control and safety in use. A greater weight of food colours is consumed in the U.S.A. than in any other single country and its method of regulatory control is different from most. The editor has, therefore, quite rightly invited American experts to write three of the chapters in Vol. 2. These are entitled: 'Influence of colour on sensory perception and food choices', 'Synthetic certified food colours of the U.S.A.' and 'Future trends'. The other chapters deal with regulatory approaches, analysis of synthetic food colours, natural colours and adverse reactions. The book should be of considerable interest to those involved with food colours, whether they are users, legislators, or manufacturers, and also to anyone wishing to gain general knowledge in the field.

The editor's stated aim is to discuss technical advances in food colours since the appearance of Vol. 1, and to examine the progress made in understanding the need for regulating the use of and controlling the quality of colours permitted for use in foodstuffs.

However, the book is not based entirely on recent trends so the contents could be described as an addition to the overview provided by Vol. 1. The individual subjects chosen are well balanced with only a slight overlap in the area of U.S. regulations.

In the chapter on 'Influence of colour on sensory perception and food choices', the auhor illustrates the importance of colour but also succeeds in showing how elusive its role is and how difficult it is to quantify. Of the five chapters dealing with more objective aspects of food colours, the one entitled 'Natural colours in foods' covers the extensive research carried out on the extraction of colours from plants and the possibility of improving their stability by further chemical reaction or by other means. Despite the lack of success judged in commercial terms, the author feels it is an area worthy of still further investment. The chapter on analysis provides a large amount of useful detailed information and rightly points out the considerable advantages of instrumental methods. In the last chapter entitled 'Future trends' F. J. Francis of the University of Massachusetts predicts a continuance of the trend towards the use of natural colorants at the expense of the synthetics, but with the latter retaining an important share of the market. A similar prediction based on somewhat different reasons was made by the editor, John Walford, in the closing chapter of Vol. 1.

The book is similar in general appearance and design to Vol. 1. It is well presented. the layout is good and no typographical errors were noted. Each chapter is adequately supported by references, many of them recent ones.

*A. Pearce*

**Manual for Social Surveys on Food Habits and Consumption in Developing Countries.** By Adel P. den Hartog and Wija A. van Staveren.
Wageningen: Pudoc, 1983. Pp. viii + 114. ISBN 90 220 0838 X. Dfl.10.00.

According to the authors, this book is intended as a guide for those with either a practical or an academic training in nutrition or home economics and who are involved in field work in food and nutrition programmes, such as nutrition education. supplementary feeding for vulnerable groups, school feeding or applied nutrition programmes in the developing world. It is not intended as an introduction to social surveys and epidemiological studies, but to provide practical information on methods of collecting survey data on food habits and food consumption. Chapter headings are: 'Food habits and food consumption patterns'; 'Food habits and ecology'; 'Orientation from subsistence farming to cash-crop farming'; 'Influence of towns on food habits and urbanization'; 'Food distribution in the household and infant feeding'; 'Dynamics of food habits'; 'Food and nutrition policy'; 'Notes on field studies'; 'Measurement of food consumption'; 'Conversion of amounts of foods into nutrients'; and 'Reporting data'. Some are chapters of no more than a few pages. There are also appendices containing suggestions for data collection and questionnaires for surveys of food habits, seven pages of up-to-date references (which include article titles). but no index.

For the most part, the book is written at a fairly basic level. For example, the

concept of a table is dealt with in considerable detail, along with a large number of examples of data tabulation. This may seem somewhat unnecessary to someone who has handled quantities of data but no doubt would be useful for field workers in many situations in the developing world. Another example of material dealt with at a basic level is the specimen calculation of nutrient intake from weighed food intake. However, despite considerable details of calculation methods, there is no indication of how to use values for nutrients so derived, in comparison with recommended daily allowance values, as this is stated to be beyond the scope of the book. Certainly anyone with a science degree should already have experience in handling data. Nevertheless, it would be a useful book for programme leaders to distribute to field workers undertaking survey work in developing countries.

*Ann F. Walker*

**Physical Properties of Foods.** Ed. by Ronald Jowitt, Felix Escher, Bengt Hallström, Hans F. Th. Meffert, Walter E.L. Spiess and Gilbert Vos.
London: Applied Science, 1983. Pp. xvii + 425. ISBN 0 85334 213 X. £46.00.

This book covers the proceedings of a seminar held under the auspices of COST (European Co-operation In Scientific and Technical Research) to mark the conclusion of the COST 90 project on physical properties of foods. The primary objectives were to present the results of 3 years' work on the project in the three following areas—water activity, rheology and thermal properties. Perhaps the choice of topics should have been reflected in the title. The seminar was attended by over 100 of the world's leading scientists, actively researching into these subjects and presents a very authorative overview of the current situation. A second project, COST 90 bis, will deal with electrical/optical, mechanical and diffusion properties.

A similar approach is taken in all three sections. The needs for the data and the practical applications are discussed. Theoretical and practical aspects are reviewed and available data are surveyed and critically assessed. In addition there is a collaborative trial within each subject area, whereby appropriate laboratories analyse the same materials and the findings are presented and discussed.

In the water activity section the use of a three parameter GAB sorption model to construct adsorption isotherms is described. A survey of existing data, including a table listing 234 products, for which sorption isotherms have been determined, is presented, although direct references are not given. The COST 90 project on water activity, to which forty laboratories contributed, is discussed. As a result it was possible to recommend: a suitable reference material (microcrystalline cellulose); a convenient, inexpensive test equipment; a 'most probable' sorption isotherm for the reference material; a procedure for using these to determine sorption isotherms for food materials and a format for recording and presentation of data. Based on these findings R. Duckworth evaluates the future needs in water sorption in foodstuffs.

The rheological section is concerned mainly with viscosity and flow characteristics of materials. A collective study, involving eight laboratories, is described. Aqueous solutions of carrageenan, guar, karaya, xanthan gums and sucrose, together with apple sauce, chocolate, cooking oil and mineral oils, were characterized over a wide range of shear rates using different rotational viscometers. The individual results are collated in thirty pages of appendices. On the whole, differences between measurements made by individual laboratories were greater than the scatter observed by a single laboratory

using any one viscometer. For sample fluids this was attributed to differences in the absolute calibration of the instruments; for more complex fluids this was further complicated by the shearing history before measurement. The need for accurate temperature control was also emphasized. Guidelines for measurement of data and a further discussion on the collection and use of such data for Newtonian and non-Newtonian fluids follow, based on experience gained from this collaborative study. Finally the flow characteristics of disperse systems are reviewed.

The thermal properties section deals with specific heat, enthalpy, thermal conductivity and diffusivity, heat penetration, initial freezing point, freezing range and unfreezable water. The main thrust is toward relating these thermal properties to the chemical composition of the food, and to other properties such as temperature and density. Many of the published equations are reviewed, and for some examples results calculated from these equations are compared with experimental values. The results of a limited collaborative trial on thermal diffusivity measurement are discussed. Some of the more common methods for measuring thermal properties are also described. H. F. Th. Meffert introduces an interesting section on error transfer. This shows how errors in thermal property values are translated to errors in calculated values of freezing and thawing times or energy requirements. A summary is given by J. D. Mellor, who concludes that future developments should concentrate on improving probes, developing a data bank and retrieval system, developing more accurate equations for estimating thermal properties, and extending the data of higher temperatures. There is some overlap between the main chapters in this section, but it is difficult to see how this could be avoided.

The book is well presented and indexed, with relatively few typographical errors. It is interesting to note that, occasionally, some data are quoted to five significant figures, despite the many comments about the absolute accuracy of experimental values. Most chapters conclude with a discussion by the participants; generally these are of a high standard and points often glossed over in the chapter are given a second airing.

To date, the original objectives of COST 90, of making available physical property data for the Food Industry, have not been fully realized. However this book offers useful advice for determining and estimating selected physical properties, and is a very welcome addition in an area which has received relatively little attention in the past. It would be very useful for final year undergraduates, research students, and scientists and engineers working in research or development.

*M. J. Lewis*

## Books received

**ARC Food Research Institute Biennial Report, 1981–1982.**
Norwich: Food Research Institute, 1983. Pp. 144. ISBN 0 7084 0287 9. £3.50.

In addition to the divisional reports and research area reports, there are five special reports on: glycoalkaloids in potatoes; chemistry of plant cell walls and dietary fibre; the analysis of folates; autoxidation of lipids and its control; and some major causes of taints in foods. The eight research areas reported on are: chemical toxicants in food; food poisoning and toxigenic micro-organisms; nutrition and food quality; microbial spoilage of foods and handling hygiene; basic chemical, biochemical, physical and biological

aspects of food; basic physiological, compositional and taxonomic aspects of micro-organisms; raw materials, byproducts and waste utilization; and processing, storage and distribution of food.

**Agricultural Research Council Annual Report, 1982–1983.**
London: Agricultural and Food Research Council, 1983. Pp. v + 119.
ISBN 0 7084 0273 9. £4.00.

Research work briefly reported includes aspects of: plant physiology; plant protection; plant breeding; animal breeding and genetics; animal physiology; animal diseases; animal nutrition; and engineering. Food research briefly reported concerns: ELISA assays of food constituents and contaminants; NMR spectroscopy; glucosinolates and bitterness in Brussels sprouts; meat species identification; determination of body composition of farm livestock by ultrasonic transmission; predictive model of toxin production by *Clostridium botulinum*: and conservation of raw milk.

**Buying Problems: Consumers, Unsatisfactory Goods and the Law. Report by the National Consumer Council.**
London: National Consumer Council, 1984. Pp. iii + 124. ISBN 0 905653 85 8.

Chapter headings comprise: Setting the scene; The nature of the problems; The search for redress; What do consumers want?; The framework of reform; Merchantable quality: a phrase not fit for its purpose; Minor defects; Durability: desired, but undefined; Remedies; Losing the right to reject; Damages and suspended payments; Privity of contract; Manufacturers and données; Appendix: faulty and unsatisfactory goods, a survey of consumers' complaints.

**Composition of New Zealand Foods. I. Characteristic Fruits and Vegetables.**
By F. R. Visser and J. K. Burrows.
(DSIR Bulletin 235). Wellington, New Zealand: Department of Scientific and Industrial Research, 1983. Pp. iii + 36. ISBN 0 477 06728 X.

The food plants dealt with in this booklet are: feijoa (*Acca sellowiana*); tamarillo (*Cyphomandra betacea*); kiwifruit (*Actinidia chinensis*); grapefruit (*Citrus* hybrid); watercress (*Nasturtium microphyllum* and *N. officinale*); puha (*Sonchus oleraceus*); silverbeet (*Beta vulgaris* var. *cicla*); kumara (*Ipomea batatas*); pumpkin (*Cucurbita maxima*) and squash (*C. moschata*).

**The Clinical Research Process in the Pharmaceutical Industry.**
Ed. by Gary M. Matoren. (Drugs and Pharmaceutical Sciences Series, Volume 19)
New York: Marcel Dekker, 1984. Pp. xxi + 549. ISBN 0 8247 1914 X. SFr163.

The twenty-seven chapters include topics such as research planning; legal and ethical problems; clinical data management and statistical design; drug safety evaluation; postmarketing surveillance; drug regulatory affairs; and the role of the FDA.

**Report of the Moredun Research Institute, 1982–83.**
Edinburgh: Moredun Institute (Animal Diseases Research Association), 1984. Pp. 83.

**Biennial Report of the Meat Research Institute, 1981–83.**
Bristol: AFRC Meat Research Institute, 1983. Pp. 148. ISBN 0 7084 0266 6. £5.00.

The research projects reported in this volume are in the following areas: mechanisms controlling growth and body composition and their manipulation; production attributes of meat animals and their modification in practice; pre-slaughter handling and stunning of animals; measurement and prediction of carcass composition and value; refrigeration and process engineering; physical measurements of meat composition; handling, processing and packaging technology; hygienic slaughter; spoilage biochemistry and bacteriology; control of microbial growth; texture, structure and eating quality of meat; structure of muscle and meat; connective tissue; biochemistry and physiology of meat fats; flavour and off-flavour investigations; meat species identification; and biochemistry of anabolic agents.

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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 | $\mu$ = $10^{-6}$ m | minute | min |
| nanometre | nm = $10^{-9}$ m | second | sec |
| litre | l = $10^{-3}$ $m^3$ | | |

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