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Quality in frozen cod and limiting factors on its shelf life

T. R. KELLY

Summary. Organoleptic assessments of toughness, dryness and overall texture and flavour acceptability have been made on cod fillets stored at −7°C. Inter-relationships between these parameters, muscle pH and time have been derived, which have shown that in high pH cod, flavour development during cold storage limits the shelf life, while low pH fish become unacceptably tough before the flavour becomes acceptable.

Introduction

The shelf life of fish, in common with other foodstuffs, depends on how quickly its 'quality' changes with time. 'Quality' in fish, as far as the consumer is concerned, is a rather ill-defined term which includes contributions from the quality attributes flavour, odour, appearance and texture. The shelf life is determined by the time taken for any of these attributes to reach a level which is unacceptable to the consumer.

It is, therefore, important to understand how and why these quality attributes change with time under any given set of conditions and to establish which of them limits shelf life. Fresh and frozen fish present different problems in this respect. Fresh fish caught and handled in the conventional manner, by storage on ice, have a shelf life which is ultimately limited by the production of spoilage flavours and odours (Shewan *et al.*, 1953). The other important attributes, texture and appearance, although they may certainly determine differences in quality, change little during ice storage and are seldom sufficiently bad to warrant rejection by the consumer on these grounds. When fish is frozen and cold stored, deteriorative changes occur which are quite different from those in wet fish. Flavours and odours characteristic of cold stored fish develop which are superimposed on the normal spoilage flavours, present at the time of freezing (Connell, 1967), and the eating texture becomes tougher and drier until eventually it too may become unacceptable. The colour of frozen fish, particularly those frozen at sea as fillets can present haeme type browning problems, whose cause and cure have been described (Kelly & Little, 1966; Kelly, 1967b).

It is the texture aspect of frozen fish deterioration which has demanded most attention in the past and considerable effort has been devoted to trying to understand the nature of the textural changes. In most of these investigations the emphasis has been on observing changes in some measured parameter, e.g. protein solubility (Love, 1962a),

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cell fragility (Love, 1962b) or free fatty acid formation (Olley & Lovern, 1960) with time under different storage conditions without necessarily relating these to taste panel results. Some recent work, however, has shown that the toughness of cold stored cod is not necessarily related to time or temperature in cold store, because muscle pH has an over-riding effect on toughness (Cowie & Little, 1966, 1967).

Because of this emphasis on textural changes, flavour changes in cold store have been rather neglected and one would perhaps conclude from this that flavour was not a limiting factor in the cold storage of fish (Bate-Smith, 1967). However, there is little or no experimental evidence available from which this conclusion can be drawn, since in most experimental work on frozen fish, the changes in texture and flavour have not been related to acceptability in consumer terms. Furthermore, since the effect of pH on texture has only recently been demonstrated, few attempts have been made to assess how the relationship changes with time (Connell, 1967; Kelly, 1967a) and what effect it has on storage life.

This paper describes a detailed study of the changes in texture and flavour acceptability, including the effects of pH, in cod stored at −7°C. From this information, conclusions regarding the relative importance of texture and flavour change in determining shelf life have been drawn.

Experimental

Material

Seventy-two cod (*Gadus morhua*) about 25 in. long were caught 10 miles off Aberdeen in mid-November 1966. The fish were landed within 12 hr of catching and stored on ice for a further 48 hr before filleting. The skinned fillets were placed in polythene pouches, frozen in a brine bath at −7°C and stored at this temperature.

Methods

Sampling technique. Twelve pairs of fillets were removed after 0, 2, 4, 6, 8 and 12 weeks storage. The anterior and middle portions of the fish were used for taste-panel assessment and pH measurement, respectively.

Taste-panel assessment. A frozen portion of the fillet was placed in a Pyrex dish and steamed for 45 min. The panel then scored each fish for flavour and texture acceptability on a 5-point scale, ranging from 5 for excellent to 0 for very poor. Toughness and dryness assessments were made on a two-dimensional score sheet as previously described (Cowie & Little, 1966), with the modification that the tough/soft Y axis was enlarged by the introduction of half units. In this system the score of 2 is considered to be perfect, scores greater than 2 on the X and Y axes indicate that the fish is drier or tougher than perfect, respectively, while scores less than 2 indicate that fish are wetter and softer than perfect, respectively.

pH measurement. Thirty grams of muscle were macerated with 60 g of water and the pH of the macerate measured at room temperature.

Results and discussion

The results of this investigation are shown in Table 1. Each value quoted is the average for two fillets. The relationship between toughness and dryness and overall texture acceptability can be extracted from these data by plotting toughness and dryness scores against texture acceptability scores. These relationships are shown in Figs 1 and 2, for toughness ($\bar{Y}$) and dryness ($\bar{X}$) respectively. The texture *versus* toughness plot (Fig. 1)

TABLE 1. Texture, flavour, toughness ($\bar{Y}$) and dryness ($\bar{X}$) assessments on cod fillets stored at −7°C

0 weeks	Fish No.											
	1	2	3	4	5	6	7	8	9	10	11	12
pH	6·28	6·43	6·68	6·72	6·87	6·93	6·50	6·60	6·47	6·80	6·80	6·47
Texture	2·9	3·2	3·8	3·6	3·2	3·0	3·2	3·8	3·5	3·2	3·1	3·7
Flavour	3·6	–*	–*	–*	3·8	3·7	3·9	4·1	4·0	3·8	3·9	4·0
$\bar{Y}$	2·9	2·6	2·2	2·2	1·3	1·4	2·7	2·1	2·6	1·5	1·4	2·3
$\bar{X}$	2·7	2·8	2·1	2·7	3·7	2·5	2·9	2·6	2·7	2·7	2·4	2·7
2 weeks	Fish No.											
	13	14	15	16	17	18	19	20	21	22	23	24
pH	6·71	6·71	6·76	6·56	6·59	6·80	6·74	6·65	6·78	6·79	6·34	6·65
Texture	3·6	3·9	3·8	3·5	3·0	4·1	3·9	3·5	3·6	3·7	2·6	3·3
Flavour	3·8	3·9	3·7	3·8	3·9	4·1	4·0	3·8	3·6	3·7	3·5	3·7
$\bar{Y}$	2·2	2·2	2·3	2·5	3·0	2·0	2·2	2·5	2·7	2·4	3·1	2·8
$\bar{X}$	2·5	2·6	2·9	2·7	2·8	2·4	2·9	2·9	3·0	2·7	3·1	2·6
4 weeks	Fish No.											
	25	26	27	28	29	30	31	32	33	34	35	36
pH	6·59	6·66	6·56	6·46	6·78	6·79	6·65	6·87	6·93	6·49	6·49	6·74
Texture	2·8	3·4	3·0	3·0	3·7	3·7	3·3	3·8	3·8	3·5	3·3	3·7
Flavour	3·2	3·3	3·3	3·4	3·6	3·7	3·5	3·7	3·7	3·7	3·6	3·7
$\bar{Y}$	3·2	2·7	2·9	3·0	2·4	2·4	2·5	2·2	2·0	2·5	2·6	2·4
$\bar{X}$	3·0	2·9	3·1	2·9	2·5	2·8	2·0	2·9	2·9	3·1	3·1	2·8
6 weeks	Fish No.											
	37	38	39	40	41	42	43	44	45	46	47	48
pH	6·62	6·75	6·50	6·58	6·84	6·65	6·96	6·95	6·93	6·71	6·43	6·52
Texture	3·2	3·6	2·5	3·1	3·6	3·3	3·5	3·6	3·5	3·2	2·9	2·7
Flavour	3·5	3·6	3·1	3·5	3·5	3·5	3·2	3·4	3·5	3·4	3·2	3·2
$\bar{Y}$	2·7	2·2	3·2	2·8	2·4	2·7	2·3	2·3	2·6	2·7	2·9	3·1
$\bar{X}$	3·1	2·9	3·1	3·1	2·9	3·1	2·9	2·8	2·8	2·8	2·9	2·8

* Imperfect seals allowed salt to penetrate sample, which masked flavour.

TABLE 1 (*continued*)

8 weeks	Fish No.											
	49	50	51	52	53	54	55	56	57	58	59	60
pH	6·51	5·63	6·75	6·61	6·74	6·73	6·60	6·35	6·51	6·56	6·81	6·39
Texture	2·9	2·6	3·5	3·0	3·0	3·4	2·5	1·9	2·4	2·7	3·5	2·4
Flavour	3·0	3·1	3·4	3·4	3·2	3·6	3·0	3·9	3·3	3·0	3·2	2·9
$\bar{Y}$	2·9	3·2	2·5	2·9	2·8	2·6	3·0	3·5	3·0	2·9	2·4	3·1
$\bar{X}$	2·9	3·0	2·8	2·8	2·9	2·8	3·4	3·5	3·3	3·0	2·7	3·4

12 weeks	Fish No.											
	61	62	63	64	65	66	67	68	69	70	71	72
pH	6·57	6·49	6·66	6·55	6·86	6·64	6·62	6·48	6·48	6·49	6·72	6·65
Texture	3·1	2·8	3·1	2·6	3·1	2·9	2·8	2·0	3·0	2·8	2·9	2·6
Flavour	3·3	3·0	2·8	2·7	2·8	2·8	2·6	2·5	2·3	2·9	2·9	2·5
Y	2·9	3·0	2·0	3·0	2·5	2·9	2·8	2·5	3·7	2·9	3·0	3·1
X	3·0	3·2	3·0	3·0	3·1	3·1	3·1	3·3	3·2	3·2	3·0	3·0

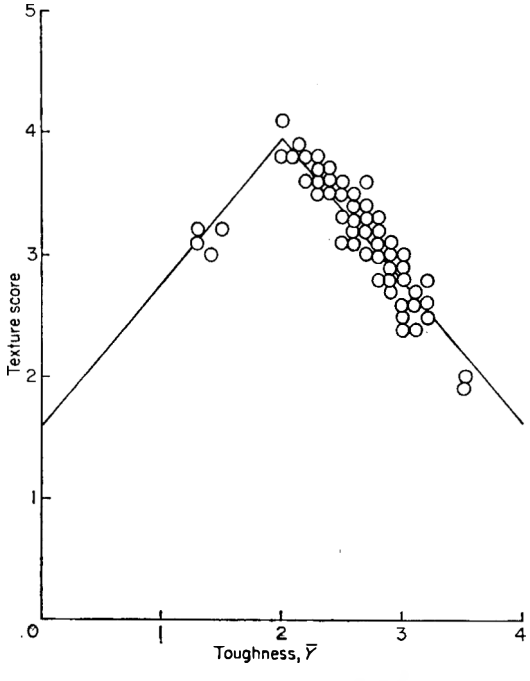

FIG. 1. Relationship between toughness ($\bar{Y}$) and texture score.

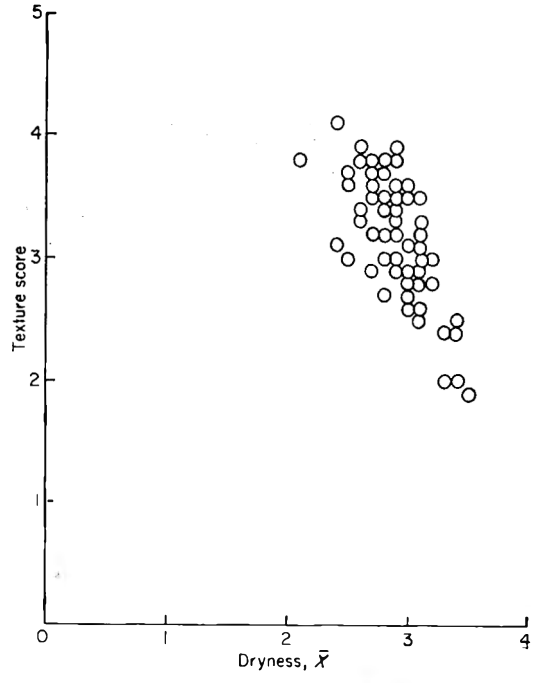

FIG. 2. Relationship between dryness ($\bar{X}$) and texture score.

produced a clear correlation in the form of an inverted V, the apex of which occurs at $Y = 2$ at a value of about 4 on the acceptability scale. This is the type of relationship we would have expected in view of the definition of the toughness scale. Ideally the maximum acceptability score should be 5 but the value of 4 obtained indicates the reluctance of taste-panels in general to give maximum scores. The relationship between texture and dryness is less clearly defined and there is a bigger spread of acceptability scores for a given value of $\bar{X}$. These relationships suggest that toughness is the major factor in determining texture acceptability.

The relationship between muscle pH and toughness and how it changes with storage time is shown in Fig. 3. Regression lines have been drawn through the points for each of the storage times. It is clear that the toughness at any given pH increases with time. Cold storage, therefore, superimposes an additional toughening effect on the original effects of pH. The nature of the toughening effects on both pH and cold storage are still unknown, but attempts have been made recently to describe the relative contributions of pH and protein solubility to the toughness of cooked cod muscle, in which it has been shown that pH is much more important than protein solubility in determining the toughness of cod stored at −29°C, and that it remains the dominant factor at higher temperatures (Cowie & Little, 1966, 1967).

It is possible to use Figs 1 and 3 to make predictions of the storage life of fish of

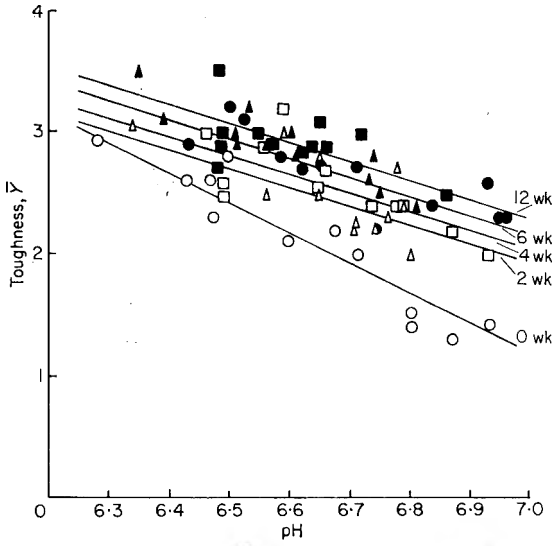

FIG. 3. Relationship between toughness ($\bar{Y}$) and muscle pH in cod fillets stored at −7°C. ○, 0 weeks storage; △, 2 weeks storage; □, 4 weeks storage; ●, 6 weeks storage; ▲, 8 weeks storage; ■, 12 weeks storage.

different pH held at −7°C. In Fig. 4, the texture acceptability score is shown plotted against pH for different storage times and it can be seen that the curve relating the

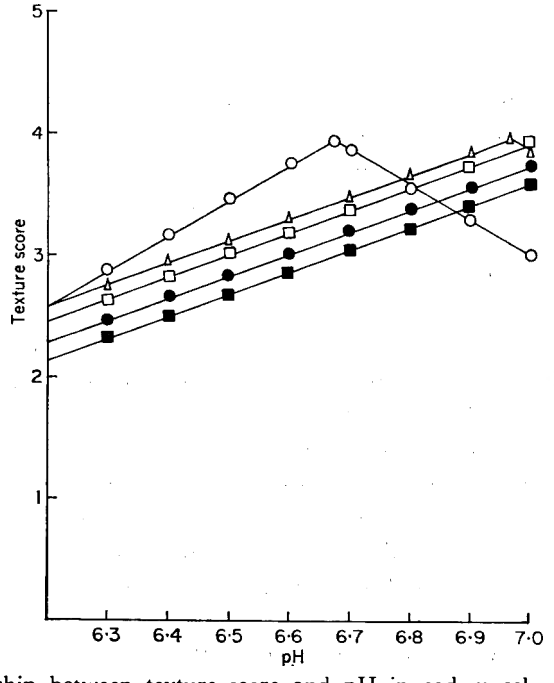

FIG. 4. Relationship between texture score and pH in cod muscle stored at −7°C. ○, 0 weeks storage; △, 2 weeks; □, 4 weeks; ●, 6 weeks; ▲, 12 weeks.

two has initially the form of an inverted V, which moves off to the right as the storage proceeds. If we use these curves to plot texture acceptabilities at various times for fish of various pH values we obtain the family of curves shown in Fig. 5. These show that fish of low pH deteriorate steadily with time, whereas the acceptability of fish whose pH is above 6·6 actually increases during the first few weeks of storage before passing through a maximum. If we set an arbitrary limit of texture acceptability at say 3, then we can read off the storage life of each pH group of fish at the intercept of the appropriate curve with the acceptability = 3 line. This would lead to the conclusion that the highest pH fish would remain acceptable for a long time, and this would be true on the basis of texture alone, without taking flavour deterioration into account. When the average flavour score at each of the storage times is plotted, we find that there is a steady deterioration in flavour throughout the period of storage which is independent of pH (Fig. 6). If we set an arbitrary limit of 3 on flavour acceptability we see that this is reached after 9 weeks of storage at −7°C.

From Figs 5 and 6, we can now specify the limiting storage times for fish of different pH on the basis of both flavour and texture, and this is shown in Fig. 7, which represents a kind of 'phase diagram' for the cold storage of cod at −7°C. All points within the shaded area describe pH and storage times which will give acceptable quality fish

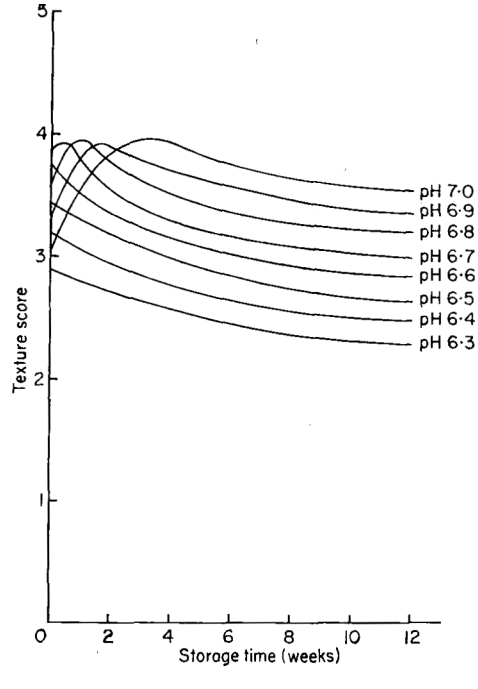

FIG. 5. Relationship between texture score and storage time for cod muscle of different pH.

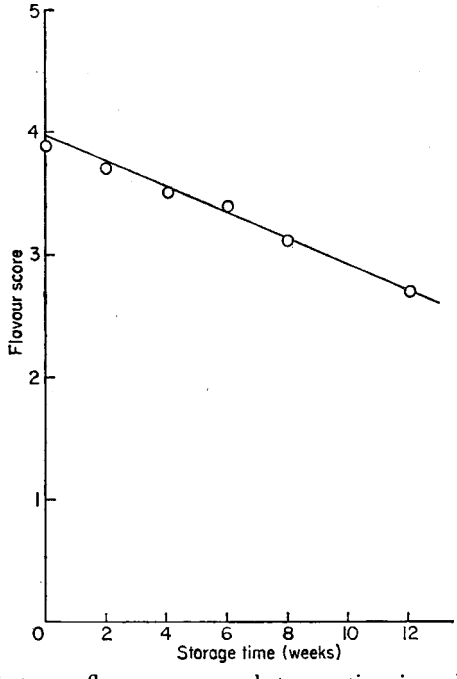

FIG. 6. Relationship between flavour score and storage time in cod fillets stored at −7°C.

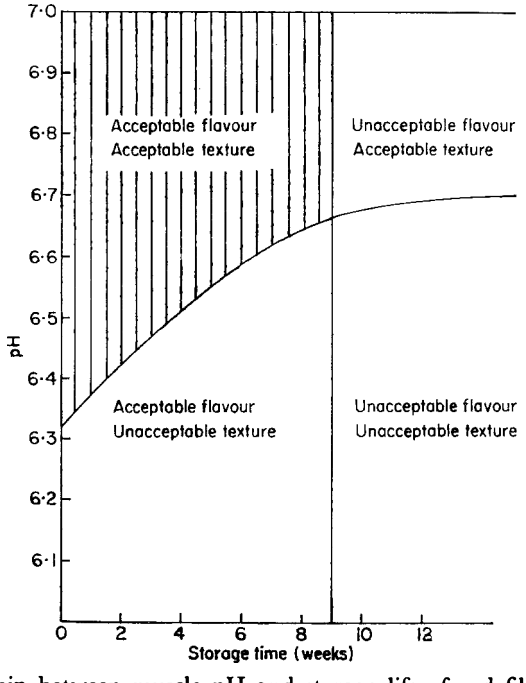

FIG. 7. Relationship between muscle pH and storage life of cod fillets held at −7°C.

both on the basis of texture and flavour. Thus, fish whose pH is between 6·30 and 6·66 will first become unacceptable because of texture change, while fish of pH greater than 6·66 will become unacceptable because of flavour change.

If diagrams such as Fig. 7 were constructed for different storage temperatures and different species they could be of use both to the fish processor and to the fish scientist. The processor could easily ascertain what would be the storage life of his fish under available cold storage conditions and organize distribution accordingly. For example, it is obvious that the low pH cod, should be handled more quickly than high pH cod, or stored at much lower temperatures, in view of their shorter shelf life. If fish could be graded according to pH before freezing this would be an additional benefit to the processor. To the fish scientist, diagrams of this type can be useful in pointing out areas in which research might more profitably be done. Most of the work which has been done on frozen cod has been concerned with texture deterioration. This present work has shown that, in high pH cod, flavour is more important. In view of the fact that most of the cod caught are of medium to high pH, we should perhaps turn more attention to the problem of cold storage flavour development in the future.

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The effect of low temperature freezing on quality changes in cold stored cod

T. R. KELLY AND J. S. DUNNETT

Summary. Cod fillets, frozen at temperatures between −7°C and −195°C have been stored at −7°C to establish whether 'deep' frozen fillets deteriorate faster than 'shallow' frozen fillets when both are stored at the same temperature. Neither objective nor organoleptic assessment of quality gave any evidence for such an effect and it was found that quality changes occurred at a rate which was independent of freezing temperature.

Introduction

When fish muscle is frozen, the tissue water crystallizes out as ice in increasing proportions as the temperature is lowered. The consequent dehydration of the tissue components and concentration of tissue salts, have given rise to the speculation that freezing may irreversibly damage the proteins and that the damage will be greater the lower the freezing temperature (Plank, 1925).

Evidence for the damaging effect of low temperatures was presented by Love & Elerian (1963) who found that in cod muscle frozen to −183°C and stored at −14°C, protein denaturation proceeded 50% faster than in equivalent samples of fish frozen and stored at −14°C. A similar effect has been shown to operate at higher temperatures (Love, 1966b, 1967a, b). When cod, initially frozen at −3°C and −30°C, were stored at −1·6°C, the 'deep' frozen samples deteriorated more rapidly than the 'shallow' frozen samples.

In the above investigations the 'cell fragility' method of Love & MacKay (1962) was used as the principal criterion of deteriorative change, although some evidence was also given to suggest that there was a corresponding difference in the rate at which toughness developed. Love suggested that this behaviour might be due to a hysteresis effect in the freeze–thaw cycle caused by the irreversible freezing out of bound water at low temperatures. He concluded that in deep frozen and rewarmed samples, the salt concentration of the aqueous phase must be considerably greater than in shallow frozen samples and that this was the cause of the increased rate of deterioration.

Piskarev & Kaminarskaya (1962) made histological measurements on frozen cod muscle and concluded that additional water freezes out from fish during cold storage at −10°C. They proposed that this was chemically bound water being set free

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to form ice and also showed that the effect was more pronounced in tissues which had been frozen initially to −195°C than in tissue frozen to −30°C before storage at −10°C.

These independent observations seem to support the idea that freezing may indeed irreversibly remove or loosen water from important sites of protein and hence provide more favourable conditions for the deteriorative reactions to occur. This is an interesting theory but evidence to show that there are actual differences in the rate of quality deterioration in deep and shallow frozen fish muscle, which could be related to these observations, is still lacking. This investigation was made to find if such evidence could be provided. Criteria other than cell fragility have been used to assess deterioration, including nitrogen extractability in 5% sodium chloride, liquor loss on thawing and organoleptic assessments of texture and flavour. An additional measurement, that of osmotic concentration of fish muscle juice has been made. This latter measurement was made to see if any changes could be detected which might be related to a decrease in the proportion of bound water as a result of freezing and cold storage.

Experimental

Material

The fish used in this experiement were codling, caught 10 miles off Aberdeen in mid November 1966. They were landed within 12 hr of catching and stored for a further 48 hr on ice before filleting. Skinned fillets from four groups of eighteen codling were sealed individually in polythene bags, alternate right and left fillets were taken from each of the seventy-two fish and frozen at −7°C in a brine bath held at this temperature. The remaining eighteen fillets in each of the groups were then frozen at −15°C, −30°C, −77°C and −195°C, by blast freezing at the former two temperatures and immersion in an acetone-CO_2 bath and liquid air bath at the latter two. In each case the fillets were held at the freezing temperature for 2 hr, the temperature being measured by a thermocouple placed in the centre of a spare fillet frozen under the required conditions. The deep frozen fillets were then placed in the −7°C bath and allowed to equilibrate at this temperature before being transferred to a deep freeze cabinet at −7°C ±1°C.

Methods

Sampling techniques

The fish were sampled after 0, 2, 4, 6 and 12 weeks storage. Three pairs of fillets from each group were removed at each time. The anterior portion of each fillet was used for taste panel assessment. Nitrogen estimations, pH measurements and histological sections were made on the centre portion and centrifugation and osmotic measurements were made on the tail portion.

Taste-panel assessment

The frozen anterior third of the fillet was steamed in a Pyrex dish for 45 min. The

panel then scored each fish for flavour and texture acceptability on a 5-point scale ranging from 5 for excellent to 0 for poor. Toughness and dryness assessments were made on a two dimensional score sheet as previously described (Cowie & Little, 1966). One modification of this system was that the tough/soft Y axis was effectively enlarged by the introduction of half units.

pH measurement

Thirty grams of muscle was macerated with 60 g of water and the pH of the macerate measured at room temperature.

Total nitrogen

The total nitrogen in three separate 1 g portions of each fillet was measured by a macro-Kjeldahl method.

Nitrogen extractability

About 1 g of muscle was weighed accurately into a 100 ml polythene measuring cylinder, 100 ml of cold 5% sodium chloride was added and the mixture macerated for 20 sec in an Ultra Turrax macerator, care being taken to prevent frothing. The macerate was then centrifuged at 17,000 ***g*** for 30 min and the nitrogen content of the supernatant was measured by a macro-Kjeldahl method. Duplicate samples were taken for nitrogen extractability determinations. Soluble nitrogen is expressed as a percentage of the total nitrogen present in the muscle.

Thaw loss

The tail portion of each fillet was cut into 0·5-in. wide strips and weighed into a polythene screw-topped centrifuge bottle. The bottles were stored for 24 hr at +1°C and then for a short while at room temperature to allow the strips to thaw completely. The bottles were then spun at 0°C in a refrigerated centrifuge for 50 min at 10,000 ***g***. The supernatant was weighed and expressed as a fraction of the total muscle weight.

Osmotic pressure measurements

The osmolality of the fluid centrifuged from each fillet was measured on a Fiske osmometer, calibrated with 300 and 500 milli-osmolal solutions of sodium chloride. Results are expressed in milli-osmoles solute per kilogram of muscle water.

Histology

Small pieces from the centre of each fillet were removed at −7°C and immediately placed in contact with solid carbon dioxide. They were then sectioned at 10 μ on a freezing microtome, fixed in 10% formal saline, stained with 1% eosine, dried and photographed.

Results

Change in nitrogen extractability, texture, flavour, toughness and dryness, thaw loss and osmotic pressure are shown in Figs 1–7, respectively. Each point represents the

average result for three fillets frozen at one temperature. These may be compared with the average results for all fillets frozen and stored at −7°C which are also shown.

During the 12 weeks of storage at −7°C the average nitrogen extractability fell from over 90% to 57%, a figure which agrees closely with the data of Cowie & Mackie (1968), who have investigated the Ultra Turrax method for the determination of extractable nitrogen in cod muscle. This indicates that denaturation of muscle proteins occurred to a considerable extent, and we would, therefore, expect any differences in rates of deterioration between 'deep' and 'shallow' frozen samples to show themselves in the present set of data.

The nitrogen extractability of the −7°C frozen controls and their deep frozen opposite numbers lie randomly on both sides of the mean values (Fig. 1). There is no indication that the deep frozen samples are consistently lower than the shallow frozen samples.

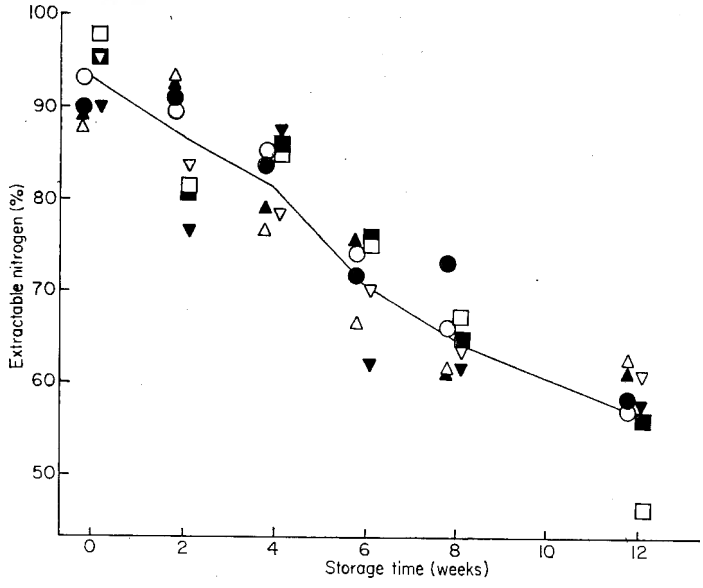

FIG. 1. Change in extractable nitrogen in cod fillets during storage at −7°C. ○, △, □, ▽, fillets frozen and stored at −7°C. ●, ▲, ■, ▼. corresponding fillets frozen at −15°C, −77°C, −30°C and −195°C. Continuous line drawn through mean values for fillets frozen and stored at −7°C.

No marked differences in texture were evident (Fig. 2) although there was a very slight preference for the deep frozen samples. The peculiar shape of the texture–time curve is characteristic for cod of relatively high pH and is dependent on the relationship which exists between toughness and pH (Kelly, 1969).

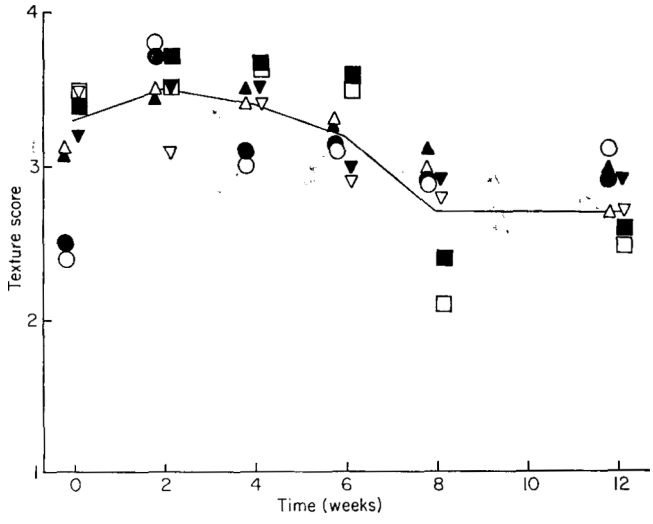

FIG. 2. Change in texture score in cod fillets during storage at —7°C. ○, △, □, ▽, fillets frozen and stored at −7°C. ●, ▲, ■, ▼, corresponding fillets frozen at −15°C, −77°C, −30°C and −195°C. Continuous line drawn through mean values for fillets frozen and stored at −7°C.

The results for flavour, toughness and dryness (Figs 3–5) confirm that no significant differences emerge between deep and shallow frozen fillets during cold storage, which can be detected organoleptically.

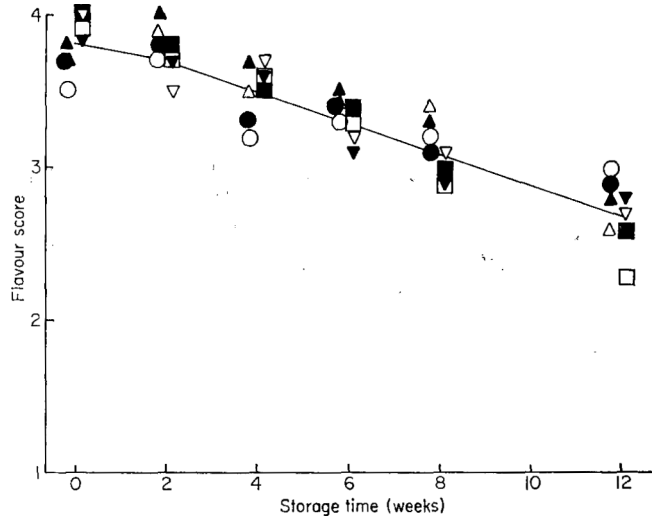

FIG. 3. Change in flavour score in cod fillets during storage at −7°C. ○, △, □, ▽, fillets frozen and stored at −7°C. ●, ▲, ■, ▼, corresponding fillets frozen at −15°C, −77°C, −30°C and −195°C. Continuous line drawn through mean values for fillets frozen and stored at −7°C.

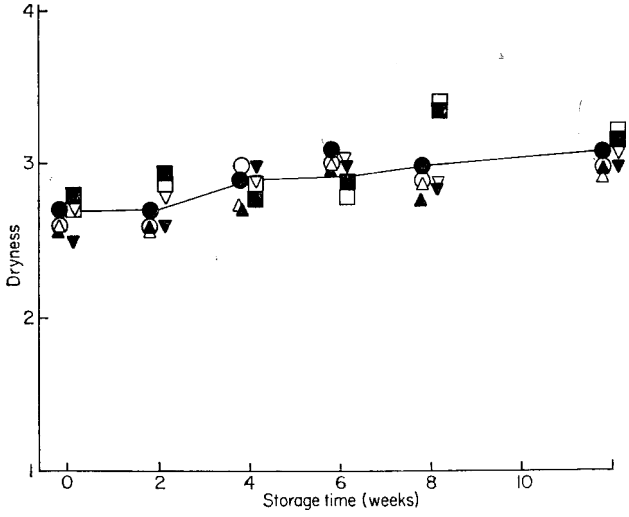

FIG. 4. Change in toughness in cod fillets during storage at −7°C. ○, △, □, ▽, fillets frozen and stored at −7°C. ●, ▲, ■, ▼, corresponding, fillets frozen at −15°C, −77°C, −30°C and −195°C. Continuous line drawn through mean values for fillets frozen and stored at −7°C.

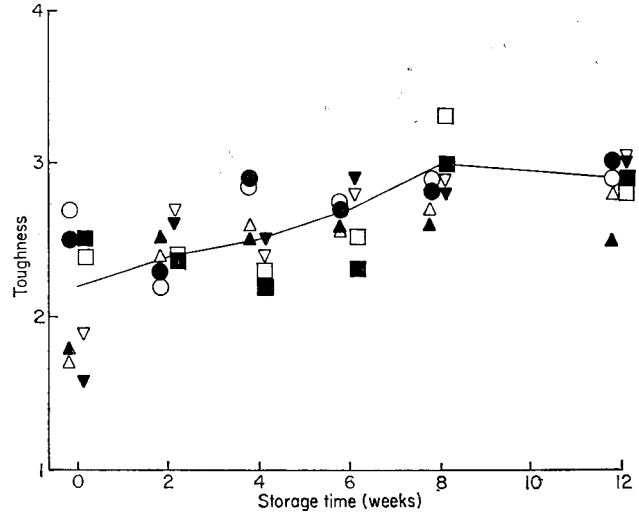

FIG. 5. Change in dryness in cod fillets during storage at −7°C. ○, △, □, ▽, fillets frozen and stored at −7°C. ●, ▲, ■, ▼, corresponding fillets frozen at −15°C, −77°C, −30°C and −195°C. Continuous line drawn through mean values for fillets frozen and stored at −7°C.

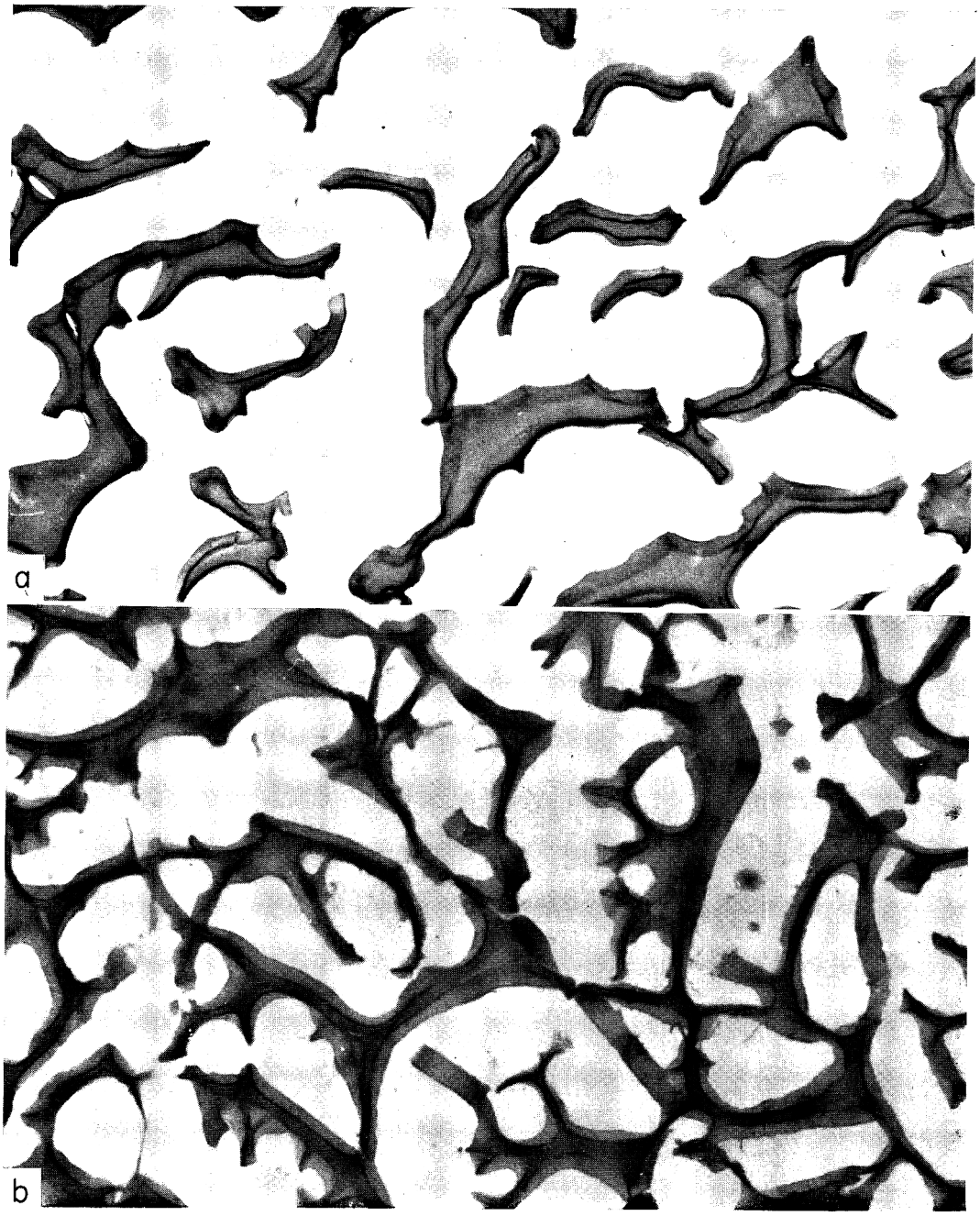

PLATE 3. Transverse sections of frozen cod muscle (× 250). (a) Frozen at −30°C and stored 0 weeks at −7°C. (b) Frozen at −30°C and stored 12 weeks at −7°C.

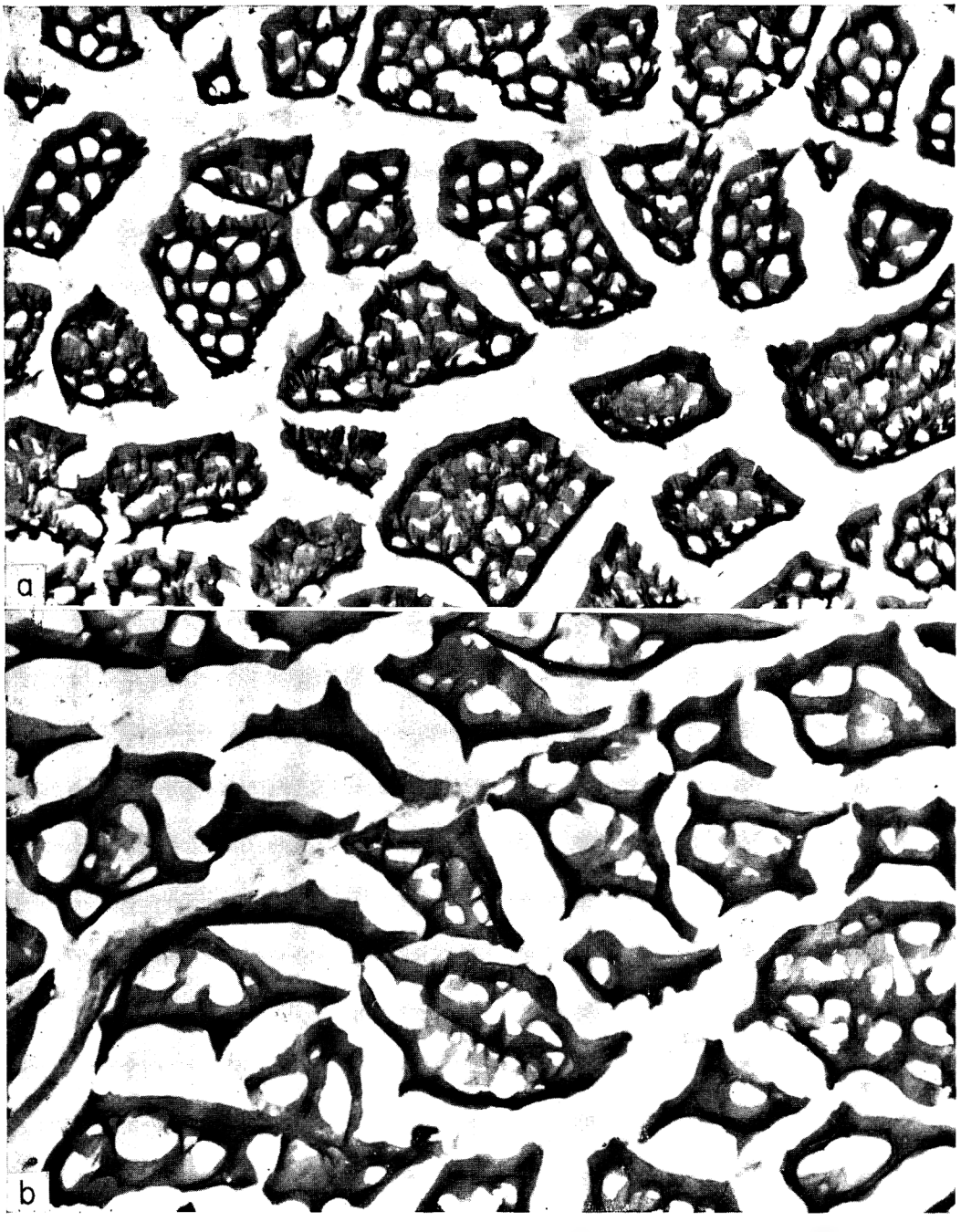

PLATE 4. Transverse sections of frozen cod muscle (× 250). (a) Frozen at −77°C and stored 0 weeks at −7°C. (b) Frozen at −77°C and stored 12 weeks at −7°C.

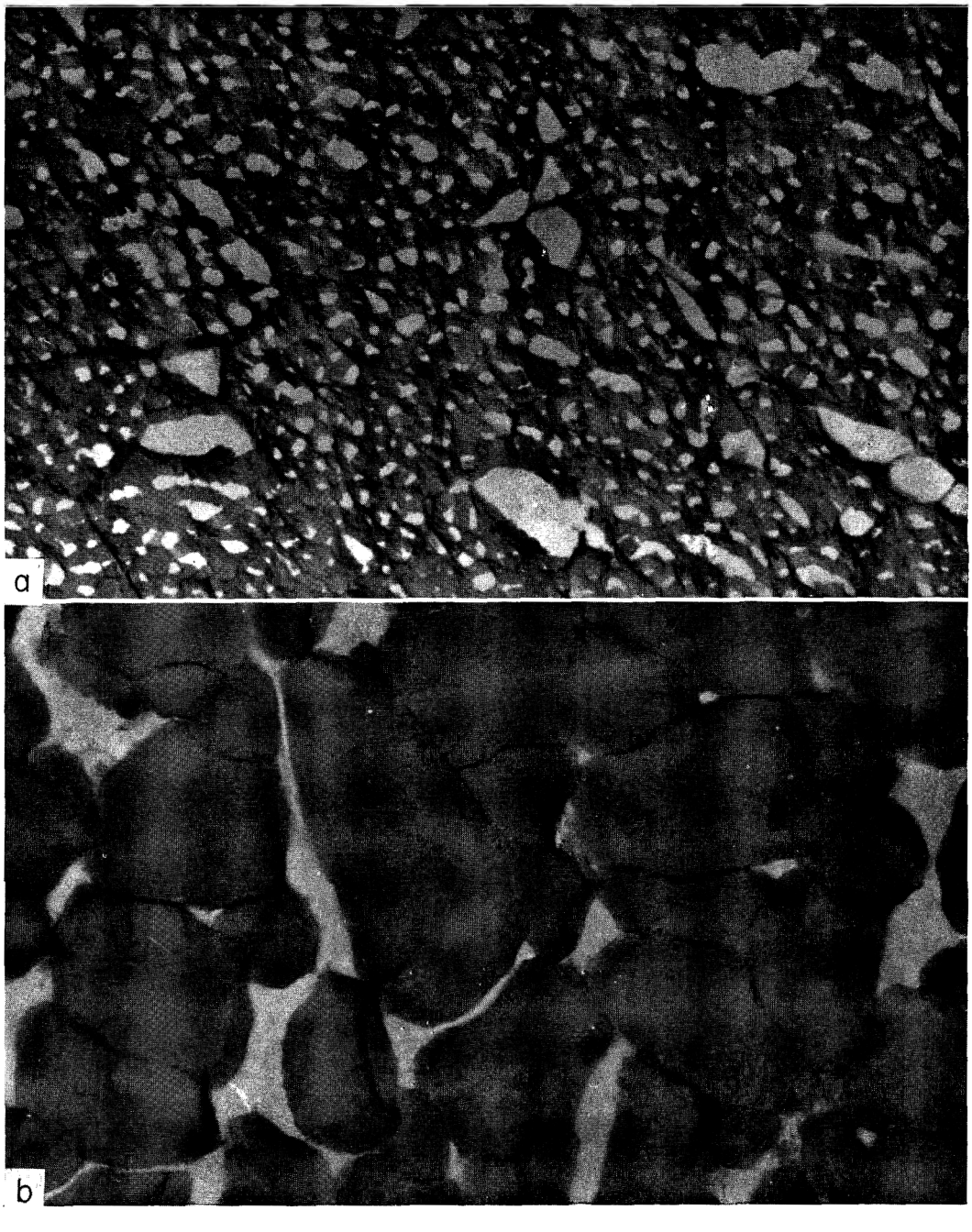

PLATE 5. Transverse sections of frozen cod muscle (×250). (b) Frozen at −195°C and stored 0 weeks at −7°C. (a) Frozen at −195°C and stored 12 weeks at −7°C.

PLATE 2. Transverse sections of frozen cod muscle (×250). (a) Frozen at −15°C and stored 0 weeks at −7°C. (b) Frozen at −15°C and stored 12 weeks at −7°C.

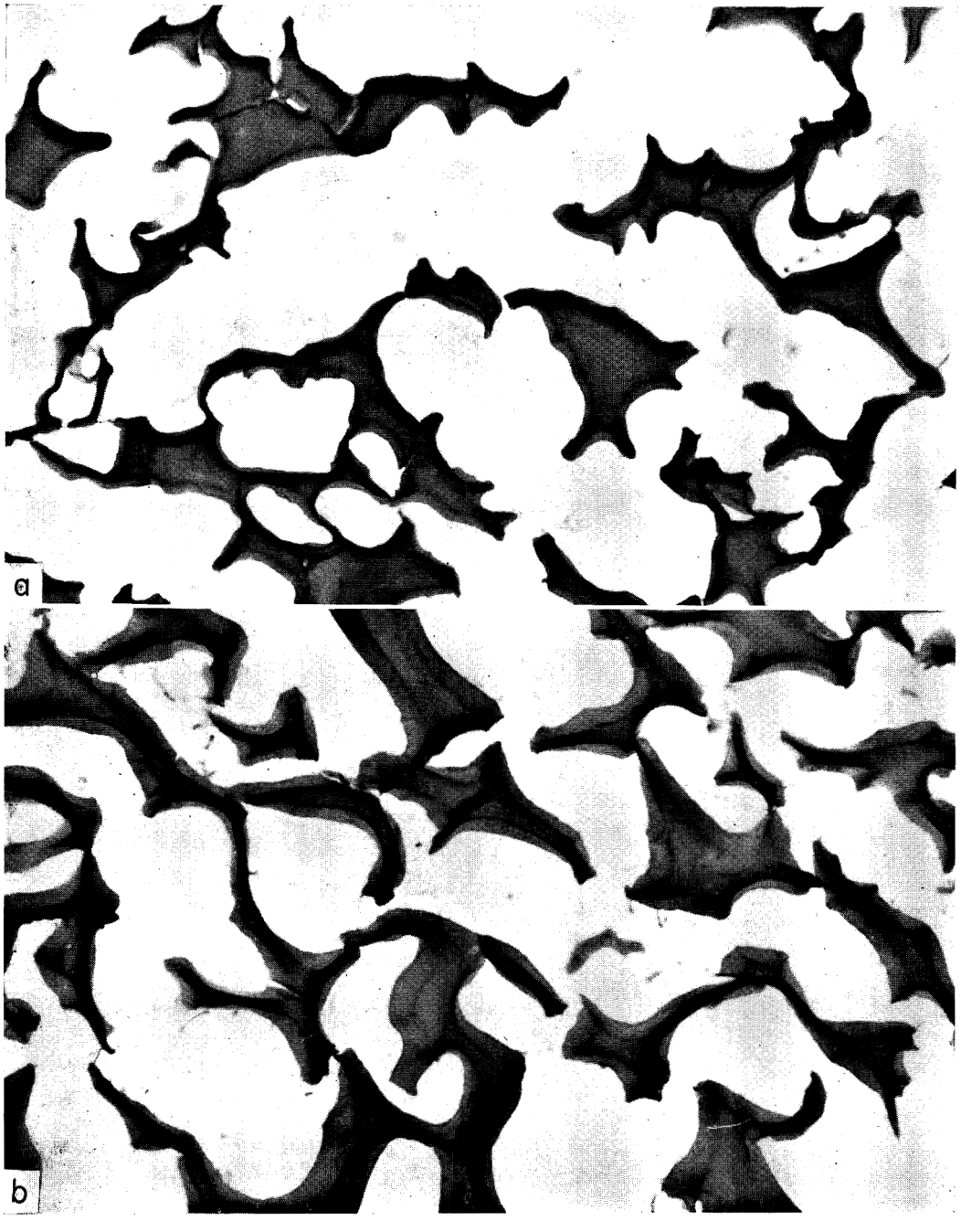

PLATE 1. Transverse sections of frozen cod muscle (× 250). (a) Frozen at −7°C and stored 0 weeks at −7°C. (b) Frozen at −7°C and stored 12 weeks at −7°C.

(*Facing p.* 110)

The osmolality of tissue fluids gave no indication of any irreversible release of bound water during deep freezing (Fig. 6). If this had happened the deep frozen samples might have been expected to give a slightly more dilute thaw liquor than the shallow frozen samples. However, there were wide deviations of up to 3% in both directions between fillets of the same pair. This is the order of magnitude of difference we might have expected to emerge during storage and the observed deviations would certainly mask any effects of irreversible freezing out of bound water.

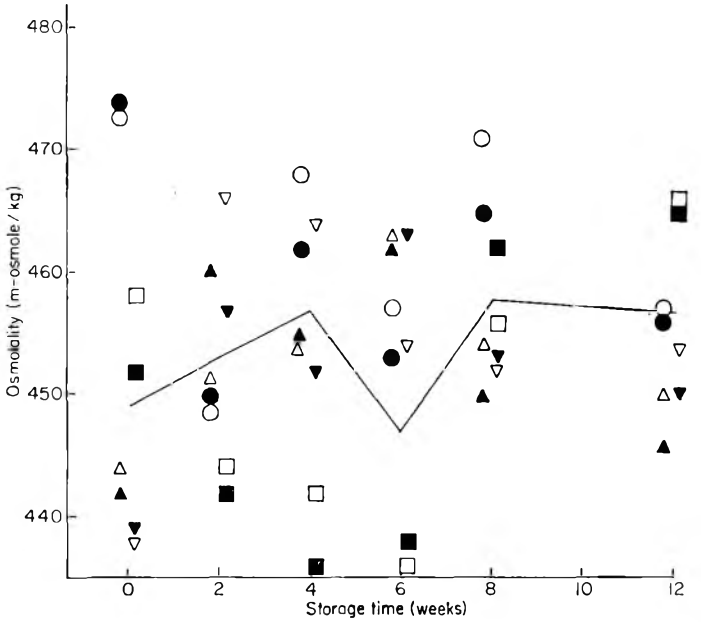

FIG. 6. Change in osmolality of thaw juice in cod fillets during storage at −7°C. ○, △, □, ▽, fillets frozen and stored at −7°C. ●, ▲, ■, ▼, corresponding fillets frozen at −15°C, −77°C, −30°C and −195°C. Continuous line drawn through mean values for fillets frozen and stored at −7°C.

When thaw losses are compared the initial freezing temperature was found to have a considerable influence (Fig. 7). The −77°C and −195°C frozen samples on the whole gave less drip than their −7°C frozen controls, while samples frozen at −15°C and −30°C gave more drip than the controls. The magnitude of these differences remained fairly constant throughout storage and there is no suggestion of any difference in rate of increase in thaw drip between deep and shallow frozen samples.

The histological observations are presented in Plates 1–5 which show sections cut from fillets frozen to −7°C, −15°C, −30°C, −77°C and −195°C, stored for zero time and 12 weeks at −7°C. These photographs show adequately the structure variations which can result from freezing at different temperatures. Initially, samples

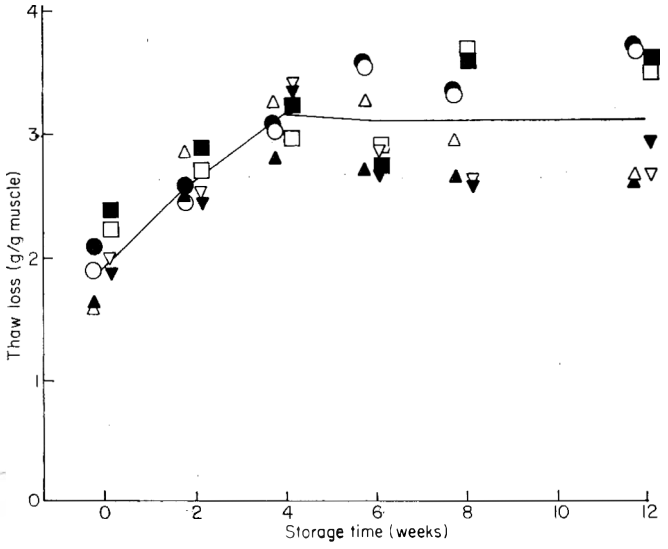

FIG. 7. Change in thaw loss in cod fillets during storage at −7°C. ○, △, □, ▽, fillets frozen and stored at −7°C. ●, ▲, ■, ▼, corresponding fillets frozen at −15°C, −77°C, −30°C and −195°C. Continuous line drawn through mean values for fillets frozen and stored at −7°C.

frozen at −7°C gave large clumps of solid matter and very large spaces which presumably originally contained ice, although sectioning obviously opened the structure still further. Samples frozen at −15°C gave rather smaller aggregates of solid material with large extracellular spaces. Samples frozen at −30°C gave even smaller aggregates with perhaps some indication of very large intracellular crystals. The samples frozen at −77°C gave a very characteristic pattern of individual cells which contained numbers of small ice crystals and freezing in liquid nitrogen produced no obvious ice crystals.

After 12 weeks storage at −7°C the samples frozen to −7°C, −15°C and −30°C had changed little in appearance. In those frozen to −77°C the average size of the crystals had increased and their number had decreased, but the overall appearance was still characteristic of that freezing temperature. In samples frozen in liquid nitrogen a number of small ice crystals had grown within each cell to give sections similar to those prepared by Piskarev & Kaminarskaya (1962).

These histological observations together with the organoleptic and other assessments confirm that neither the size nor site of ice crystal formation significantly affects the rate at which frozen fish muscle deteriorates.

As can be seen in Fig. 6, there is a large fish to fish variation in the osmolality of the thaw drip of cod muscle. When values for individual fish are plotted against the

corresponding muscle pH a clear relationship emerges (Fig. 8). The lower the pH the higher is the osmolality. This is presumably related to the greater content of lactic acid in tissue of low pH. There are no indications that this relationship changes with time of frozen storage, although it is conceivable that the osmotic pressure might decrease if more bound water was being released during cold storage. However, an effect such as this could easily be compensated by any autolyptic processes which continue to go on in the frozen muscle, with the production of low molecular weight material. The relationship between osmotic pressure and pH has important implications on investigations of water binding in biological tissue which involve measurement of ice formation curves and this aspect will be discussed below.

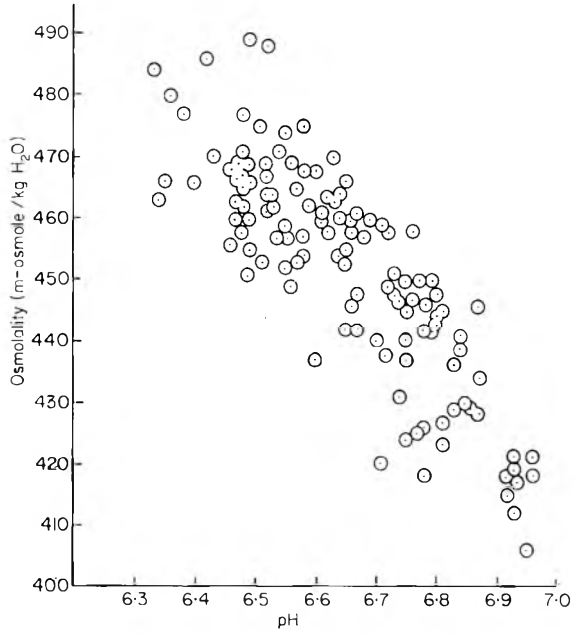

FIG. 8 Relationship between osmolality of thaw juice from cod fillets and pH.

Discussion

Love & Elerian (1963) claimed that in cod muscle frozen in liquid air and subsequently stored at −14°C the protein denatured 50% faster than in muscle frozen and stored at −14°C, when denaturation was measured by the cell fragility method, and it was suggested (Love, 1966) that denaturation rate differences of this order would be quite easily detected by a taste panel. The present investigation has not shown an effect of this type at −7°C using other objective and organoleptic methods of quality assessment. It must be concluded from this that while low temperature freezing

may bring about some specific effects on storage behaviour of cod muscle which are detectable by the cell fragility method, these effects are not related to the commercially important deteriorative changes which occur. This would seem to limit the usefulness of the cell fragility method for determining the quality of frozen fish when the initial freezing temperature is not known.

The nature of these specific effects of low temperature freezing remains unknown but it is still possible that an explanation involving hysteresis in the freeze–thaw cycle may be the correct one. Experimental evidence in support of this is still lacking. Love & Elerian (1963) provided qualitative evidence for frozen cod and there are other references to the presence of 'considerable hysteresis' in the percentage of ice in the freeze–thaw cycle in biological tissue (Meryman, 1957; Williams & Meryman, 1965) neither of which is supported by quantitative evidence.

The suggestion that in 'deep' frozen and rewarmed samples the salt concentration will be considerably greater than in 'shallow' frozen samples at the same temperature, cannot be correct, because the aqueous phase of partially frozen muscle is in equilibrium with ice. It follows from this that the osmotic concentration of the aqueous phase in frozen fish at a given temperature is dependent only on temperature and quite independent of freezing history.

The osmotic pressure measurements in this work have shown that the osmolality in the tissue fluid in cod muscle can vary quite widely, depending on the pH of the muscle. Since the osmotic concentration of the aqueous phase in muscle largely determines the amount of ice formed at any given temperature, it is not sufficient simply to compare fractions of water converted to ice for different samples of tissue and relate this directly to water binding. For example we would predict from the present results that, in general, high pH fish would have a higher proportion of water frozen than low pH fish at a given temperature due to osmotic differences. Any differences in water binding by the proteins would be superimposed on these effects. Absolute values of ice formation at given temperatures are, therefore, meaningless in terms of water binding unless osmotic pressure data are also available. This would be particularly important in any studies of hysteresis in the freeze–thaw cycle where spurious results could easily be produced by small changes in concentration of osmotically active species during freezing or cold storage.

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An improved method of adjusting flour moisture in studies on lipid binding

R. J. DAVIES,* N. W. R. DANIELS* AND R. N. GREENSHIELDS†

Summary. A method is described for the controlled adjustment of flour moisture level over the range 14–60%. Water was added as powdered ice to flour dispersed in liquid nitrogen thereby avoiding the introduction of mechanical work. The effect of moisture on the distribution of free and bound lipids in a hard wheat flour was investigated using this technique. Free lipid decreased above 20% moisture, while extractable bound lipid increased only above 25% moisture. These different critical moisture levels are considered with reference to the distribution of neutral and polar lipid fractions in the moistened flour.

Introduction

Changes in the relative proportions of free and bound lipids occur during the mixing of wheat flour doughs (Baldwin *et al.*, 1963; Ponte *et al.*, 1954, 1966; Chien-Mei & Pomeranz, 1966; Wootton, 1966) and have been shown to be influenced by the rate of dough mixing, by the total mechanical work input and by the atmosphere in the dough mixing chamber, (Daniels *et al.*, 1966, 1967).

The simple addition of water to flour has been shown to bring about the binding of flour lipids even in the absence of mechanical work (Olcott & Mecham, 1947). It was found that while increasing the flour moisture from 10% to 20% was without effect, ether extractable lipid fell from 70% of the total lipid to 39% at 30% moisture. Further addition of water caused no change in lipid binding provided dough development was avoided.

The object of the present investigation was to obtain a method that would allow a more precise control of flour moisture adjustment in the absence of mechanical work over a wider range than that used previously and to investigate lipid binding in detail in the critical area between 20% and 30% moisture.

Materials and development of method

The flour used in these experiments was an untreated unbleached commercial bread flour containing 14·0% moisture and, on a dry basis, 12·45% protein (nitrogen ×

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5·7), 0·5% ash and 1·65% total lipid. Light petroleum boiling between 40° and 60°C was redistilled before use. All other solvents were of analytical reagent quality and were used without further purification. Silicic acid, 100 mesh (Mallinckrodt Chemical Works, St Louis, U.S.A.), was freed of fines by sedimentation in methanol and dried at 105°C in a vacuum oven before use in the fractionation of neutral and polar lipid.

Attempts to raise the flour moisture content by equilibration over water in a closed vessel failed owing to mould growth on the damp flour. The addition of mould inhibitors (e.g. toluene, as used by Olcott & Mecham, 1947) was avoided in view of possible effects on lipid distribution. Moisture was more effectively raised by allowing the flour to fall repeatedly through a column of dense fog generated by passing steam over blocks of solid carbon dioxide. However, above 20% moisture the method failed owing to the caking of moist flour particles which prevented their even access to the fog. The addition of a measured amount of water in the form of a fine spray to a thin layer of flour also produced an unsatisfactory distribution of moisture. The greatest disadvantage of these methods was the lack of fine control over the moisture level that could be obtained. The unpredictable nature of the results of these techniques also produced a time lag since a moisture determination was necessary before proceeding further.

The addition of powdered ice to flour at sub-zero temperatures (Olcott & Mecham, 1947) appeared to offer a more precise control of moisture content but had the disadvantage that a long period of storage (1 month at −9·4°C) was required to allow the ice to sublime into the flour. Trial experiments showed that it was difficult to obtain a powdered ice fine enough for intimate dispersion in flour by grinding ice in a mortar cooled to −20°C. Invariably, even after prolonged storage below freezing point, when the mixture was brought to room temperature the moisture was found to be unevenly distributed in the flour, giving rise to damp clumps in surrounding dry flour.

It was noticed that when ice was ground at −20°C, the ice surface melted under the pressure of the pestle preventing the required breakdown to a fine powder. A considerable improvement in the fineness of grinding was obtained when ice was ground below the surface of liquid nitrogen (−196°C) added to the mortar. Moreover, the addition of the required weight of flour to the finely divided ice suspended in boiling liquid nitrogen produced a mixture in which the ice was intimately mixed with the flour before storage and equilibration at −20°C. The procedure finally used in this work for moisture adjustments in the range 14–60% was to measure the required amount of water into liquid nitrogen held in an iron mortar (a ceramic mortar was unable to withstand the extremely low temperatures used) and grind quickly to a finely divided powder. This was added to flour (100 g) similarly suspended in boiling liquid nitrogen in a beaker and the slurry transferred to a deep-freeze cabinet at −20°C to allow the nitrogen to boil off and the ice–flour mixture to reach equilibration by sublimation. It was found that even with the higher moisture levels an equilibrium was reached rapidly and the mixture was ready for use within 24 hr.

The effect of moisture level on lipid binding was studied by allowing the frozen

moisture to thaw at room temperature for at least 30 min before taking a sample for moisture determination by the A.A.C.C. vacuum oven method (Anon, 1962). The remaining damp flour was dried in the frozen state and the free and bound lipids quantitatively extracted.

The procedures followed were those adopted by Daniels *et al.* (1966). Free lipid was defined as that lipid removed by a 7-hr soxhlet extraction with light petroleum (b.p. 40–60°C). Bound lipid was defined as that remaining after the extraction of free lipid and removed by the solvent system recommended by Tsen, Levi & Hlynka (1962). The lipids obtained by these extractions were then quantitatively separated into neutral and polar fractions using a method based on that described by Parkes & Hummel (1965). The lipid material (100 mg) was dissolved in chloroform (50 ml) and shaken for 10 min with silica gel (2 g), filtered on a sintered glass funnel (Pyrex POR 4) and washed with chloroform (4×25 ml). The neutral lipid was that present in the solution. Polar lipid was then removed by washing the silica gel with methanol (5×25 ml). Solvents were removed by rotary evaporation and the proportions of neutral and polar lipid determined from the dry weight present in the two solutions. Thin layer chromatography was used to ensure that the method classified the lipid classes consistently. Plates were developed with either a solvent comprised of light petroleum (b.p. 40–60°C), ethyl ether and glacial acetic acid 60 : 40 : 1 v/v to separate the neutral lipids or chloroform methanol and water 80 : 25 : 2 v/v to separate the polar lipids.

Results and discussion

The method of moisture adjustment described proved most effective since a particular moisture level could be obtained to within 1% of that required with an even moisture distribution throughout the flour. This was verified both by repeated moisture determinations on the same sample and by the absence of small dough particles in the dry bulk of the flour which were seen when spray moistening of the flour was tried. The conditions of the technique eliminated completely any question of mechanical development of the wetted flour at all moisture levels and also avoided the possibility of mould contamination. The possibility that treatment with liquid nitrogen or freeze drying could have influenced the extractability of lipids was examined by subjecting a sample of flour to the routine described but with the omission of thawing before freeze drying. There was no change in free and bound distribution even though sufficient ice had been added to raise the moisture to 40% indicating that any change in free and bound lipid distribution was due to the increase in moisture level of the flour alone.

The effect of moisture on extractability of lipids was studied over the range 14–60%. Samples were prepared at intervals of less than 5% moisture throughout the range and at smaller intervals (about 1%) in the area of particular interest (18–35% moisture). The results of this study are shown in Fig. 1. Expressing all extracted lipid as a percentage of the total lipid extractable from flour at 14% moisture, the amount of free lipid

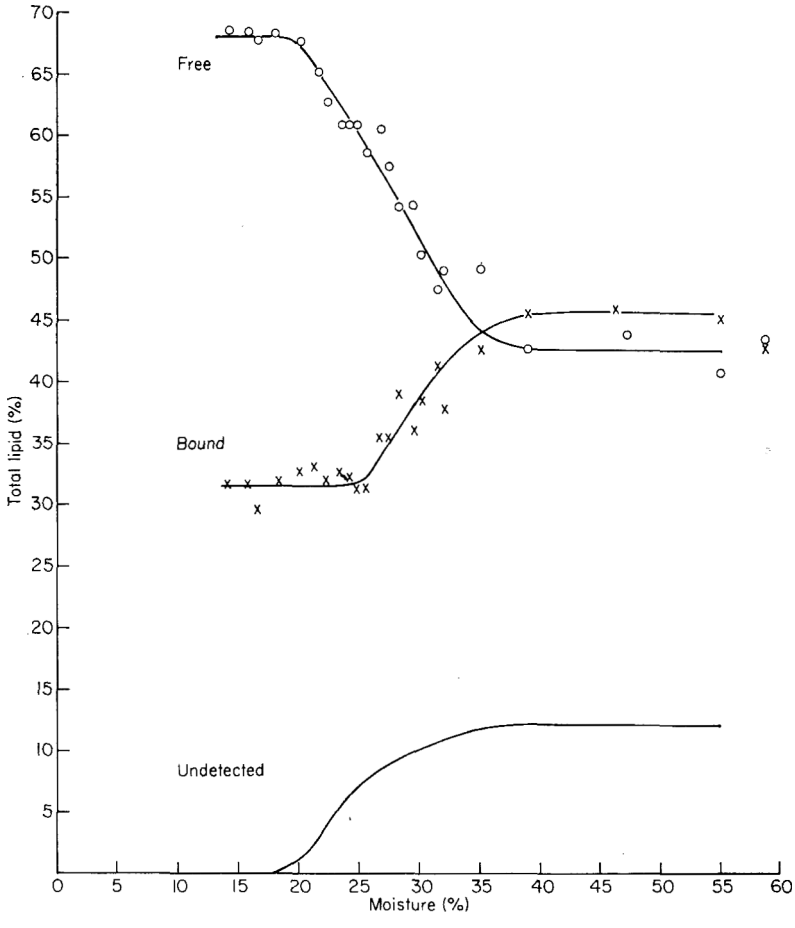

FIG. 1. Variation of lipid distribution with moisture level. ○, Free lipid; ×, Bound lipid; undetected calculated by difference. Standard errors of the plotted means; free ± 0·60%; bound ± 0·97%.

remained constant at 68% as the flour moisture was raised to 20%. Above 20% and up to 40% moisture the free lipid decreased steadily to give a constant value of 42·5% above 40% moisture. However, the amount of bound lipid at 14% flour moisture, 31·5%, remained constant until the moisture level rose to 25%. At this point bound lipid started to increase until it reached a maximum value of 45·5% at a moisture level of 40% and remained constant thereafter. These results were in agreement with those of Olcott & Mecham (1947) and further indicated that the increase in binding was a gradual process.

An interesting feature of the lipid binding results was the discrepancy between the loss of free lipid and the corresponding gain in bound lipid in the 20–25% moisture

range. It would appear that between 20 and 25% moisture a proportion of lipid material became undetectable even though subjected to the very efficient Tsen solvent extraction. A possible explanation may be that this lipid was bound in a very strong manner in a form which was resistant to solvent attack. Although this change commenced when 20% moisture was attained, further weaker binding, which increased the (extractable lipid) fraction referred to as 'bound', did not occur until 25% moisture was reached.

The fractionation of the free and bound lipids into neutral and polar gave a better insight into these changes, not only in this critical range but over the whole range. For instance it was evident that the initial strong binding in the undetected form involved polar lipid since the polar free lipid fell from 15·2 to 11·0% between 15 and 25% moisture without any increase in the extractable polar bound lipid. Unextractable lipid, some 12% of the total lipid at its maximum, remained fairly constant above 30% moisture although the proportion of neutral to polar lipid increased with increasing moisture. These results were in agreement with the findings of Olcott & Mecham who observed that loss of free lipid was matched by a loss of ether extractable phosphorus. They concluded that binding of phospholipids preferential to other constituents of flour lipids occurred during hydration of the flour although the initial stages of hydration were not studied in detail.

It is interesting that these quite significant changes in the lipid distribution pattern occurred only because the moisture level had increased. The possibility that these changes coincided with the presence of free water in the flour is suggested from the results of Toledo, Steinberg & Nelson (1968) which confirmed an earlier result from Vail & Bailey (1940). These workers, the former using nuclear magnetic resonance and the latter a freezing point depression technique, found that only above 25% moisture could liquid (free) water be detected in moistened flour. The amount of non-liquid (bound) water remained constant and independent of total moisture content of the flour above moisture levels of about 25%.

Across the range of moisture levels, the wet flour or dough that resulted when the flour–ice mixture was allowed to thaw showed interesting changes of physical form. Between 14 and 28% the flour changed from a free flowing to a sticky powder. The onset of this change occurred at a moisture level of about 20% and coincided with the onset of changes in lipid distribution (Fig. 1). Above 28%, towards the end of the changes in lipid distribution the moist flour exhibited for the first time a tendency towards a continuous structure. The material could hardly be called a dough yet over a period of some 2 hr the volume significantly decreased. This shrinking effect became more pronounced as the moisture level was further raised to the 50% level. At 50% moisture the dough appeared to have a wet surface after 1 hr at room temperature and had the consistency of a stiff batter after 2 hr.

Fig. 2 shows the appearance of the moistened flours over the range of moisture levels discussed above. The flour–ice mixtures were placed in open ended cylinders

(3·5 cm × 4·5 cm) and allowed to thaw for 2 hr at room temperature after which the cylinders were lifted off. The flour at 18% moisture collapsed. Between 20 and 28% the cylindrical form remained but did not shrink. Above 28% the volume decreased with increasing moisture so that at 45% moisture the volume was reduced to less than half the original.

Other workers in these laboratories (Webb *et al.*, 1969) concluded that a minimum moisture level of some 35% was required to hydrate an unworked flour of similar composition. This would suggest that a redistribution of lipids during the increase of moisture from 14 to 35% was necessary before dough formation was possible. The formation of a structure that contracted at this same moisture level (Fig. 2) was also

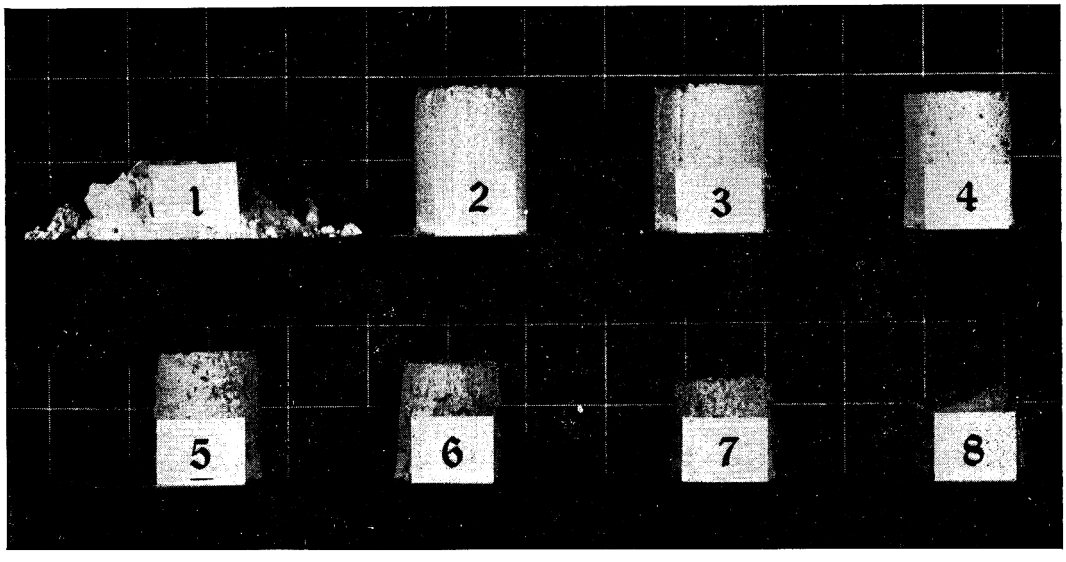

FIG. 2. Effect of moisture on the shrinkage of flour cylinders. Moisture levels; (1) 18%, (2) 23%, (3) 28%, (4) 30%, (5) 34%, (6) 36%, (7) 39%, (8) 45%

considered significant, particularly as Webb *et al.* (1969) found that a dough could be formed by the introduction of mechanical work to a flour at 35% moisture.

The intimate dispersion of finely divided ice in flour suspended in liquid nitrogen permitted the controlled adjustment of moisture level in flour without the introduction of mechanical work. This method further allowed the examination of changes in lipid distribution resulting from the effect of moisture at initial stages of dough formation. It was apparent that some interactions between flour components occurred at moisture levels at least 20% below those normally employed in breadmaking. Further work is in hand to elucidate the full significance of the pattern of events which occurs when water is added to flour both with and without the introduction of mechanical work and also to correlate these events with the presence of free water. It is proposed to ex-

tend this work to include soft wheat flour and so obtain further information regarding the nature of the interactions between different components of flour during dough formation.

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Variations in the composition of the flora on a Wiltshire cured bacon side

G. A. GARDNER AND J. PATTON

Summary. The number and types of micro-organisms on Wiltshire bacon sides after maturing at 4°C in both stacked and hanging positions were determined. Three sites on each bacon side were investigated: (i) singed rind, (ii) unsinged rind, and (iii) meat. On average the unsinged rind samples had a higher bacterial load than the singed rind. The meat samples had the lowest levels of contamination. There were, however, wide variations in counts on any one site both between sides from one factory, as well as factory differences.

The bacteria most frequently isolated from all sites were micrococci. The meat samples differed from the rind samples by a marked increase in the proportions of Gram-negative bacteria such as *Acinetobacter* spp. and *Vibrio* spp. The composition of the flora of bacon from each of three factories was slightly different; there was no appreciable difference attributable to the methods of maturation.

Introduction

The microbial association of any food is dependent on numerous physical and chemical influences. These vary between and even within a single product. The physical, chemical and microbiological principles of Wiltshire bacon production have been reviewed by Gibbons (1953). The most numerous bacteria on sides of bacon are micrococci (e.g. Garrard & Lochhead, 1939; Ingram, 1952). Other organisms such as *Clostridium*, *Streptococcus*, Enterobacteriaceae and *Alcaligenes* are less frequently found (Ingram & Hobbs, 1954).

In the production of bacon the whole sides, after immersion in a curing brine, are matured either in a stack or on a rail for 7–14 days at *c.* 4°C. This period of maturation permits the drainage of excess brine from the side and also ensures the even distribution of curing salts.

The work reported in this paper was carried out to determine the numbers and types of bacteria on a bacon side at the end of the maturation period. Three areas of the side were examined: the singed rind, the unsinged rind, and the cut muscle surface. The latter two areas are those recognized by the factory personnel as the positions where bacterial slime production can be first noted.

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Methods and materials

Bacon sides

Bacon sides were obtained from three factories (X, Y and Z). The sides were matured in a stack for 9 and 8 days in factories X and Y, respectively. Those from factory Z were suspended from a rail for 7 days in the maturation cellar.

Samples

Using a metal template and sterile instruments, samples of 10 cm^2 from each of three areas were taken from each side. The areas sampled were: (i) the skin from the outside of the hind leg (singed rind), (ii) the skin from the fold underneath the foreleg (unsinged rind), and (iii) the cut surface of the *gracilis* muscle (meat).

Enumeration of bacteria

Samples for analysis were transported to the laboratory in bottles containing sterile sand. To each was added 10 ml of sterile 0·1% (w/v) peptone water and the bottles were shaken vigorously on a mechanical shaker for 3 min. Serial dilutions were prepared in the same diluent. Aliquots of 0·1 ml were spread on the surface of plates by the technique of Davis & Bell (1959). The medium used for samples from factory X was the basal medium of Gardner (1966) with the addition of 4% (w/v) NaCl; other samples were plated out on the same medium, in which the level of glycerol was reduced to 0·5% (w/v) and to which 1% (w/v) glucose was added. Colonies were enumerated after 4 days' incubation at 22°C.

Classification of isolates

After counting, each plate was divided into eight sectors and all the colonies on a number of sectors were picked off to give, where possible, about thirty isolates per sample. Purified cultures were classified by the methods shown in Table 1. Yeasts were identified solely on the morphology of Gram-stained preparations.

TABLE 1. Classification of bacon bacteria

Gram reaction	Morphology	Catalase	Method of classification
−	Rods	+	Harrigan & McCance (1966)
+	Cocci	+	Anaerobic acid production from glucose: +*Staphylococcus*, −*Micrococcus* (Baird-Parker, 1966)
+	Rods or cocci	−	Lactic acid bacteria
+	Pleomorphic rods	+	Coryneform bacteria
+	Rods	+	For *Microbacterium thermosphactum* (Gardner, 1966)

Results

The numbers and types of bacteria on the bacon sides matured in a stack are shown in Tables 2 and 3 and for those matured in a hanging position in Table 4. Within

TABLE 2. The number of types of bacteria found on Wiltshire cured bacon sides which were matured in a stack (factory X)

Site	Total no. of sides examined	Count/cm^2 ($\times 10^4$)			Total no. isolates examined	Incidence (%) of:				
		Min.	Max.	Mean		*Micrococcus*	*Staphylococcus*	*Acinetobacter*	*Vibrio*	Miscellaneous
Singed rind	5	0·78	83·5	20·2	101	88·3 (5)*	5·7 (2)	2·8 (1)	0	3·2†
Unsinged rind	5	1·2	16·5	7·98	99	94·4 (5)	1·6 (1)	4·0 (1)	0	0
Meat	5	0·03	8·5	2·72	104	64·2 (5)	0	5·9 (4)	19·0 (2)	10·9‡

* The figures in parentheses are the number of bacon sides on which the organism was found.
† Includes *M. thermosphactum* and yeasts.
‡ Includes *M. thermosphactum*, yeasts, *Alcaligenes*, *Aeromonas* and flavobacteria.

TABLE 3. The number and types of bacteria found on Wiltshire cured bacon sides which were matured in a stack (factory Y)

Site	Total no. of sides examined	Count/cm^2 ($\times 10^4$)			Total no. isolates examined	Incidence (%) of:						
		Min.	Max.	Mean		*Micrococcus*	*Staphylococcus*	*Acinetobacter*	coryneform bacteria	*Vibrio*	Lactic acid bacteria	Miscellaneous
Singed rind	5	0·13	7·7	2·4	107	80·5 (5)*	0	9·2 (4)	1·6 (1)	3·3 (2)	0·5 (1)	4·6†
Unsinged rind	5	0·15	50	15·3	168	64·9 (5)	10·4 (5)	10·4 (5)	3·1 (3)	1·2 (2)	1·2 (2)	8·8‡
Meat	5	0·08	4·35	1·7	141	59·8 (5)	1·8 (1)	13·0 (5)	6·9 (3)	2·7 (3)	3·7 (2)	12·1§

* The figures in parentheses are the number of bacon sides on which the organism was found.
† Includes *Pseudomonas-Achromobacter*, *Alcaligenes* and Enterobacteriaceae.
‡ Includes *Pseudomonas-Achromobacter*, yeasts and *M. thermosphactum*.
§ Includes *Pseudomonas-Achromobacter*, yeasts, *M. thermosphactum* and Enterobacteriaceae.

TABLE 4. The number and types of bacteria found on Wiltshire cured bacon sides which were matured in a hanging position (factory Z)

Site	Total no. of sides examined	Count/cm^2 ($\times 10^5$)			Total no. isolates examined	Incidence (%) of:		
		Min.	Max.	Mean		*Micrococcus*	*Acinetobacter*	Miscellaneous
Singed rind	4	0·38	11·4	4·5	100	95·2 (4)*	0	4·8†
Unsigned rind	5	0·66	30·4	13·8	119	90·5 (5)	5·8 (3)	3·7‡
Meat	5	1·4	23·5	9·1	110	86·0 (5)	9·8 (3)	4·1§

* The figures in parentheses are the number of bacon sides on which the organism was found.
† Includes *Aeromonas*, yeasts and unclassified Gram-positive, catalase positive rods.
‡ Includes *Aeromonas*, Enterobacteriaceae and unclassified Gram-positive, catalase-positive rods.
§ Includes yeasts, lactic acid bacteria and unclassified Gram-positive, catalase-positive rods.

one treatment there was a great variation in the number of bacteria/cm²; in a few cases there was a difference of a factor of $\times 200$. Therefore, because of the small number of samples, the figures only give a general indication of the level of contamination. In general the numbers on the unsinged sites were higher than the singed sites, and both were higher than the meat site. The overall degree of contamination was higher in the sides examined from factory Z.

The flora in all three sites was dominated by micrococci. On both the singed and unsinged rind samples there was from 3% to 12% *Acinetobacter* and *Vibrio*. Even though the total bacterial counts were on average higher on the latter site, there was no difference in the composition of the flora.

Micrococcus also predominated on the muscle samples, but to a lesser extent. *Acinetobacter* spp. were isolated from most sides, and other organisms such as *Vibrio* spp., coryneform bacteria and lactic acid bacteria were more frequently found. Also, the flora on all sites of the bacon sides from factory Y were more diverse in composition than that from factories X or Z.

Discussion

The average levels of contamination of the three sites examined in the present study ranged from 10^4–10^6/cm². Using an excision method of sampling, Gibbons (1940) reported mean counts on the pleural membrane of sides on receipt from the factory of $10^{4\cdot2}$–$10^{4\cdot5}$/cm². After 10–12 days' storage at 1·1°C these mean counts had risen to $10^{5\cdot18}$–$10^{6\cdot94}$/cm². Gibbons found large variations in counts of particular sites at any one time, e.g. the difference between the maximum and minimum loads on sides as received from the factory were log 3·0 (i.e. $1000x$)/cm² whereas after 10–12 days at 1·1°C this had increased to about log 5·0 ($100{,}000x$)/cm².

These variations may result from differences in the flora, different types of growth of the same organism, or variability in the method of detection (Gibbons, 1940). Garrard & Lochhead (1939) found that sides which were highly contaminated before going into cure remained contaminated after maturation. Therefore, variation in levels of contamination brought about by slaughter and butchery operations would be reflected in the bacon. Different practices at each factory would contribute to this. In most maturation cellars temperature is controlled, but variations between factories do exist. The relative humidity can vary within and between different factories. The results from factory Z suggest that the overall level of contamination of bacon sides is higher. This is not due to a difference in the method of maturation, as some of our unpublished data has shown that there is no difference between the rates of growth of bacteria on bacon sides matured in a stack or in a hanging position over a period of 7 days at 4°C. In addition, the types of bacteria isolated from sides from factory Y were more diverse than the two other factories examined.

Tofte Jespersen & Riemann (1958) demonstrated that the bacterial load on the rind was greater than that from the meat of a bacon side. After 7 days' maturation at 5°C,

counts on nutrient agar containing 4% NaCl were *c.* $10^5/cm^2$ for the rind and *c.* $10^4/cm^2$ for the meat. Ingram (1960) reported that bacon immediately out of cure carried a surface load of 10^4–$10^5/cm^2$, which increased more than ten-fold during a week of maturation at 4°C. Counts on the skin were on average higher than counts from the *longissimus dorsi* at both times.

Counts on the bacon sides in the present study, although variable, are of the same order as those reported by other workers. The bacterial contamination of the meat site tended to be lower than either the singed or unsinged rind, thus confirming the observations of Tofte Jespersen & Riemann (1958) and Ingram (1960). Of all the sites this would probably have the least opportunity for outside contamination. The unsinged rind had on average the highest levels of contamination. This is probably due to the fact that most of the organisms on the skin of the pig are killed in the singeing process. Also, the area of unsinged rind underneath the foreleg tends to be less well scraped during butchery.

It is now well established that micrococci are the predominant bacteria on matured Wiltshire bacon (Garrard & Lochhead, 1939; Brooks *et al.*, 1940; Ingram, 1952, 1960). The present work has confirmed this for both the rind and meat surfaces of a bacon side. However, there does appear to be an increase on the meat site of a proportion of Gram-negative organisms, such as *Acinetobacter* spp. and *Vibrio* spp.

Tofte Jesperson & Riemann (1958) found that on a nutrient agar with 4% NaCl cocci predominated on the rind side at most stages during a maturation period of 14 days at 5°C. However, on the meat side rods became predominant after 7 days at this temperature. Kitchell (1958) using a 10% NaCl medium found 42% Gram-positive bacteria on the skin and 63% Gram-positive bacteria on the muscle of bacon after 14 days' maturation. Tofte Jespersen & Riemann (1958) obtained higher counts from bacon using a brine agar (20% NaCl) on both the rind and meat of bacon after curing. Rod-shaped bacteria predominated in the meat, whereas both cocci and rods were of the same order on the rind except after 7 days, when rods appeared to outnumber the cocci. Gibbons (1940) found that the highest counts of bacon samples were obtained on media with 3–6% NaCl. Occasionally counts equal to or higher than those on 4% NaCl were obtained on 10% NaCl. It is difficult to assess these results because of the different media used and also the isolates were not fully identified.

Ingram (1960) found that the flora of matured bacon consisted of micrococci plus a roughly similar number of lactobacilli and a smaller proportion of Gram-negative rods. The latter accounted for 4–79% of the flora of the *l. dorsi* samples and 0·1–20% of the skin samples of bacon sides after 7 days' maturation. Slime on the muscle of refrigerated bacon can contain 50% lactobacilli, 33% *Micrococcus*, 10% Enterobacteriaceae and 7% *M. thermospactum* (Gardner, unpublished data).

The microbiological effect of salt in a food probably depends on osmotic withdrawal of water. This will be reflected in the water activity of the food. A brine concentration in bacon exceeding about 5% will exclude the normal pork spoilage flora, *Pseudomonas*,

but permit the growth of the *Micrococcus* (Ingram & Kitchell, 1967). On the basis of a_w it could be postulated that the musculature of bacon would have a higher value than the skin and thus explain the apparent increasing importance of *Acinetobacter* spp. and *Vibrio* spp. in the flora.

Gibbons & Rose (1950) found that with cured *psoas* muscle at a pH of 5·7 the flora was almost entirely composed of yeasts, whereas at pH 6·3 the flora was predominantly micrococci. Kitchell & Ingram (1965) demonstrated that the *ante-mortem* feeding of sugar to pigs resulted in a lowering of the pH of the musculature. There were fewer bacteria on the muscles of bacon from pigs treated in this manner, but there was no difference in the loads from the skin. These authors also concluded that there may be factors other than pH which effect the numbers and types of micro-organisms on bacon sides.

However, factors such as level of NaCl (a_w), amount of NO_2^- and pH (i.e. the physical and chemical characteristics of the environment) would have to be accurately defined in order to obtain a better understanding of the microbial ecology of Wiltshire cured bacon.

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The effect of various substances on the blooming of chocolate

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Summary. The effects of many edible substances and pure chemicals on chocolate have been studied in relation to the prevention of chocolate blooming. Bile acids, cholesterol, other sterols and choline promoted the blooming of chocolate. Tripalmitin, added in good dispersion, made chocolate very resistant to fat bloom and chocolate had a high gloss. Hydrogenated fats made very difficult temperable chocolate and it had a waxy taste. Only Delft 37 and some Edelfette improved the resistance of chocolate, but they made it waxy. Butylated hydroxyanisole (BHA) has undesirable effects.

Anhydrous glucose added at the rate of 15–20% of the weight of chocolate increased the resistance. Other sugars were either inert or diminished the resistance. Glycerol had a strikingly unfavourable effect on both quality and resistance. Amino acids did not exhibit any special influence on chocolate. Chemical additives and biological treatment also influenced the blooming of milk chocolate. Unusually long bloom crystals were produced on the surface of milk chocolate on special occasions.

Introduction

Dark chocolate is an intimate mixture of cacao butter, sugar particles, cacao particles and lecithin. Milk chocolate contains, in addition, dry milk solids and some other ingredients.

The surface of dark chocolate turns grey in storage, which is accompanied by a loss of gloss. This phenomenon is described as 'chocolate bloom'; there is 'fat bloom' and 'sugar bloom'. Fat bloom is more unsightly, and so is an important commercial factor (Clay, 1952). The bloom problem of milk chocolate is less important than that of dark chocolate.

The cause of bloom is not known, although it has been assumed that the unstable crystalline forms of cacao butter are the responsible factors (Vaeck, 1960; Wille & Lutton, 1966). Careful observations have shown that there must be many other reasons for the chocolate bloom (Clay, 1952; Sachsse & Rosenstein, 1953; Cerbulis,

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1955). Thorough chemical analysis shows that the chemical composition of the bloom fat differs from the original cacao fat (Cerbulis, Clay & Mack, 1957).

Many methods and additives have been proposed for the prevention of fat bloom (Clay, 1952; Martin, 1955a) but none are of practical value and most have the disadvantage of unfavourably affecting the flavour or texture of the chocolate (Clay, 1952).

The present work was undertaken to investigate which substances, present in chocolate or added to it, promote or prevent the appearance of chocolate bloom. In addition, an investigation was made of such edible substances which would have no effect on the flavour and texture of chocolate, and would prevent the appearance of chocolate bloom.

Experimental

The composition of standard chocolate used in all the tests was the same and the formula is as follows: cacao liquor, 40%; sugar, 48%; cacao butter, 10·17%; lecithin/cacao butter (1:1), 0·45%; vanilla paste, 1·35%; and vanillin, 0·03%. The average fat content of the chocolate was 32·3%. The formulation of all other testing samples was maintained the same throughout the tests, plus certain additives.

The chocolate was tempered by Clay's method (Martin, 1955b). The mass was agitated at 46–49°C, then tempered at 29–30°C and molded at 32°C in blocks of 20 g. Twenty blocks were prepared from each batch and placed into fibre cases in regular layers. A plastic-coated paper was inserted between each layer. Each

TABLE 1. Chocolate grading

Grade	Abbreviations used in Table 2	Explanation
Very good or good	vg	Chocolate has high gloss and no changes were observed on the surface of chocolate
Dull	O	Just recognizable or threshold tendency toward change; lustreless, gloss has disappeared but no bloom
Slightly bloomed	X	Some visible fat crystals on the surface
Very bloomed	XX	A grey layer of fat bloom of different thickness is formed on the surface of the chocolate. No recognizable changes present in the structure of chocolate
Very bloomed with changes in structure	XXX	A thick layer of fat bloom on the surface of chocolate. The structure of chocolate has changed; it is granular, brittle and light in colour

case contained twenty blocks. These cases were stored at a constant temperature of 19–20°C for 18 months or more. This test was applied to all samples, to evaluate the influence of additives to the chocolate, kept at constant temperature. The chocolate was examined every 3 months.

A second test was also carried out by keeping chocolate in an oven at 32–35°C during the days and at room temperature of 20°C at nights. This test was applied to chocolate containing some saturated fat or sugar as additive. These samples were examined every morning. Very different results were obtained with samples of the same batch when using both testing methods. These findings are discussed in 'Results.'

Intensity scale

A simple, constant six-point scale was employed for rating the intensity of the disappearance of the gloss of chocolate, i.e. the extent of appearance of 'chocolate bloom' (Table 1). There was, of course, a slight degree of difference within each unit. However, the skeleton scale was usually adequate, and had proved to be quite practical because it was surprisingly easy to decide whether a characteristic was just recognizable, or present at a slight or strong level. This system was used for the comparison purposes only.

Standard

The standard samples of chocolate were prepared periodically and kept in the same place and tested in the same time intervals as the experimental chocolate.

Results

Numerous combinations of compounds in different amounts were added to chocolate and numerous samples were prepared. Table 2 shows the results only from the most characteristic samples of each added substance or a combination of substances.

TABLE 2. Chocolate in storage

Substance added to chocolate	Weight of chocolate (%)	Appearance of chocolate during storage (months)					
		3	6	9	12	15	18
Standard (control)		g	O	X	XX	XX	XX
Bittersweet		vg	g	O	X	X	XX
Choline-HCl	0.075	O	XXX	XXX	XXX	XXX	XXX
Choline-HCl	0·125	XXX	XXX	XXX	XXX	XXX	XXX
Cholesterol	0·125	O	XX	XXX	XXX	XXX	XXX
Bile acids	0·0025	g	X	X	XX	XX	XX
Ceryl alcohol	0·1	X	XX	XX	XX	XX	XX
Phytic acid	0·01	X	X	X	XX	XX	XX
Ergosterol	0·01–0·1	O	X	XX	XX	XX	XX
Stigmasterol	0·01–0·1	O	X	XX	XX	XX	XX

TABLE 2 (*continued*)

Soybean sterols	0·01–0·1	O	X	X	XX	XX	XX
Tripalmitin	1·0*	vg	vg	vg	vg	vg	g
Sterotex	1·0	g	O	X	XX	XX	XX
Sterotex	3·0	XX	XX	XX	XX	XX	XX
Biscuitine	5·0	O	X	XX	XX	XX	XX
Delft C.B.S.A.	10·0*	O	O	O	X	X	X
Delft C.B.S.A.	20·0*	vg	vg	vg	g	g	g
Delft C.B.S.A.	40·0*	XX	XX	XX	XX	XX	XX
Edelfette 926/866	10·0*	vg	vg	vg	vg	vg	vg
BHA (butylated hydroxyanisole)		XX	XXX	XXX	XXX	XXX	XXX
Anhydrous glucose	10·0	vg	vg	vg	vg	vg	g
Cerelose	10–20	vg	vg	vg	g	X	X
Fructose	5–7	g	X	XX	XX	XX	XX
Lactose	3·5	g	g	g	O	X	X
Sorbose	2·5–7	g	X	XX	XX	XX	XX
Methyl-α-D-glucoside	2·5–7	g	X	XX	XX	XX	XX
Glycerol	0·125	g	O	X	XX	XX	XX
Glycerol	0·25–0·5	X	XX	XX	XX	XX	XX
Glycerol	1·0	X	XX	XX	XX	XX	XX
Glycerol	2·0	XXX	XXX	XXX	XXX	XXX	XXX
Methionine	1·0	O	X	XX	XX	XX	XX
Glutamic acid	1·0	X	X	XX	XX	XX	XX
Glycine	0·25	g	g	X	X	XX	XX
Glycine	0·5	g	X	X	XX	XX	XX
Glycine	1·0	X	XX	XX	XX	XX	XX
α-Amino-n-butyric acid	0·25	g	O	X	XX	XX	XX
α-Amino-n-butryic acid	1·0	O	X	XX	XX	XX	XX
Alanine	0·25	g	X	XX	XX	XX	XX
Alanine	1·0	X	XX	XX	XX	XX	XX
Tyrosine	0·25–1·0	X	XX	XX	XX	XX	XX
Cystine	0·25	vg	vg	X	X	XX	XX
Cystine	0·5	vg	g	X	XX	XX	XX
Cystine	1·0	X	X	XX	XX	XX	XX
Taurine	0·25	O	X	X	XX	XX	XX
Taurine	0·5	g	g	O	X	X	X
Taurine	1·0	g	g	g	X	X	X

*Chocolate fat (%).

Bile acids, cholesterol and choline

Bile acids made the chocolate unpalatable but did not influence the blooming of chocolate. Choline, a component of cacao bean (Polstonff, 1909) and cholesterol promoted the blooming of chocolate and completely destroyed the structure; it became granular and grey. When both substances were added to the same sample of chocolate, the negative influence was completely avoided, and the chocolate behaved like the

standard sample. Cholesterol in combination with bile acids did not influence chocolate bloom significantly.

Plant sterols, ceryl alcohol and phytic acid

Cacao bean contains stigmasterol, β-sitosterol, μ-sitosterol, two minor sterols (Eisner & Firestone, 1963), ceryl alcohol (Ruppol, 1943) and phytic acid (Arbenz, 1922). These substances were added separately and in different quantities to chocolate samples. Ceryl alcohol and phytic acid favoured the blooming of chocolate. Sterols influenced it very slightly.

Fats

The influence of different fats added to chocolate was tested. Some of these fats were hydrogenated or modified by special processing, and have been recommended for the use in the production of non-blooming chocolate. Raw materials used in the production of the modified fats were not known to the author and therefore only trade names could be used.

Tripalmitin (c.p.). 1% w/w of fats or 0·33% w/w of chocolate was added to the chocolate mass. It was dissolved in diethyl-ether and added dropwise to the chocolate mass by agitation at 45°C. Chocolate was agitated until all the ether was evaporated and then tempered as usual. The chocolate produced by this method had no tendency to bloom during the 36 months in storage and the taste was not affected by the additive.

Hydrogenated fats. Hydrogenated fats added to the chocolate mass introduced some difficulties in the preparation of chocolate. The chocolate crystallized very slowly, if at all (6 months and longer). The standard tempering procedure was not applicable and the chocolate mass had to be tempered in a different manner. The chocolate had no gloss and it had a waxy taste due to saturated fats. The same difficulties have been observed by others (Clay, 1952; Koch, 1954). Saturated cottonseed oil and saturated peanut fat promoted the blooming of chocolate.

Biscuitine (Swiss product). This had been recommended for the use in the production of bloom-resistant chocolate (Kleinert, 1953). Present experiments showed that Biscuitine did not improve the resistance of chocolate even if 15% of cacao fat was substituted by Biscuitine. There was no difficulty in tempering and crystallizing the chocolate and it did not have a waxy taste.

C.B.S.A. 37 (Oilworks Calve-Delft, Holland). This is a vegetable fat with a melting point of 37°C treated in a special way. This fat was recommended as a substitute for cacao fat to produce a non-blooming chocolate. In present experiments, with a substitution of 30–40% cacao fat with C.B.S.A. 37, the chocolate mass was very difficult to temper and had no tendency to crystallize. The chocolate was molded after tempering and seeding with scrap chocolate. This chocolate did not crystallize and had no gloss. After substituting only 20% of cacao fat with C.B.S.A. 37 and seeding the chocolate mass with scrap chocolate, the crystallization took place. Such chocolate had a

very high gloss and was very resistant to blooming. There were no appreciable changes after 2 years in storage. The only one disadvantage was that the chocolate had a slightly waxy taste.

Edelfette 926/866 (*Edelfettwerke, GmbH, Hamburg-Eidelstedt, Germany*). This was recommended as a substitute for cacao fat to produce non-blooming chocolate (Herzer, 1954). Present experiments showed if 30% of cacao fat was substituted with Edelfette 926/866, the crystallization did not occur and the chocolate was soft and with a waxy taste. There was no difficulty in tempering and molding the chocolate when only 10% of cacao fat was substituted by 926/866. The chocolate was very glossy and there were no appreciable changes even after 3 years in storage. The taste of the chocolate was satisfactory, with practically no waxiness.

Hydrogenated butter fat (with different degrees of hydrogenation). This was not fit for use in chocolate because it was soft and had a very unpleasant taste; fats separated out even when 3% of cacao fat was substituted.

Antioxident BHA (butylated hydroxyanisole). Even in small quantities, such as when used in fats, this had very unfavourable influence on the structure of chocolate, becoming very unsightly, white and granular.

Carbohydrates

Anhydrous glucose (Veatch, 1940; Pisarev, Karpov & Falunina, 1939; Sarotti, 1943; Fincke, 1951) and lactose (Sarotti, 1943) have been recommended as additives for the production of heat and fat-bloom resistant chocolate.

In present experiments, many sugars and polysaccharides were tested for the same reason. These substances were added to chocolate either by themselves or several together in many combinations, and in different weight ratios.

Anhydrous glucose gave high resistance against fat bloom when 15–20% of the weight of chocolate was added. The only drawback was that this chocolate left specific 'glucose aftertaste' in the throat after eating.

Lactose as an additive diminished the sweetness of chocolate which limited its use. It does not prevent fat bloom or improve other desired qualities of chocolate. The addition of fructose presented difficulties in tempering and the fat bloom results were controversial. A sample of the same chocolate, held at 20°C, bloomed more rapidly than the standard. On the other hand, the sample which was tested at high temperatures and unregulated humidity, presented a brilliant resistance to blooming. Sorbose, methyl-α-D-glucoside and guar flour accelerated the blooming of chocolate.

Glycerol. This had a subversive influence on the tempering and structure of chocolate. The addition of glycerol, 2% by weight of chocolate, resulted in an inability to harden the chocolate, and its structure was completely destroyed. Such chocolate was soft and with an unpleasant appearance. The addition of 1% or less of glycerol resulted in very accelerated blooming, even though the structure of the chocolate was unchanged. Glycerol is a constituent of cacao bean (Cerbulis, 1955).

Amino acids

Amino acids were powdered and then slowly added to the chocolate mass. The results were different but characteristic for each amino acid. Methionine and glutamic acid were inert to the blooming of chocolate. Glycine, α-amino-n-butyric acid, alanine, and tyrosine slightly accelerated the blooming. Cystine, added in small quantities (0·25%), improved the resistance, however, used in 1% addition seemed to have unfavourable influence. Taurine definitely improved the resistance of chocolate.

Milk chocolate

Milk chocolate is regarded as a very bloom-resistant chocolate. Actually, the blooming occurs more frequently than assumed and is hardly noticeable. This is the reason for overestimation of the bloom-resistance of milk chocolate (Cerbulis, 1955).

Milk chocolate was compared with dark chocolate in present experiments, and the latter proved to be much more susceptible to fat bloom. However, on several special occasions, quite extensive 'blooming' of milk chocolate was observed and the bloom so formed was very long (6 mm and more) and peculiar in shape. This was quite different from the bloom observed on dark chocolate (Cerbulis, 1955; Sachsse & Rosenstein, 1953). The bloom on milk chocolate was obtained by following two different procedures: (a) adding certain chemicals to milk chocolate mass, and (b) biological treatment of cacao beans before roasting. This particular type of bloom was a characteristic only of milk chocolate. Dark chocolate did not react in such a manner when submitted to the same kind of treatment.

(a) *Chemicals added.* The intimate mixture of sodium taurocholate (0·00125%), sodium glycocholate (0·00125%) and cholesterol (0·0025%) was added to milk chocolate. Long, white crystals appeared on the surface of milk chocolate within a few months. This experiment was easily reproducible.

(b) *Biological.* Fermented cacao beans were soaked in water for 24–48 hr, then slowly roasted and submitted to regular processing. Long, white crystals appeared on the surface of milk chocolate after 6–9 months. The same phenomenon can be observed when yeast is added to the soaking water. Enzyme diastaze, added to the soaking water, produced milk chocolate which bloomed similarly to dark chocolate without the forming of long crystals.

The soaking of cacao beans in water had considerable influence on the tempering of chocolate. Such chocolate crystallized slowly or not at all and had no gloss. In order to determine the cause of this phenomenon more experiments would be necessary.

Discussion

Fat bloom is a pure fat fraction of cacao fat. It might well be that bloom is simply the growth of crystal agglomerates of the VI crystalline state (Wille & Lutton, 1966) from submicroscopic to macroscopic size brought about by digestion, migration and

resolidification of fat molecules under the influence of small or large temperature variations, and has nothing to do with the internal crystal modifications.

Some separation of glycerides has been taking place and part of the more saturated glycerides is forced to the surface of the chocolate (Cerbulis, Clay & Mack, 1957). It is known that a more tempered chocolate forms fat bloom much slower than a less tempered one. This finding favours Becker's several component phase theory (Becker, 1957, 1958) but not the theory of polymorphic changes of cacao fat (Vaeck, 1960). As found in present experiments and in previous observations (Clay, 1952; Cerbulis, 1955), some non-fat substances take a distinct part in fat bloom phenomenon. From this point of view all tested substances could be classified in three groups: (a) inert, no influence on chocolate; (b) substances which promote the blooming, and (c) substances which retard the blooming of chocolate.

The bloom formation of milk chocolate which was prepared from cacao beans treated by biological methods is unexplained. Some milk components probably take an active part in the formation of chocolate bloom.

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Effect of blanching on mineral and oxalate content of spinach

BENGT L. BENGTSSON

Summary. Unblanched and blanched spinach were sampled from a factory line with conventional water blanchers. The samples were analysed for potassium, sodium, calcium, magnesium, phosphorus, nitrate and soluble and insoluble oxalate. The main results show that spinach takes up calcium from the water during blanching. This increase in calcium content of the spinach has a direct influence on the ratio between the soluble and insoluble form of oxalate. Loss of nitrate is higher than that of the other water soluble constituents.

In a small trial, an attempt was made to increase the nutritional value of spinach by precipitation of all soluble oxalate through addition of excess of calcium chloride to processed, chopped spinach. However, a taste panel rated the enriched sample as inferior owing to its colour. A small blanching trial with tap, distilled and softened water showed that the amount of calcium in tap water has no such undesirable effect on colour.

Introduction

Before being frozen, most vegetables require blanching to inactivate enzymes, to reduce bacterial contamination and to remove raw or bitter flavour. Blanching is carried out only to such an extent that the most heat stable enzymes, such as peroxidase, are barely inactivated. Blanching involves an unavoidable loss of nutrients such as ascorbic acid and sucrose and the loss is especially serious for leafy vegetables owing to its large surface-to-volume ratio. The above-mentioned general effects of the blanching process have been reviewed by Lee (1958). Below, some earlier relevant publications are summarized.

Kramer & Smith (1947), who studied the influence of steam and water blanching on calcium and phosphorus content of spinach, found that calcium increases and that phosphorus decreases during water blanching.

In a more recent investigation, Schaller (1962) showed that the total oxalate content of spinach is reduced to about 80% of that of the raw material during 3 min blanching in distilled water.

It is well known that spinach is rich in oxalate ranging from 5 to 10% of its dry

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weight. Only part of this oxalate is balanced by calcium and magnesium in the spinach plant, the rest being in a water soluble form and physiologically active in depriving calcium from other ingredients of the diet. As oxalate is regarded as a negative factor for the nutritive value, it seemed worth-while to try to find out whether or to what extent large scale processing affects the oxalate content.

Experimental

Variety and fertilization

The same variety of spinach (*Spinacia oleracea* L.)—a line of Viking—was used throughout. The spinach was fertilized with 1500 kg/ha of a compound fertilizer containing 12% N, 12% P_2O_5 and 17% K_2O. Irrigation was adjusted to secure optimum growth.

Sampling

The spinach was sampled from a factory line with conventional water blanchers. The unblanched samples were taken after they had been washed (about 10 l/kg spinach); the blanched ones, after they had been washed, blanched, cooled and chopped. The unblanched and blanched samples were taken from the same batch of raw material. The blanching was carried out for 3 min with a water blancher with a draper belt. The estimated amount of water used for blanching and cooling was 10 l/kg spinach. The blanching trial with tap water, distilled water and softened water was carried out in a pilot plant under conditions as similar as possible to those in the factory. The values given in Table 1 are means of fifteen samples taken on different dates during the spring season.

Chemical analysis

The different analytical methods used have previously been described (Bengtsson, Bosund & Hylmö, 1967). All values are given as percentages of the dry weight of the spinach.

Results and discussion

Blanching of spinach involves a considerable loss of dry matter, the size of the loss varying with different conditions such as the ripeness of the spinach and the load on the spinach line. Analyses covering several years have shown that during blanching and cooling, spinach loses on the average about 25% of its total dry matter. This value has been used below in the estimation of blanching losses and retention of the various minerals and oxalate (Table 1).

In Table 1 (columns A and B) it is shown that the amounts of calcium and insoluble oxalate increase through the blanching process. This result is in agreement with the figures given by Kramer & Smith (1947). The increase in calcium has a

direct effect on the ratio between the soluble and insoluble form of oxalate, as insoluble oxalate consists mainly of calcium oxalate.

The total oxalate decreases only slightly and much less than in the studies carried out by Schaller (1962). This difference can be explained by the fact that we used very hard (360 ppm $CaCO_3$) water. This results in a high retention of the calcium by the spinach and in a precipitation of soluble oxalate otherwise extracted with the blanching water. From a nutritional point of view, it is desirable to have a low content of soluble oxalate, as this form is physiologically active and can deprive calcium from other parts of the diet. It is evident that blanching reduces soluble oxalate content by about half (Table 1).

TABLE 1. Composition of unblanched and blanched spinach and estimation of blanching losses and retention of the various minerals and oxalate (assuming a 25% loss of total dry matter)

	A Unblanched (g/100 g)	B Blanched (g/100 g)	C Blanched $B \times \frac{75}{100}$ (g/75 g)	D Blanching losses A − C (g/100 g)	E Retention $\frac{C}{A} \times 100$ (%)
Potassium	6·93	4·04	3·03	3·90	43·7
Sodium	0·54	0·41	0·31	0·23	57·4
Calcium	2·21	3·05	2·29	−0·08	103·6
Magnesium	0·28	0·24	0·18	0·10	64·3
Phosphorus	0·64	0·55	0·41	0·23	64·1
Nitrate	2·50	1·00	0·75	1·75	30·0
Total oxalate	8·80	8·37	6·28	2·52	71·4
Soluble oxalate	4·17	2·17	1·63	2·54	39·1
Insoluble oxalate	4·63	6·20	4·65	−0·02	100·4

The values shown are the means found for fifteen unblanched and fifteen blanched samples.

The values shown in Table 1 (columns A and B) give only the percentage composition of unblanched and blanched spinach. In order to estimate total blanching losses of the various substances, column C was calculated from column B. Columns D and E give blanching losses and percentage retention of the various substances. The sum of the losses shown in column D is 11·17 g or about 45% of the total loss of dry matter.

Calcium is increased during blanching (columns A and B), but the retention is rather close to 100% (column E). The relative increase of calcium can be explained by the loss of other solids (estimated at 25%; see above). It is, however, not possible to deduce from our data whether any exchange of calcium occurs in the spinach with calcium

in the water. This could, however, be possible to determine after blanching spinach in water with added ^{45}Ca.

The relative increase of calcium during blanching results in a relative increase of insoluble oxalate. It can be calculated from the values in Table 1 that the increase of calcium is not equivalent to the increase of insoluble oxalate. Thus about 20% of the increase of calcium is bound in other compounds such as pectates. That calcium during blanching reacts with pectates is well known and this reaction results in toughening of tissues such as peas. The toughening of such a product as chopped spinach is, however, of no significance.

Table 1 shows the effect of blanching on the nitrate content. The nitrate values have already been presented in detail elsewhere (Schuphan *et al.*, 1967). It is, however, interesting to note that the loss of nitrate is larger than that of the other elements. This is a great advantage since the nitrate content of spinach should be as small as possible from a nutritional point of view. The larger loss of nitrate compared with potassium and sodium is due to the higher blanching loss of soluble substances in the petioles compared with the leaves and the uneven distribution of nitrate in the spinach plant. Thus the concentration of nitrate is 5–10 times higher in the petioles than in the leaves of the spinach plant, while the corresponding figures for potassium and sodium are between 1·2 and 1·5.

Table 1 also shows that the retention of magnesium and phosphorus is higher than that of potassium, sodium and nitrate. This could be expected as part of the magnesium and the phosphorus is bound in compounds not soluble in water. Magnesium is a component of the chlorophylls and phosphorus a component of the phospholipids.

The blanching process has a desirable effect on the nutritional value of spinach in that it reduces the amount of nitrate and soluble oxalate. This effect could be enhanced by the use of still more water per weight of spinach during blanching and cooling. But such a treatment would result in higher and undesirable losses of total solids and valuable nutrients such as asorbic acid. The reduction of the amount of soluble oxalate can, however, be achieved by precipitation with calcium chloride. Such calcium-enriched spinach samples were evaluated by a taste panel, who found the enriched sample to be inferior in colour. This discoloration is due to the precipitated oxalate that gives the spinach a greyish-white shade. Since no loss of the bright-green colour of spinach can be tolerated, this form of calcium enrichment is unacceptable.

As addition of excess of calcium chloride to chopped spinach had an undesirable effect on colour, a small trial was carried out to find out whether the calcium content of the blanching water also had an undesirable effect. Spinach was blanched in tap water, distilled water and softened water. No difference in colour was demonstrable. But chemical analysis of oxalate revealed interesting differences as shown in Table 2. Blanching in tap water resulted in the highest content of calcium and insoluble oxalate, but the lowest content of soluble oxalate. No differences in nitrate content could be expected and none was found.

TABLE 2. Effect of blanching in tap water, distilled water and softened water on the composition and colour of spinach

	Tap water (g/100 g)	Distilled water (g/100 g)	Softened water (g/100 g)
Calcium	3·69	2·72	2·71
Nitrate	1·05	1·01	1·06
Total oxalate	7·05	5·80	5·70
Soluble oxalate	0·64	0·84	0·83
Insoluble oxalate	6·41	4·96	4·87
Colour	Normal	Normal	Normal

Although no analyses were carried out on the unblanched spinach used in the trial illustrated in Table 2, it can be estimated from the data given in Tables 1 and 2 that the total oxalate content of spinach blanched in distilled water or softened water is about 80% of that of the raw material. This figure is in good agreement with the value reported by Schaller (1962).

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The relationship between the subjective and objective measurement of pork colour

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Summary. The colour of ninety-seven pig *longissimus dorsi* muscles was measured using a spectrophotometer, a simple colour reflectometer, and an untrained panel of ten graders. The results obtained from the colour meters and the graders were compared with Munsell V value of the samples, calculated from the reflection spectra.

Visual colour grading produced a near linear correlation with the Munsell V value. Dark samples tended to be underestimated, whereas pale samples were overestimated by the panel. There was considerable variation between observers' scores, between methods of visual grading, and between visual and instrumental grading. Results obtained from the colour meter were reproducible. The error in comparison with the spectrophotometer was small. The colour meter provides a convenient portable and inexpensive method of measuring the colour lightness of pork muscle.

The experiment showed the practicability of objective measurement of meat colour lightness. The determination of colour quality, however, requires both subjective and objective measurements.

Introduction

An initial reaction of a consumer in purchasing meat is to determine whether the meat colour is 'acceptable' or 'unacceptable'. If acceptable and a choice is available, the meat with the 'most desirable' colour will be selected, cost, presentation and the fat-lean ratio being constant. The acceptable/unacceptable boundary and the most desirable point will vary among consumers.

In pork muscle, which contains less pigment than beef (Lawrie, Pomeroy & Cuthbertson, 1968), the change in hue and chroma is small between the 'fresh cut' and 'bloomed' surfaces (Elliott, 1969). Differences in muscle colour between carcases are in lightness, influenced by rate of glycolysis *post mortem* (Pfau, 1967, 1968). Only prolonged or unsuitable storage will cause brown discolouration, due to the oxidation of the surface pigment (Brooks, 1938).

Because of the nature of meat, it is difficult to prepare accurate and permanent

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standards for use in visual colour grading. This has promoted the measurement of meat colour using spectrophotometers and colour filter reflectometers, which have a reference standard such as magnesium oxide, carbonate or colour stable materials.

The colour lightness grading of the pig *longissimus dorsi* is carried out at most progeny testing stations. Portable colour meters have replaced in many cases the former systems of visual grading (Lohse & Pfau, 1964; Steinhauf, Pahl & Weniger, 1964; Mirna, 1965; Charpentier & Verge, 1967). The E.E.L. 'Smoke stain' reflectometer has recently been introduced in England for the colour grading of pig muscle (MacDougall, Cuthbertson & Smith, 1968).

Most investigators, when using instruments for colour grading of foods, have found sizable variations between instruments (Robinson *et al.*, 1952; Friedman, Marsh & Mackinney, 1952; Shau & Worthington, 1954; Schmidt & Idler, 1958; Livingston, Gersten & Shore, 1958; Barrett *et al.*, 1965). In a detailed examination using sixteen instruments Little, Chichester & Mackinney (1958) found differences between models of the same instrument. Some workers select a spectrophotometer as their reference instrument (Pfau, Lohse & Bohne, 1965; Weniger, Vold & Steinhauf, 1965) and use it or a colour meter which has been calibrated against it for the examination of meat samples.

The relationship between 'colour quality' as recognized by the consumer, 'colour grade' as used in the meat industry by producer and processer, and 'colour specification' as defined by colour science is inadequately defined. The present work compares the colour measurement of pork using a spectrophotometer, a colour meter and a panel of graders. Meat colour quality and colour grades are defined in psychophysical colour terminology, thus permitting quantitative colour analyses.

Materials and methods

The muscle used for analysis was the *l. dorsi* of ninety-seven Landrace and Large White pigs. Samples were taken between the fourth thoracic and fifth lumbar vertebra 20 hr *post mortem*.

(a) *Spectrophotometric analysis*

From the centre of the sample a core 2·5 cm diameter and 1·4 cm in length was placed in a glass cell with similar interior dimensions. The reflection spectrum from 400 to 700 mμ was recorded with an Optica CF 4R spectrophotometer [Optica (U.K.) Ltd, Walthamstow, London], fitted with 10 cm diameter integration spheres. The procedure is described elsewhere (Elliott, 1967).

The C.I.E. tristimulus values were calculated from the reflectance curve using the 10 point selected ordinate method (Hardy, 1936; Mackinney & Little, 1962; Elliott, 1968). The C.I.E. Y brightness reading was converted to the Munsell V value using a nomogram (Elliott, 1968).

(b) *Colour meter analysis*

Immediately following the spectrophotometric analyses, the samples were measured by the E.E.L. 'smoke stain' reflectometer (Evans Electroselenium Ltd, Halstead, Essex). The percentage reflection (% R) was recorded for each sample after adjusting the meter to 100% R with the MgO standard used in (a). The samples were again measured after the reflectometer was adjusted to 50% R with a Formica disc, B.S.I. Sarum grey 9/096 (MacDougall *et al.*, 1968).

(c) *Visual colour analysis*

The cell containing the sample was placed in a lighting cabinet, painted matt grey. Illumination was provided by two 2 ft 40 W fluorescent tubes, type 55 [Philips Electrical (Lighting Division) Ltd, London W.C.2], positioned at 45° to the sample. The light intensity at the sample position was 200 lumens. The angle of viewing by the grader was 90° to the sample surface. The distance from eye to sample was approximately 70 cm. These conditions duplicate as closely as possible the 1931 C.I.E. system of colour specification for illuminant C (Wright, 1964).

Ten members of the laboratory staff, who had been screened for colour blindness defects with test cards (Ishihara, 1959), were used as colour graders. Each grader performed individually the following tests in the same order, without knowledge of previous graders' scores or instrument readings. All graders were not available each week.

(i) The sample was placed in the cabinet and scored as being either acceptable or unacceptable in colour lightness to the grader.

(ii) After the 1st week of colour grading the following test, a memory score, was included after test (i). The samples were randomized and placed individually in the cabinet and the observer was asked to grade it from 4·5 to 6·5 based on the memory of the previous week's standards (test iii).

(iii) Two samples were selected each week with a V value of 5·0 $\pm$ 0·1 and 5·5 $\pm$0·1 (determined from reflection spectra). These were placed in the cabinet and the observer informed that these samples represented the range of medium colour lightness and should be scored as grade 5·5. Any sample lighter than the paler standard should be graded as pale (grade 6) or very pale (grade 6·5). Samples darker than the high standard should be graded as dark (grade 5) or very dark (grade 4·5). Each grade was considered to have the same range as that shown by the grade 5·5 standards. The randomized test samples were placed individually between the standards and the observer asked to grade the sample.

(iv) The standards were removed and all samples placed together in the cabinet. The grader was requested to arrange the samples in groups with just perceptible colour lightness differences, members within each group being indistinguishable. When the groups had been compiled and any rearrangements completed, the standards were re-introduced. The observer was asked to grade each group in relation to the

standards, with the possibility that several groups could receive the same grade number.

Results

Instrumental analysis

(a) *Spectrophotometer.* All samples were grouped in 0·5 units of *V* and are shown in Table 1. The colour lightness of the samples was within the range 4–6·5 *V* with the majority between 4·5 and 6 *V*.

When the % *R* at three wavelengths, 480 mμ a reflection maximum, 525 mμ, isobestic point of oxygenated, reduced and oxidized myoglobin, and 618 mμ reflection maximum, was compared with *V* (Fig. 1), the highest correlation was obtained at 480 mμ, which decreased with increasing wavelength.

(b) *Colour meter.* The results by the two methods of measuring % *R* were compared

TABLE 1. Distribution of the colour lightness of the muscle samples

Munsell V value	Visual grade number	No. of samples per grade	Description of colour lightness
4·0–4·5	4½	2	Very dark
4·5–5·0	5	26	Dark
5·0–5·5	5½	44	Medium
5·5–6·0	6	18	Pale
6·0–6·5	6½	7	Very pale

TABLE 2. Visual colour grading by ten observers of pig *l. dorsi* muscle. Comparison of visual grading methods

Grader	No. of sessions	No. of samples	% of samples graded similarly using spectrophotometric and visual grading with: (a) without standards	(b) with standards	(c) with standards after grouping	% of samples graded similarly by visual grading with standards (d) with and without grouping
A.N.	6	97	80	81	79	82
H.D.	4	61	66	69	72	57
A.M.	6	97	68	76	78	78
D.W.	6	97	70	74	70	88
A.C.	5	73	63	67	73	61
J.M.	5	73	68	67	65	72
M.K.	6	84	74	74	80	86
A.P.	4	68	64	75	80	71
S.G.	3	49	47	80	72	71
A.G.	3	52	80	77	91	78

with the *V* value. The MgO reference method (Fig. 2a) and the grey standard (Fig. 2b) were similar.

This was confirmed when % *R* of the spectrophotometer at three wavelengths was compared with the 100% *R* and 50% reference colour meter readings (Fig. 3a and b).

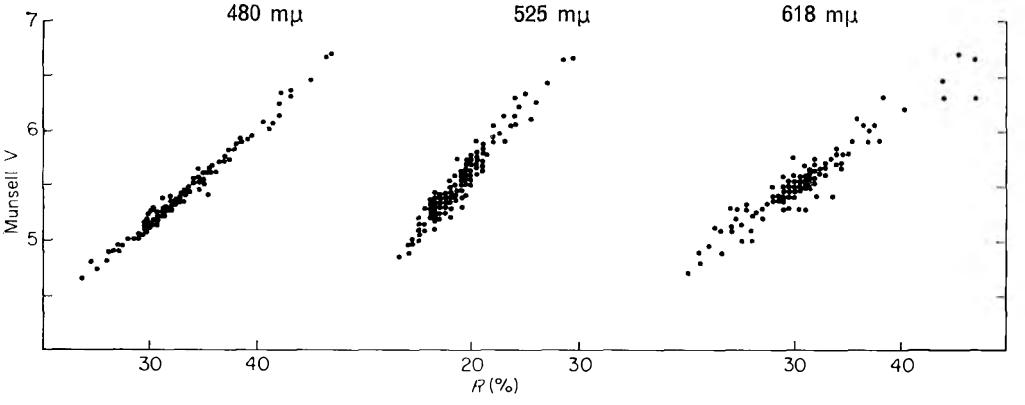

FIG. 1. The correlation between the spectrophotometer % *R* readings at 480, 525 and 618 mμ and Munsell V values computed from % *R* readings between 400 and 700 mμ.

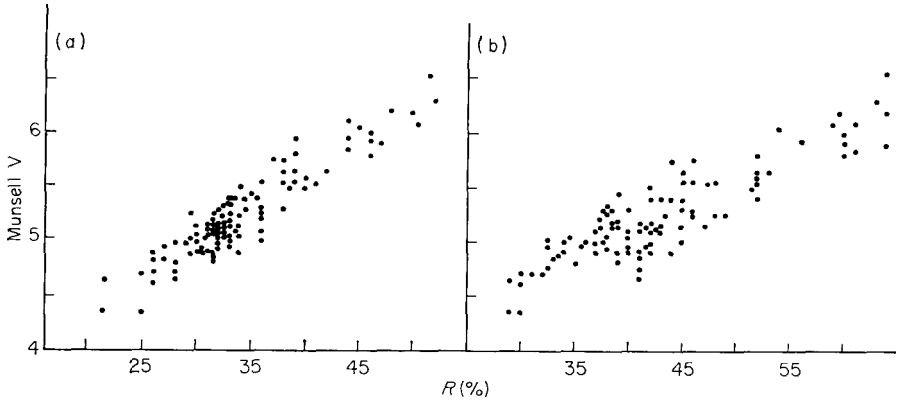

FIG. 2. A comparison of the colour meter (% *R* readings) with the spectrophotometer values (after transformation to Munsell V) (a) colour meter set at 100% *R* with MgO; (b) colour meter set at 50% *R* with grey Formica.

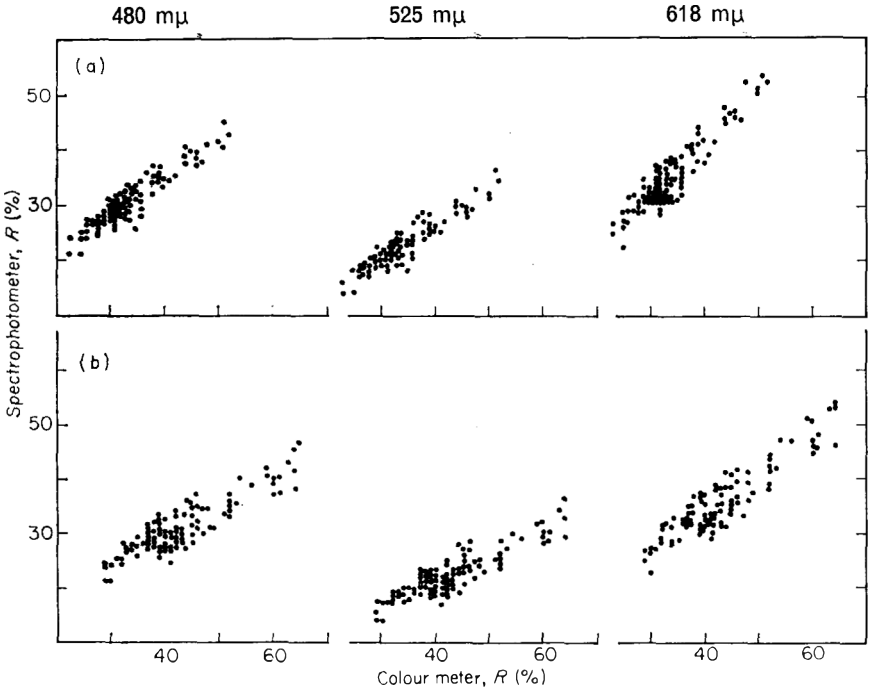

FIG. 3. A comparison of the colour meter values (a) 100% R with MgO; (b) 50% R with grey formica, and the spectrophotometer values (% R at 480, 525 and 618 mμ) 100%R 400 to 700 mμ with MgO.

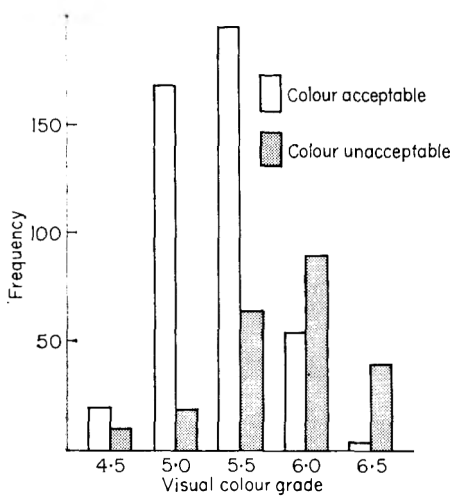

FIG. 4. The acceptable and unacceptable colour scores of ten graders for ninety-seven samples of *l dorsi*. Samples grouped in 0·5 units of Munsell V.

Visual colour grading

(a) *Acceptability ratings.* The largest acceptablity frequencies were found in grades 5 and 5·5 (medium to dark), which decreased with increasing paleness, e.g. in grade 5, 91% of the scores were acceptable, whilst in grade 6·5 only 5% were acceptable (Fig. 4).

(b) *Colour grading.* The visual grading scale is based on 0·5 units of Munsell V. The *V* values as calculated from the reflection spectra were assumed to have a standard error of $\pm 0{\cdot}1$ *V*. This reading was taken as the reference value for each sample. The ability of the panel to judge the samples correctly by the three methods is summarized in Table 2.

About half of the panel members correctly scored the same percentage of samples by all three methods. For the remainder of the panel there was little difference between the percentage scored correctly by the two methods employing standards, but these methods were better than the systems relying on a memory standard. Using standards, the panel was able to score by both methods on average 75% of samples correctly; if five graders were chosen on merit by their ability to score correctly, the overall average would be increased to 80%. When the two methods of visual grading with standards are compared, an average of 74% was similarly scored by the panel.

The experimental design permitted an examination of the nature of the difference between the visual and instrumental grading methods. The grading scores occurring within each grade as determined with the spectrophotometer were averaged and the standard deviation calculated. This was performed for the three methods of visual grading and the results are shown in Fig. 5. All comparisons showed the same effect, the medium colour lightness, grade 5·5, was close to the reference point. Darker samples were underestimated, whereas pale samples were overestimated.

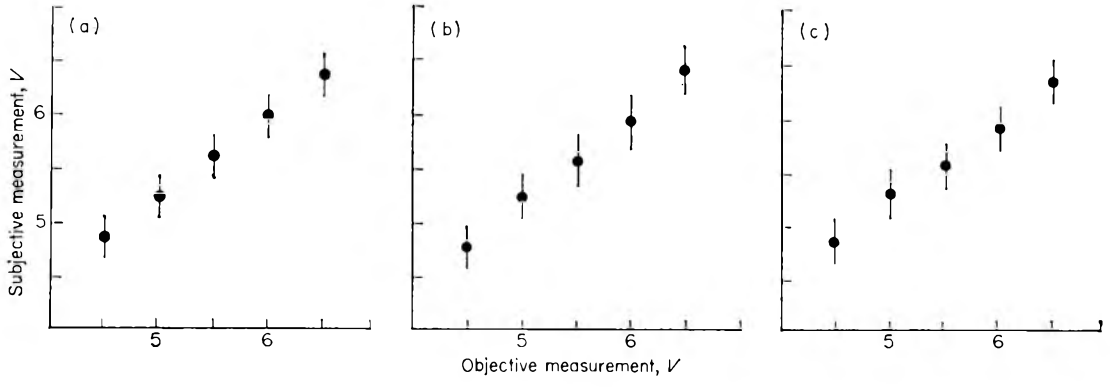

FIG. 5. Tests for linearity of visual colour lightness grading of pig *l. dorsi*. Mean and standard deviations of ten graders. (a) Memory grading; (b) grading with standards; (c) grading with standards after grouping, *versus* spectrophotometer grading.

Discussion

The 'brightness' (C.I.E.) or 'lightness' (Munsell) ordinate of colour is the property which is involved in visual colour grading of fresh meat (Mirna, 1955; Grau & Mirna, 1957; Pfau *et al.*, 1965; Steinhauf *et al.*, 1966). The results obtained in this experiment have shown that visual grading of pork has a linear correlation with Munsell V, which is a linear scale of colour lightness (Nickerson 1946; Wyszecki & Stiles, 1967). The deviation from linearity which occurs at the pale and dark ends of the scale indicates the need for a range of colour standards when visually grading meat lightness.

The acceptable/unacceptable ratings over the lightness range of the material used in this study may not apply to other groups of observers. Marion & Stadelman (1958), when measuring the acceptability of frozen turkey skin colour, found differences in preference between panel members from urban and rural areas. Allen (1968) has indicated that there is a preference for pale coloured meat in southern England, whilst in northern England a darker colour is more desirable.

The results indicate that there is a relationship between the colour lightness and the colour quality of the samples (Fig. 4). However, with the number of samples and observers employed in this experiment, caution is required in the interpretation.

Colour grading can be used to provide either a guide to conditions during processing or as a measurement of consumer acceptability. In the latter case a colour grading system is an attempt to produce a quantitative measurement of colour quality.

The assessment of meat quality is an important aspect of meat science. By definition it is difficult to measure in practical terms (Bate-Smith, 1953).

The conditions for visual assessment in the present study were standardized as far as possible, as the panel were asked to score only on colour. The samples were presented in a standard shape and size with the absence of fat, which could perhaps influence the grader. When pork was viewed under tungsten light, the panel found differences between samples more difficult to distinguish (Elliott, unpublished data). Daylight type fluorescent lighting is commonly used in retail stores and, therefore, its use is more appropriate for subjective assessments (Mirna, 1965; Pfau *et al.*, 1965; Heinz, 1968).

The panel error can only be partially estimated from grading reproducibility. On comparing the results between the two methods of grading with standards (Table 2), the panel average was 74% similarly scored. This is of the same order as the 75% of samples scored by the panel in the same grades as the spectrophotometer. It is, however, difficult to relate these data to the overall panel error. By providing grading standards *c.* one-half of the panel members' scores improved. This could form a useful method for screening graders.

Little (1964) using a series of apple sauce mixtures with small colour lightness increments obtained panel scores between 85% and 96% correct. To improve subjective scores, meat may require larger colour lightness intervals or further selection and training of the grading panel.

For the measurement of pork colour photoelectrical instrumental methods have many advantages over subjective assessments, such as precision, reproducibility and ease of operation. Of the two instruments used in this work the reflectance spectrophotometer can be regarded as the reference instrument (Nickerson, 1946). For routine use (e.g. in-line measurements) a cheaper, portable colour meter would be satisfactory.

Several portable colour meters have been designed or modified to provide meat colour grading on a factory processing line and at carcase dissection stations (Winkler, 1939; Steinhauf *et al.*, 1964; Lohse *et al.*, 1964; Mirna, 1965; Charpentier *et al.*, 1967; MacDougal *et al.*, 1968; Mohler, 1968). The tungsten light source and the selenium cell of the E.E.L. 'Smoke stain' reflectometer contain no monochromatic filters. In Fig. 6 it can be seen that over 50% of the relative energy of an incandescent lamp is at wavelengths above 600 mμ; most of this light, however, will be reflected from the meat surface (Elliott, 1967). Meat appears 'redder' under this light as compared with daylight or fluorescent illumination (Fig. 6).

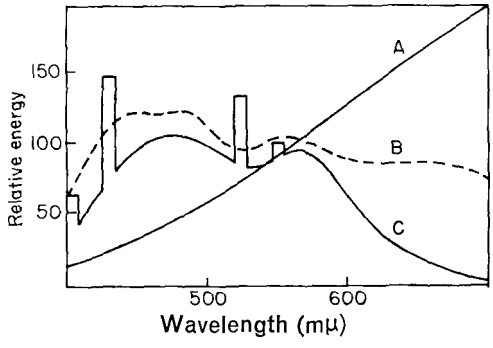

FIG. 6. Relative spectral energy distribution of three sources of illumination: (A) incandescent lamp, (B) sunlight, (C) white fluorescent lamp (after Wyszecki & Stiles, 1967).

The final arbiter of the colour quality of meat is the eye, and any instrumental procedure must of necessity be closely related to this.

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Caking of onion powder

Y. PELEG AND C. H. MANNHEIM

Summary. Caking of onion powders during storage reduces their commercial value. The addition of anti-caking agents at several moisture levels was investigated. Low storage temperature, moisture content of less than 4% in the powder and some of the anti-caking agents tested prevented caking of onion powders for prolonged periods.

Introduction

One of the properties expected from food powders is their ability to remain free flowing. However, it is often found that powders, even when packed in hermetically sealed containers, lose the free flowing property in storage by caking which reduces their commercial value. Several methods are known for reducing the occurrence of caking and one, widely used in the case of onion powder, is the incorporation of anti-caking agents. Very little is published on the caking phenomenon, its causes, mechanism, rate, methods of inhibition, etc.

Factors known to affect caking of powders include moisture, temperature, particle size, composition and electrical charge.

The presence of moisture, whether naturally present in the sample or artificially introduced, is necessary for caking to take place. If a material is relatively insoluble in water the effect of water on its caking characteristics is greatly decreased (Irani, Calls & Liu, 1959). Caking was found to be related to moisture content of granular fertilizers and it was greatly reduced by drying to less than 1% moisture content (Whynes & Dee, 1957).

Caking is known to be induced by fluctuation in temperature. It has been postulated (Irani *et al.*, 1959) that as the temperature falls, the saturated solutions which are present on the surface undergo crystallization thus binding one particle to another. However, other investigations (Whynes & Dee, 1957; Lazar & Morgan, 1966) have found that caking is rather related to an increase in temperature. Particle size was reported to become a significant factor below 100 μ, since small particles tend to adhere (Craik & Miller, 1958; Craik, 1958). Prevention of adhesion-caking can be achieved by the addition of adjuncts with particle size sufficient to prevent the adhesion phenomena. The negative charge of the anti-caking-agent's particles was found to be effective in reducing caking tendency (Irani *et al.*, 1959; Nash, Keither & Johnson, 1965).

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Recently a review on chemicals for improving the flow property of powders has been published (Burak, 1967). These anti-caking agents may be silicates, phosphates, starch, etc., which have a small particle size compared to the powder.

The purpose of this work was to determine the efficiency of various anti-caking agents in preventing caking in locally produced onion powders, and to investigate the influence of moisture content on the tendency to undergo caking.

Materials and methods

Caking

As yet there is no method for the prediction of the tendency of a powder to undergo caking, nor for measuring the rate of caking. The method adopted for evaluating the suitability of anti-caking agents for onion powder was by preparing a test pack as follows:

Commercially dehydrated kibbled onion flakes (Egyptian variety) were milled in a Waring Blender and screened through a 30 mesh sieve. The different kinds of commercial anti-caking agents were added at 2% level (see Table 2) and mixed well in a blender. All products were packed in 100 cm^3 (211 × 108) tin cans, sealed and stored at 35°C.

The additives were chemicals recommended by their manufacturers as anti-caking agents and approved by the U.S. Food and Drug Administration for use in foods. Samples were checked visually, for the occurrence of caking.

Moisture content of onion powder was determined by drying a sample of 5–10 g at 70°C in vacuum oven at 25 mmHg absolute pressure, for 16 hr.

TABLE 1. Influence of temperature on caking tendency of onion powder without adjuncts

Storage temperature	Tendency of caking
35°C	Caking occurred after 72 hr
30°C	Caking appeared after 7–10 days
25°C	Agglomerates formed during 7–10 days
15°C	The powder could be stored even for 6 months without anti-caking agents, without any significant effect on the flow properties

Results and discussion

The caking of onion powder at the 4–5% moisture level was greatly affected by storage temperature when stored without any adjuncts (see Table 1). It was found that caking is strongly accelerated by higher storage temperature.

TABLE 2. Rate of occurrence of caking in onion powder with 2% of anti-caking agent

No.	Anti-caking agent	Rate of occurrence (days)
1.	Calcium stereate; Technical; B.D.H., England	More than 35
2.	Aluminium silicate; Precipitated; B.D.H., England	More than 35
3.	Zeolex 7; J. M. Huber Co., U.S.A.	Less than 3
4.	Zeolex 23A; J. M. Huber Co., U.S.A.	4–14
5.	Flowgard; Pittsburg Plate Glass International, U.S.A.	4–14
6.	Syloid 72; W. R. Grace, U.S.A.	4–14
7.	Syloid 244; W. R. Grace, U.S.A.	4–14
8.	Magnesium silicate, Sepdelen Werken, Germany	3–6
9.	Calcium stereate M.K., Penick Co., U.S.A.	14–25
10.	Magnesium stereate, Penick Co., U.S.A.	More than 14
11.	Felco, John Manville Co., U.S.A.	Less than 3
12.	Santocel C, Monsanto Co., U.S.A.	Less than 3
13.	Santocel 62, Monsanto Co., U.S.A.	More than 25
14.	Cab-O-Sil, Cabot, U.S.A.	17–25 days
15.	Aluminium silicate (Alusil), J. Crossfield Co., England	More than 25
16.	Calcium aluminium silicate, J. Crossfield Co., England	3–7
17.	Neosyl, J. Crossfield Co., England	3–7
Control		Less than 3

The effect of anti-caking agents on the occurrence of caking at 35°C is shown in Table 2. From these results it can be concluded that out of the eighteen chemicals tested only seven were effective. They are: calcium and magnesium stearates, aluminium silicate, Santocel 62 and Cab-O-Sil. The addition of aluminium silicate was found to have also a significant beneficial effect on the colour of the powder.

When onion powders contained 7% moisture there was no advantage in the addition of anti-caking agents. Caking occured after 72 hr at 35°C. Onion powder which had been dried to 3% moisture level remained free flowing even after 30 days at 35°C, without anti-caking agents.

Conclusions

The results obtained revealed means for maintaining the free-flowing property of onion powder, a quality which affects the commercial value.

Onion powder can be kept for extended periods at ambient temperature, by reducing the moisture content to about 3%. Alternatively, when an onion powder has a moisture content of 4–5% it should be stored below 15°C, or when stored at ambient temperature —a suitable anti-caking agent must be incorporated. The increase in drying expenses

should be checked against the cost of low temperature storage, or the addition of anti-caking agents.

The lack of information on caking of food powders calls for an intensive investigation of this phenomenon.

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Texture studies on mushrooms

T. R. GORMLEY

Summary. Measurement of texture is a useful quality control test for mushrooms. Results of these studies suggest that texture differences in mushrooms may be divided into primary and secondary differences. The former refer to differences caused largely by variation in the dry matter content of mushrooms, the latter to differences caused by variation in the nature of the dry matter content.

The shear press was used for measuring mushroom texture. Shearing mushrooms previously sliced with a household egg slicer gave more accurate results than shearing whole individual mushrooms. The relative precision of the shearing operation was the same for different weights of sample but increasing slice size had a slight positive effect on the shear press reading.

Taste panels were capable of detecting texture differences in cooked mushrooms which were also detected by the shear press.

Introduction

Texture, dry matter content and whiteness are three quality control tests that can be carried out on mushrooms. The latter two are straightforward and simple to assess; texture, however, is more involved and needs further investigation.

Texture studies on mushrooms have shown that mushrooms covered with a synthetic film continued to toughen for a period after harvesting (Gormley & MacCanna, 1967). Mushrooms were sheared individually on an Allo–Kramer shear press using a standard test cell. Initial weight plotted against shear press reading for a number of mushrooms on any particular day of the 5-day experiment gave a texture line for that day ($r \geqslant 0{\cdot}875$) (Gormley & MacCanna, 1967). Shearing mushrooms individually is time consuming and it was decided in the present study to shear mushrooms that have previously been sliced; a representative sample can be easily obtained and thus fewer shear press readings are required. Size of slice and variation in weight of material sheared were also studied.

Since mushrooms for shearing are weighed into the shear press cell it is necessary to find whether texture of mushrooms is related to both moisture and cellular material (dry matter) content. If texture is caused largely by cellular material the shear press value should be correlated with the dry matter as well as with the fresh weight.

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Shearing equal fresh weights of mushrooms, from growing treatments which give different moisture contents, may only show texture differences which are caused by variations in dry matter content. In this paper these are referred to as primary texture differences. However, other texture differences may be caused by variations in the nature of the cellular material and these are referred to as secondary texture differences. To obtain an indication of secondary texture differences it would be useful to express texture of mushrooms as the force required to shear mushrooms containing a certain weight of cellular material. The dry matter figure can be chosen arbitrarily, e.g. 3 g would be suitable since 35 g of fresh mushrooms (the fresh weight normally sheared) contain approximately 3 g of cellular material.

Experimental

Slice size and sample weight

Freshly harvested mushrooms (*Agaricus bisporus*) from the same growing treatment were sorted into sizes by weight, i.e. 4–6 g, 6–8 g, . . ., 18–20 g. Stipes were cut flush with the underside of the cap and the mushrooms in each group were sliced vertically with an egg slicer giving slices $\frac{3}{16}$ in. thick. Slices (35 g fresh weight) from each group were sheared on an Allo–Kramer shear press using the standard test cell (Kramer, Burkhardt & Rogers, 1951) to study the effect of slice size on the shear press value.

Batches of sliced mushrooms ranging in weight from 5 to 50 g (5 g increments) were sheared (fifteen readings each) to obtain the relative precision of the shearing operation at each weight.

Dry matter estimations

Two methods of measuring dry matter were compared for precision and convenience:

(1) Thirty mushrooms (stipes trimmed) were sliced with an egg slicer. Ten grams of slices were put in each of ten weighed dishes and dried in a vacuum oven at 70°C and 560 mmHg.

(2) One hundered grams of sliced mushrooms were blended with 100 ml of water for 3 min. Twenty sub-samples were removed, placed in dishes, and dried in a vacuum oven at 70°C and 560 mmHg. Most of the water had been previously removed by placing the dishes on a steam bath for 0·5 hr. The result was multiplied by two to account for the added water.

Texture and dry matter content

Three separate shearing experiments were carried out.

Experiment 1. Twelve samples of freshly harvested sliced mushrooms, from the same nitrogen growing treatment, were sheared to study the contribution of moisture and dry matter contents to texture. Samples weighing 10–50 g (5 g increments) were sheared in triplicate and three dry matter estimations were made. Correlations between shear press reading × dry matter content and shear press reading × fresh weight were obtained.

Experiment 2. Mushrooms from a watering trial and a storage trial were utilized to show that equal fresh weights of mushrooms (35 g) containing different dry matter contents gave different shear press values. It was necessary to combine mushrooms from the seven replications of the watering trial in order to obtain sufficient material for the shearing experiment.

Shear press readings and dry matter contents were carried out in triplicate on sliced mushrooms (35 g) from each treatment. Mushrooms from the water treatments were sheared 0·5 hr after harvesting. The experiment was replicated once.

Water treatments

(a) Compost containing five times its own weight of water (83·3%).
(b) Compost containing four times its own weight of water (80·0%).
(c) Compost containing three times its own weight of water (75·0%).
(d) Compost containing twice its own weight of water (66·6%).

Storage treatments

(a) Harvested mushrooms, covered and stored at 15–21°C for 24 hr.
(b) Harvested mushrooms, covered and stored at 15–21°C for 48 hr.
(c) Harvested mushrooms, covered and stored at 15–21°C for 72 hr.
(d) Harvested mushrooms, uncovered and stored at 15–21°C for 24 hr.
(e) Harvested mushrooms, uncovered and stored at 15–21°C for 48 hr.
(f) Harvested mushrooms, uncovered and stored at 15–21°C for 72 hr.

All mushrooms used in the storage part of the experiment were obtained from the same growing treatment. The term 'covered' mushrooms refers to 8–12 mushrooms in a Hartman Foodtainer tray $5\frac{1}{2} \times 5\frac{1}{2} \times \frac{3}{4}$ in. covered with the synthetic PVC film Resinite. The term 'uncovered' mushrooms refers to 8–12 mushrooms in a similar unwrapped container.

Experiment 3. Six samples of sliced mushrooms (15–40 g; 5 g increments) from each of twelve different treatments were sheared to examine the linearity of the relationship between shear press reading and fresh and dry weights of sliced mushrooms. The different treatments used are listed below.

(a) Nitrogen supplementation treatment (cottonseed meal).
(b) Nitrogen supplementation treatment (dried blood).
(c) Harvested mushrooms, uncovered and stored at 15–21°C for 24 hr.
(d) Harvested mushrooms, uncovered and stored at 15–21°C for 48 hr.
(e) Harvested mushrooms, uncovered and stored at 15–21°C for 72 hr.
(f) Harvested mushrooms, prepacked (Resinite) and stored 15–21°C for 24 hr.
(g) Harvested mushrooms, prepacked (Resinite) and stored 15–21°C for 48 hr.
(h) Harvested mushrooms, prepacked (Resinite) and stored 15–21°C for 72 hr.

(i) Compost containing five times its own weight of water.
(j) Compost containing four times its own weight of water.
(k) Compost containing three times its own weight of water.
(l) Compost containing twice its own weight of water.

Texture of cooked mushrooms

Mushrooms from different growing treatments, harvested 48, 24 and 0·5 hr, were sliced with a household egg slicer and simmered gently with margarine in three separate dishes for 20 min. Prior to cooking, the mushrooms were stored at 15–21°C in Hartmann Foodtainer dishes $5\frac{1}{2} \times 5\frac{1}{2} \times \frac{3}{4}$ in. wrapped with the synthetic film Resinite. Texture and dry matter estimations were made at harvest time and again before cooking. After cooking the three samples were allowed to drain on wire sieves and were submitted to a six-member taste panel. The panel was asked to rate texture only. The most recently harvested sample (0·5 hr) was offered as standard (0) and also as a coded sample. Increasing chewiness was given as +1, +2 and decreasing chewiness as −1, −2. The panel was repeated once.

After draining for 10 min triplicate samples (35 g) of the cooked mushrooms from each of the sieves were removed and sheared.

Results and discussion

Slice size and sample weight

Large mushroom slices gave a higher shear press reading than small slices (Table 1). Therefore, when shearing a sample it is important to mix the slices thoroughly before taking the 35 g sub-sample.

TABLE 1. The effect of slice size on the shear press reading using a 35 g sample

Slice size (mushrooms grouped by weight) (g)	Shear press reading (lb force) (mean of five readings)
4–6	122
6–8	114
8–10	129
10–12	136
12–14	140
14–16	143
16–18	144
18–20	141

The relative precision of the shearing operation using different sample weights was calculated from equation (1) (Kramer & Twigg, 1962):

$$n = \left(\frac{ks}{p}\right)^2, \qquad (1)$$

where n = relative precision;
s = standard deviation;
k = 3 for 99% assurance; and
p = 0·01 for 99% assurance.

A relative precision of three was obtained for the different sample weights tested. Thirty five grams was chosen for most of the shearing operations because this quantity filled the standard test cell to about three-quarters of its capacity and ensured a good subsample size.

Dry matter estimations

Of the two methods compared the blender method gave the more precise results (Table 2) and was also the more convenient. It is essential that the sub-sample removed contains added water and fresh mushrooms in the ratio 1 : 1. This ratio is obtained provided the sub-sample is taken rapidly after blending. Multiplication of the weight of dried residue by two gives the dry matter content. If the mushrooms contain more than 10% dry matter it is sometimes necessary to blend one part mushroom with two parts water in order to obtain a blend of the desired consistency for subsampling.

TABLE 2. Comparison of two methods for estimating the dry matter content of mushrooms

Method	Mean dry matter (%)	Standard error	Time for drying at 70°C and 560 mmHg
(1) Drying slices directly (ten estimations)	7·395	±0·367	12 hr
(2) Drying blended slices (twenty estimations)	7·586	±0·064	5 hr

Texture and dry matter content

Experiment 1. A high linear correlation (r = 0·982) was obtained when fresh mushroom sample weights (10–50 g, 5 g increments) were correlated with the corresponding shear press readings. The dry weights and water contents associated with

the above fresh weights gave correlation coefficients of 0·979 and 0·991, respectively, when correlated with the shear press values. It was hoped at this stage to obtain partial correlation coefficients for dry weight × shear press and water content × shear press and thus have an indication of the relative contributions of cellular material and water content (associated with cellular material) to the overall texture reading. However, the partial correlation coefficients could not be calculated because of the high relationship between dry matter and water contents (r = 0·988).

Experiment 2. Equal fresh weights (35 g) of mushrooms from a watering trial and a storage trial gave different shear press readings (Tables 3 and 4). Those mushrooms containing the highest dry matter content also had the highest shear press reading (Tables 3 and 4) which suggests that the higher the ratio of cellular material (dry matter) to water content, the higher the shear press reading.

The results (Table 3) show that the texture of mushrooms from different water treatments is quite similar if the results are calculated on a dry weight basis. When calculated on a fresh weight basis, however, texture differences do exist. This suggests that these differences are largely dependent on variations in the dry matter content of the 35 g samples sheared and can be called primary texture differences.

Previous work has shown that covering harvested mushrooms with a synthetic film reduces water loss and causes the mushrooms to toughen during storage (Gormley & MacCanna, 1967). Texture differences are obtained for covered mushrooms when the shear press readings are taken either on a fresh weight or dry weight basis (Table 4). Since equal dry weights give different shear press values this suggests that the nature of the cellular material is changing during storage. Texture differences due to changes in the nature of the cellular material can be called secondary texture differences.

TABLE 3. The effect of different water levels in the growing compost on the dry matter content and texture of mushrooms

Weight of water in compost (%)	Dry matter (%)	Dry weight §	Shear press reading† (lb force)	Shear press reading per 3 g dry weight‡
83·3	9·45	3·31	138	123
80·0	8·69	3·04	126	124
75·0	9·06	3·17	137	130
66·6	10·76	3·77	159	126
F-test (treatments)	***	***	***	N.S.
SE (df = 3)	±0·0493	±0·0185	±1·2750	±1·2250

† Shear press reading for 35 g fresh weight.
‡ Obtained by interpolation.
§ Dry weight corresponding to 35 g fresh weight.
*** Significant (P=0·001).
N.S., Not significant.
SE, Standard error.

In the case of uncovered mushrooms large texture differences were obtained on a fresh weight basis but only very slight differences on a dry weight basis (Table 4). This suggests that texture differences for uncovered mushrooms are primary texture differences caused mainly by variation in dry matter content due to the moisture losses taking place during storage. It must be assumed, however, that the moisture content does contribute to some extent to the texture reading because of its effect on cell turgor pressures. The loss of moisture, therefore, in uncovered mushrooms should cause a reduction in the shear press reading (expressed on a dry weight basis) with time. Since no reduction in shear press reading was observed it is possible that secondary texture differences existed and their positive effect on the shear press reading was counterbalanced by the negative effect caused by moisture loss.

The shear press readings are adjusted to a common denominator (Tables 3 and 4), i.e. 3 g dry weight, by approximation or interpolation. The approximation method is based on the fact that the texture line for shear press reading × weight of material sheared (either fresh weight or dry matter basis) is linear and almost goes through the origin. Hence, as an approximation, it suffices to divide the shear press reading by the dry weight and multiply by three. The interpolation method is more accurate and is again based on the fact that texture lines for all treatments tested were linear (see Experiment 3).

TABLE 4. The effect of different storage treatments on the dry matter content and texture of mushrooms obtained from the same growing treatment

Storage treatment	Dry matter (%)	Dry weight §	Shear press reading† (lb force)	Shear press reading per 3 g dry weight‡ (lb force)
Covered 24 hr	7·62	2·67	132	149
48	7·68	2·69	156	170
72	7·56	2·65	172	194
Uncovered 24 hr	9·86	3·45	170	152
48	11·04	3·86	198	151
72	12·15	4·25	221	157
F-test for two factor interaction	***	***	*	*
SE ($df = 5$)	±0·18	±0·02	±1·41	±4·58

† Shear press reading for 35 g fresh weight.
‡ Obtained by interpolation.
§ Dry weight corresponding to 35 g fresh weight.
* Significant ($P=0·05$).
*** Significant ($P=0·001$).
N.S., Not significant; SE, standard error.

Experiment 3. Six batches of mushrooms, from each of twelve treatments, were sheared to examine the linearity of the relationship between weight of mushrooms sheared and the shear press value. Twenty four texture lines (two for each treatment) were drawn by plotting both the fresh weights and the corresponding dry weights against the shear press readings. These relationships were linear and correlations between weight of material sheared and shear press reading were $\geqslant 0{\cdot}989$ excepting one which was 0·971. It will suffice in future experiments to shear only two mushroom samples (25 and 40 g) in order to obtain a texture line. The corresponding texture line for dry weight versus shear press reading can be readily obtained and the shear press reading equivalent to 3 g dry weight calculated by interpolation.

Texture of cooked mushrooms

In all but one case the relative texture differences between batches of mushrooms were not changed by cooking. Absolute texture readings fell by 32·2–48·7% depending on the texture of the mushrooms before cooking (Table 5). The mushrooms used were from various growing treatments and had different shear press values when harvested. However, those stored for 48 hr were tougher than those stored for 24 hr irrespective of the texture when freshly harvested. The panels were carried out on consecutive weeks but were not replicated each day because of difficulties in obtaining cooking facilities. The panel members rated the two batches of samples in the same

TABLE 5. Taste panels on cooked mushrooms from different storage treatments

Storage treatment	Shear press reading when freshly harvested‡ (lb force)	Shear press reading before cooking‡ (lb force)	Shear press reading after cooking† (lb force)	Average panel score for texture	Loss in shear press reading on cooking (%)
Panel 1					
Covered 48 hr	110	164	84	−1·17	48·7
24 hr	124	154	95	+0·33	38·3
Freshly harvested	134	134	90	−0·33	32·8
Panel 2					
Covered 48 hr	128	166	102	+0·67	38·7
24 hr	126	156	86	+0·50	45·0
Freshly harvested	106	106	72	0·00	32·2

† Reading for a 35 g sample on a cooked weight basis.
‡ Reading for a 35 g sample on a fresh weight basis.

order as the shear press. The reliability of the panels is borne out by the texture scores given to the coded sample of the standard, i.e. −0·33 (downgraded) in panel one and 0·00 (correct score) in panel two. The percentage loss in shear press reading on cooking is greatest for the stored mushrooms. Toughening during storage is probably caused by changes in the nature of the cellular material and cooking seems to nullify this process to some extent. Adhering margarine on the 35 g mushroom slices sheared after cooking also lowers the shear press value since the 35 g sample contains less mushroom tissue. In panel one the mushrooms covered for 48 hr were the toughest before cooking and softest after cooking. This may have been due to the fact that they had a low dry matter content when harvested as indicated by the shear press reading of 110 lb.

Conclusion

This investigation shows that dry matter contents and textural properties of mushrooms vary under different conditions. Tests for these characteristics should be part of any quality control programme. Shearing a given fresh weight of sliced mushrooms gives an incomplete picture of texture and it is necessary to express texture differences on a dry weight basis as well; this gives the opportunity to distinguish between primary and secondary texture differences. Secondary texture differences seem to develop to the greatest extent in covered (prepacked) mushrooms and this may be due to certain properties of the covering film, e.g. its ability to reduce water loss and modify the atmosphere in the prepack.

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Studies on the quality characteristics of canned grapefruit segments

I. Factors affecting the drained weight and texture*

A. LUDIN, ZDENKA SAMISH, A. LEVI
AND ESTER HERSHKOWITZ

Summary. Segments prepared from grapefruits (var. Marsh Seedless) soften rapidly in the course of maturation of the fruit as evident from shear press readings for freshly peeled and canned segments. Storage of the fresh fruit for up to 9 days decreases firmness of the fresh segments and the loss in firmness increases with progressing maturation. Time and temperature of pasteurization affect significantly the firmness of the segments as well as their 'tendency to break'. The storage of the canned product, for up to 1 year had little effect upon drained weight, whereas texture and colour of the canned grapefruit segments were affected significantly.

Introduction

Grapefruit segments tend to lose considerable weight and firmness upon canning. This loss varies according to the characteristics of the fresh fruit and to the canning technique, particularly the concentration of the syrup and the conditions employed during pasteurization. The highly acid taste of the fresh grapefruit segments is usually compensated for by the addition of a heavy syrup. With a syrup of about 40° Brix, as much as 400 g or more of fresh peeled grapefruit segments have to be placed in a size A2 can (307 × 411) in order to assure a drained weight of not less than 300 g canned segments, at about 20° Brix.

The high loss in weight of grapefruit segments is accounted for mainly by diffusion (Sterling, 1959), because of the gradient in sugar concentration between the added syrup and the grapefruit segments. The drained weight appears to be affected also by other solutes such as amino acids, metal ions, organic acids, etc. Even if so important, diffusion is by no means the only factor involved in the change in weight due to canning. The middle lamela of the grapefruit cells is composed of pectic (Webber & Batchelor,

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1948) as well as celluloitic material (Sterling, 1959). Both of these substance, which are also present in the ripe segments, can affect the texture as well as the drained weight of canned segments by absorbing water (or sugar syrup) which is rapidly imbibed throughout the cell wall (Webber & Batchelor, 1948). In the living cell, water movement is primarily an osmotic phenomenon, but the lethal heat treatment during the canning process destroys the selective permeability of the living parenchyma cells.

The increase in weight of canned fruits is explained by Sterling (1959) to be due to a swelling of the polysaccharides (pectins, cellulose) during cooking. Sterling & Chichester (1960), using sucrose ^{14}C, observed in canned peaches that sucrose penetrated from the syrup with higher sucrose concentration into the fruit, mainly near the cell walls of the conducting tissue. Luh, Leonard & Mrak (1959) report an increase in the drained weight of canned apricots, as the concentration of the sugar syrup is increased. With canned grapefruit segments it seems that even under the best processing techniques, the drained weight will decrease due to the rupture of the delicate juice sacs. Changes in the organoleptic properties, such as texture and colour of canned, stored grapefruit segments, were studied by Hugart, Wenzel & Moor (1959). The relation between fresh fruit quality, heat treatment during canning, storage conditions of the product and the quality of the canned grapefruit segments, is reported in this paper.

Materials and methods

Grapefruits (var. Marsh Seedless) were picked from one group of trees in a commercial grove for all pilot plant experiments, while fruits for the industrial trials were obtained from packing houses.

The firmness of the fresh grapefruit segments was evaluated on fruit picked every fortnight throughout the harvesting season from November to March. The fruit was collected from ten trees in a 7-year old, healthy grove. Shear press measurements were made on fresh and on canned segments prepared from identical samples.

In order to evaluate the effect of the storage of the fresh fruit on the firmness of the grapefruit segments, 100 grapefruits were stored at 21°C and lots of 20–25 fruits were checked for up to 9 days of storage.

If not stated otherwise the pilot plant trials were based on batches of ten cans, and the industrial trials on batches of seventy-two cans for each treatment.

The effects of temperature and of length of storage upon the quality of canned grapefruit segments were determined in a uniform batch of 192 cans divided into lots of forty-eight cans each, which were stored at four different temperatures (0°, 10°, 21°C and room temperature—15–29°C) for period up to 1 year. Samples of twelve cans each were examined after 30, 180 and 360 days of storage, respectively.

The basic technological steps in preparing the canned segments were as follows: the freshly picked fruit was stored at 21°C overnight, before treatment and then washed and dipped in boiling water for 4–5 min to facilitate removal of the peel. The peeled

fruit was segmented by hand and the segments were immersed in 2% lye solution at 98–99°C for about 12 sec, washed with tap water sprays and dipped in a 2% citric acid, solution for 30 sec. Peeled segments (420 g) were placed in cans (size A2) with 170 g of hot (90°C) sugar-syrup per can. The strength of the syrup was adjusted to reach a cut-out strength of 20% T.S.S. The cans were exhausted in a water bath at 80°C for 10 min, pasteurized, cooled to about 35°C and then stored at 21°C.

After 30 days of storage of the cans, the following examinations were made:

(1) The *drained weight* and the amount of *broken segments* were determined as described in Israeli Standard No. 112 for 'Canned Grapefruit Segments' (Standards Institute of Israel, 1966).

(2) *Firmness* was measured with the help of a Lee-Kramer shear press with a 250-lb ring and hydraulic pressure of 150 lb/in^2. The whole segments were placed in the standard cell to a uniform volume. The firmness is expressed in shear press units (S.P.U.).

(3) *The tendency of the segments to break* during handling and transportation was evaluated by a newly developed method, which serves as an indicator of the capacity of the segments to remain whole until the product reaches the customer. The method consists of placing 100 g of whole segments in a standard jar (70 mm diameter and 120 mm in height), adding 100 ml of water, shaking the closed jar for 5 sec in the case of canned segments and for 15 sec in the case of fresh segments, under the following standard conditions: the jar was placed in a horizontal position and exposed to 180 movements per minute at a distance of 24 cm from the axis through an arc of 8 cm (see Fig. 1). The contents of the jar were transferred immediately to a flat tray and the unbroken, whole pieces were removed. The broken segments and water were placed on a 1-mm sieve and weighed after draining for 2 min. The tendency to break was expressed as the percentage of broken segment out of the total. Since samples of 100 g were used, the percentage is identical to the weight of the broken segments. The deter-

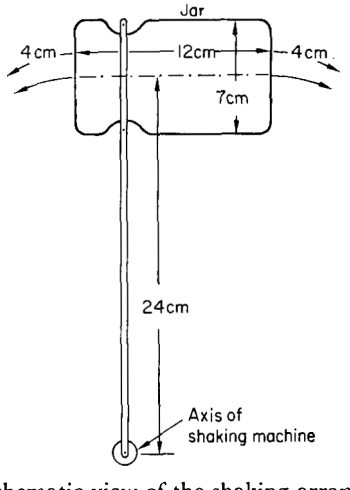

FIG. 1. Schematic view of the shaking arrangement.

mination was repeated with five or more samples taken at random, and the reported results are the averages of five measurements—so as to compensate for the natural differences occurring between the grapefruit segments.

Results and discussion

A panel of judges which was asked to grade canned grapefruit segments according to their firmness graded all samples with a reading of less than 450 shear press units (S.P.U.) as 'too soft', samples between about 450 and 600 S.P.U. as 'soft', and those between 600 and about 850 S.P.U. as 'firm'. Segments with a reading of more than 850 S.P.U. were considered to be 'tough'.

The firmness of the grapefruit segments is shown in Fig. 2 to decrease rapidly during the course of maturation of the fruit. During early November fresh segments measured, on the average, 930 S.P.U. and the canned segments, 660 S.P.U. At the end of the citrus season (mid-March) the fresh segments averaged only about 460 S.P.U. and the canned segments about 380 S.P.U.

The storage of fresh grapefruits before canning was shown to cause a decrease in the firmness of the segments. Shear press readings were made on the day of picking and after 2, 7 and 9 days of storage. The average change in firmness of the segments is reported in Fig. 3 as the difference in S.P.U. between the initial firmness of the fresh fruit and that of the stored samples. Fig. 2 shows that softening of the segments progresses more rapidly at the end of the season (March), when the fresh grapefruits are already quite soft. Differences in S.P.U. after 7 days of storage of grapefruits harvested in March were 120 S.P.U., but only 80 S.P.U. for fruit harvested during January–February.

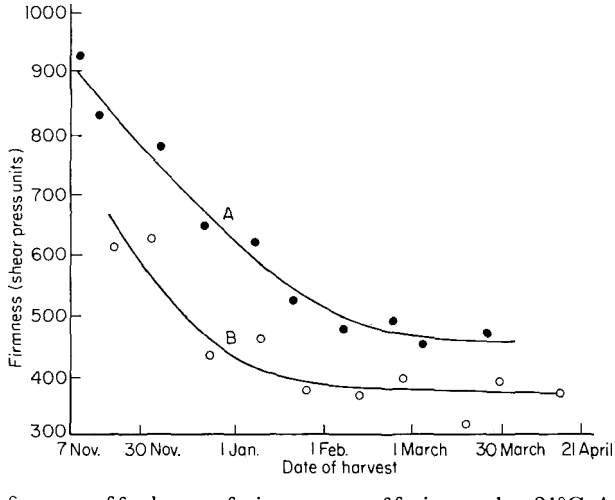

FIG. 2. Loss in firmness of fresh grapefruit segments of fruit stored at 21°C. A, Fresh segments; canned segments.

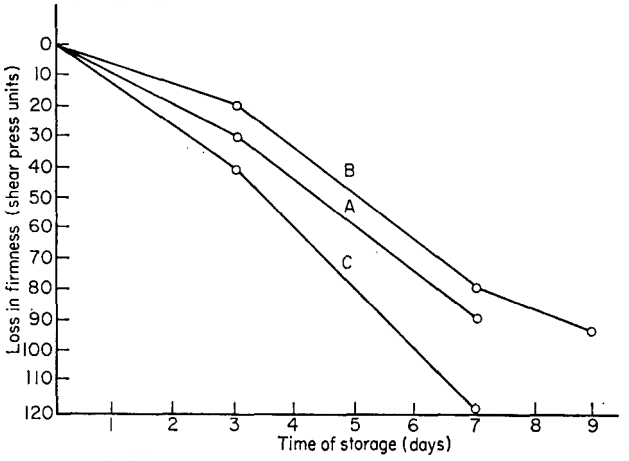

FIG. 3. Seasonal changes in firmness of fresh and canned grapefruit segments. A, November–December; B, January–February; C, March.

TABLE 1. The relation between time of pasteurization of canned grapefruit segments and their texture

Pasteurization		Firmness (S.P.U.)		Broken segments	Tendency to break
Temp. (°C)	Time (min)	A	B	(g/can) B	(%) B
80	5	840	—		
	10	790	1060	34	15
	15	600	—	—	—
	20	630	1030	26	14
	25	510	870	28	16
	30	480	740	24	22
	35	490	—	—	—
	40	410	600	28	29
	50	—	500	31	30
90	20	—	800	33	25
100	15	—	510	38	33
F-value		Highly significant (99% level)	significant (95% level)	Highly significant (99% level)	Highly significant ((99% level)
LSD ($P = 0{\cdot}05$)		± 58	± 175	± 5·6	± 3·5

A = Pilot plant trials; B = industrial trial.

Prolonged heat processing of the segments is liable to cause rupture of the juice sacs as well as softening of the grapefruit segments, resulting in lower drained weight, loss in firmness and higher tendency to disintegration. Such changes were recorded for canned berries by Board, Gallop & Sykes (1966)

The firmness and wholeness of canned grapefruit segments after different times and temperatures of heat processing are reported in Table 1 for cans after 30 days of storage. In the industrial trials the firmness of the segments decreased significantly ($P = 0{\cdot}05$), and in the pilot plant trials, decreased very significantly ($P = 0{\cdot}01$) as the length of time of pasteurization was increased. The quantity of broken segments and their tendency to break also increased correspondingly when either the time or the temperature of the heat treatment was increased. Segments canned in A2 cans were

TABLE 2. Effect of length and temperature of storage of canned grapefruit segments upon their drained weight, texture and colour

Temperature of storage (°C)	Length of storage (days)	Drained weight (g/can)	Firmness (S.P.U.)	Tendency to break (%)	Visual colour changes*
0	30	298	540	20	None
	180	320	630	25	None
	360	316	520	32	None
10	30	318	530	24	None
	180	320	690	21	None
	360	323	780	32	None
21	30	313	600	44	None
	180	309	580	26	*
	360	325	—	31	†
Room (15–29°C)	30	305	620	44	None
	180	308	590	26	*
	360	315	1020	35	‡
F-value	30	Significant (95% level)	Not significant	Highly significant (99% level)	
	180	N.S.	N.S.	H.S.	
	360	N.S.	H.S. (95% level)	N.S.	
LSD ($P = 0{\cdot}05$)	30	± 18·4	± 111	± 3·8	
	180	± 22	± 51	± 1·8	
	360	± 18	± 42	± 6·1	

* Slight browning. † Moderate browning. ‡ Strong browning.

found to require about 26 min at 80°C for proper preservation. Therefore, 400 ppm of sodium benzoate were added to all samples, so as to measure the physical effects of sub-lethal temperatures. The higher amount of broken segments found in these cans (heat treated for less than 26 min) may be due to incomplete enzyme inactivation.

Results of the tests on the effects of temperature and of length of storage on canned segments' quality are given in Table 2. Samples stored at 0°C were found to differ significantly in drained weight after 30 days of storage from samples stored at higher temperatures, possibly because at this temperature no equilibrium was reached between the sugar syrup and the segments. These differences disappeared after 180 and 360 days of storage, at which times no significant difference in drained weight existed among the four storage temperatures.

Samples stored at the different temperatures did not differ significantly in firmness after 30 or 180 days, but a highly significant difference was recorded after 360 days. The firmness of canned grapefruit segments stored at room temperature increased conspicuously, from 590 up to 1020 S.P.U., thus turning the segments from soft (nearly firm) to tough.

The temperature of storage also influenced the tendency of the segments to break. After 30 days of storage this tendency was much greater at higher temperatures, but no significant differences were found between the samples stored at 0°C and 10°C, and those stored at 21°C and room temperature (15–29°C).

After 360 days of storage, the samples had a greater tendency to break, but differences between samples stored at different temperatures were not significant.

The deterioration of quality which, according to Hugart *et al.* (1955), increases with increasing storage, was found to be pronounced in respect to changes in colour of the segments. The colour of the samples was always compared with the samples stored at 0°C. No marked changes were observed in parallel samples stored at 10°C for up to 360 days. Storage at room temperature and at 21°C did not affect the colour during the first months, while these samples darkened slightly after 180 days and markedly so after 360 days of storage. These results indicate the desirability of a lower storage temperature for canned grapefruit segments.

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Studies on the quality characteristics of canned grapefruit segments

II. Additives improving their drained weight and texture*

A. LEVI, ZDENKA SAMISH, A. LUDIN
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Summary. An increase in syrup concentration was found to lower the drained weight and to alter the firmness of canned grapefruit segments. Addition of $CaCl_2$ in the syrup improved the textural properties of the product; addition of low-methoxyl pectin increased the drained weight and improved the firmness and other textural properties of the canned grapefruit segments without influencing the taste or colour even after 180 days of storage.

Introduction

An attempt to improve the texture and the drained weight of canned grapefruit segments has been made, by adding calcium salts and low-methoxyl pectin (L.M.P.), to the covering syrup.

The middle lamela of the citrus segments, which, according to Webber & Batchelor (1948), is composed of pectic and celluloitic materials, is retained in part also in the ripe fruit. These compounds are able to imbibe and absorb water, passing it to the cells more rapidly than by the difference in osmotic pressure or diffusion processes. Canned fruits with a high pectin content, such as peaches and apricots, increase their weight during storage (Luh, Leonard & Mrak, 1959), the increase in weight probably caused, according to Sterling (1959), by the absorbing and imbibing polysaccharides. Luh *et al.* (1959) found that the drained weight of canned apricots increases with increasing sugar concentration of the syrup. Addition of L.M.P. to the sugar syrup, was reported by Sidwell & Cain (1953) and Board, Gallop & Sykes (1966) to increase the drained weight of canned berry fruits. According to Sidwell & Cain (1953), the addition of Ca salts to the canned fruits also improves their drained weight. Canned grapefruit segments, however, are known to decrease in drained weight and the addition of Ca salts may be expected to cause additional loss

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in drained weight, due to their low pectic content and the increase in difference in osmotic pressure between the sugar syrup and the segments, caused by the addition of Ca^{++} and Cl^{-} ions.

Materials and methods

Canned grapefruit segments were prepared in size A2 cans according to the technique described in the first paper of this series (Ludin *et al.*, 1969). This includes also a description of the method of measuring firmness, drained weight, the amount of broken segments and their tendency to break.

The strength of the sugar syrup was adjusted so as to reach, after equilibrium, a value of 20° Brix while in the experiment testing the influence of the sugar syrup concentration on the weight loss and the firmness of canned grapefruit segments the strength of the syrup was changed accordingly. Each can was filled with 420 g of fresh segments and 170 g of syrup.

$$\text{The loss in weight was expressed as percentage} = \frac{420 - \text{drained weight}}{420} \times 100.$$

In the experiment where $CaCl_2$ was added to the sugar syrup its content is expressed as percent $CaCl_2$ of the total net content of the can.

The low-methoxyl pectin (L.M.P.) added to the sugar syrup was obtained from Sunkist Growers Inc.—'Exchange' L.M.P. 100 gel power lot No. 3466. The L.M.P. content is given as percent of the sugar syrup added.

All cans were stored at 21°C and the tests were made after a storage period of not less than 30 days so that equilibrium would be reached in the T.S.S. content between the segments and the sugar syrup.

Results and discussion

Seventy-two A2 cans (for each treatment) of grapefruit segments were canned with different sugar syrup concentrations (Table 1) and processed under standard factory conditions.

TABLE 1. Effect of strength of syrup concentration upon the drained weight and the firmness of canned grapefruit segments

T.S.S. of syrup after equilibrium (degrees Brix)	Loss in weight (%)	Firmness (S.P.U.)
18·1	21·0	400
19·1	23·3	530
21·4	27·9	620
F-value	Highly significant (99% level)	Highly significant (99% level)
LSD ($P = 0{\cdot}05$)	±0·9	±80

After about 1 month of storage, laboratory tests were carried out on twenty-four cans taken at random from each batch. With increasing concentration of the syrup a highly significant loss in weight, as well as an increase in firmness of the segments were recorded due to the higher initial differences in osmotic pressure between the syrup and the grapefruit segments.

The effect of addition of calcium chloride upon the texture of the canned grapefruit segments is shown in Table 2. The firmness of the control sample measured 560 shear press units (S.P.U.). A panel judged the control samples as soft, those with 0·05–0·10% added $CaCl_2$ as firm, and those with 0·15% added $CaCl_2$ as tough. $CaCl_2$ additions reduced very significantly the amount of broken segments found in the can, as well as their tendency to break.

Dipping the fresh grapefruit segments in a bath of 1% $CaCl_2$ solution as well as the combined action of dipping in $CaCl_2$ and adding L.M.P. to the syrup, also affected favourably the textural properties of the canned segments (Table 2). The drained weight of the segments was not affected significantly.

TABLE 2. Effect of $CaCl_2$ on the drained weight and texture of canned grapefruit segments

	$CaCl_2$ added (% net can content)				Dip in 1% $CaCl_2$, 10 min		*F*-value	LSD (P=0·05)
	0	0·05	0·10	0·15	+0 L.M.P.	+0·10% L.M.P.		
Drained weight (g/can)	291	295	287	286	287	288	Not significant	±10·6
Firmness (S.P.U.)	560	780	670	1000	890	830	Highly significant (99% level)	±80
Broken segments (g/can)	37	21	26	18	15	17	Highly significant (99% level)	± 4·2
Tendency to break (%)	37	26	22	19	18	17	Highly significant (99% level)	± 2·3

The addition of L.M.P. to the syrup had a positive influence upon the texture as well as upon the drained weight of the canned grapefruit segments (Table 3). Twenty-four cans (out of seventy-two) from each treatment, taken at random, were tested after 39 days of storage. The effect of L.M.P. on the drained weight, the firmness, the broken segments and the tendency to break of the canned grapefruit segments was found to be affected very significantly (P = 0·01). However, it seems that there are no significant differences between the samples containing different quantities of L.M.P., and together with $CaCl_2$—except for drained weight, which seems to be affected negatively by the addition of $CaCl_2$.

TABLE 3. Effect of L.M.P. added to the syrup on the drained weight, texture and tendency to break of canned grapefruit segments

L.M.P. added (%)	Drained weight (g/can)	Firmness (S.P.U.)	Broken segments (g/can)	Tendency to break (%)
0	297	470	44	24
0·05	307	800	16	7
0·10	319	680	5	10
0·15	312	760	14	6
0·20	320	710	16	12
0·1 + 0·1 $CaCl_2$	292	710	9	6
F-value	Highly significant (99% level)	Highly significant (99% level)	Highly significant (99% level)	Highly significant (99% level)
LSD ($P=0·05$)	±9·3	±120	±12·1	±4·7

TABLE 4. Effect of L.M.P. added to the syrup on the drained weight of canned grapefruit segments

L.M.P. added (%)	Drained weight (g/can)
0·0	304
0·1	312
0·2	327
0·3	355
F-value	Highly significant (99% level)
LSD ($P = 0·05$)	±4·9

In later experiments carried out on 120 cans per treatment, with higher concentrations of L.M.P. to the syrup (Table 4), the drained weight was even more affected. The difference in drained weight between the control sample and the sample containing 0·3% of L.M.P. added, was about 17%. In these cans, the sugar syrup was found after storage to contain lumps of pectin–sugar gel.

No significant differences were recorded between cans containing L.M.P. or $CaCl_2$ stored either 30 or 180 days at 21°C.

A panel of judges found no taste or colour differences between the control sample and the samples containing the additives.

Acknowledgment

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An evaluation of the botulism hazard in vacuum packed smoked fish

G. HOBBS, D. C. CANN AND BARBARA B. WILSON

Summary. The potential hazard of botulism occurring from the consumption of vacuum packed smoked fish products available in the United Kingdom is discussed. Measurements of time and temperature during boil-in-the-bag cooking, and inactivation of toxin during this process, show that a boiling time in excess of 5 min is necessary to ensure that no toxin survives the cooking process. Current manufacturers specifications for cooking times are considered adequate for destruction of *Clostridium botulinum* type E toxin.

Introduction

Since the outbreaks of type E botulism in the U.S.A. during 1961–63 (Anon., 1963; 1964) due to the consumption of smoked fish, considerable attention has been paid to the association of *Clostridium botulinum* type E with this kind of product. Initially, it was thought that one reason for the occurrence of these outbreaks was that the fish had been vacuum packed. A direct result of this was the imposition of severe restrictions regarding the manufacture and sale of vacuum packed smoked fish in the U.S.A. (Anon, 1964) and Canada [Department of Fisheries and Food, and Drug Directorate, Section 55 (4)]. It is now known that vacuum packaging of itself only slightly increases the rate of toxin production in contaminated fish (Johannsen, 1961; Abrahamsson, de Silva & Molin, 1965; Torry Research Station, unpublished data) and, in fact, not all the smoked fish concerned in these outbreaks was vacuum packed.

Vacuum packaging of fish does slow down the rate of bacterial spoilage (Shewan & Hobbs, 1963) whilst it has slightly the opposite effect on growth and toxin production of *Cl. botulinum* type E. Presentation of fish in this way could lead to less careful handling since the product may be regarded as less perishable than unpackaged fish. A combination of such factors might lead to products contaminated with *Cl. botulinum* becoming toxic before they reach a stage of overt spoilage when they would be discarded.

Before an outbreak of botulism can occur the fish must be contaminated at some stage with the causative organism. From the available evidence it is clear that fish caught in many parts of the world are contaminated before they are caught. *Cl. botulinum* type E occurs in freshly caught fish to a varying degree in different parts of the world. Its occurrence in the U.S.A., Canada, Japan and Russia has been known for

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some time (Ando & Inoue, 1957; Dolman, 1960; Dolman & Iida, 1963). More recently work in the U.S.A. has shown a widespread distribution in both freshwater and marine fish (Chapman & Naylor, 1966; Bott, Deffner & Foster, 1967; Ward *et al.*, 1967; Craig & Pilcher, 1967; Nickerson *et al.*, 1967; Pace *et al.*, 1967). An unusually high incidence has been reported in Swedish coastal waters and fish by Johannsen (1965a) and this was confirmed by Cann *et al.* (1965, 1967a). A very low incidence was found in British coastal waters and fish on sale in Britain (Cann *et al.*, 1966; Cann, Wilson & Hobbs, 1968).

In addition to type E, non-proteolytic type B and F strains have been reported in sediment samples collected off the Pacific coast of the U.S.A. and off the British coast (Craig & Pilcher, 1966; Eklund, Wieler & Poysky, 1967; Cann *et al.*, 1968). Whilst neither of these have so far been implicated in an outbreak of botulism from fish, the occurrence of these strains must be considered a potential danger since they have growth properties similar to type E.

Since botulism results from the ingestion of preformed toxin, the fish must support growth of the organism and must be stored under conditions of time and temperature which permit toxin production. There is evidence that raw fish products will support growth and significant toxin production by *Cl. botulinum* type E at temperatures as low as 5°C (Cann *et al.*, 1965b, 1967b).

In the case of smoked fish either the toxin must survive the smoking process, or the smoked fish must support growth and toxin production.

With cold smoked products the process involves exposing the fish to a temperature of 30°C for 3–4 hr in a modern smoking kiln. This process will, if anything, encourage growth and toxin production. The hot smoking process involves exposure of the fish to 82°C for ½–2 hr and this could inactivate preformed toxin but does not necessarily kill all spores present. In both processes, however, there remains the problem of recontamination after smoking.

In addition to temperature, the presence of curing agents will also influence growth and toxin production. A concentration of 5% w/w sodium chloride has been shown to be necessary to prevent outgrowth of *Cl. botulinum* type E spores (Pedersen, 1957; Segner, Schmidt & Boltz, 1966). In smoked fish, where other curing agents are also present, a brine concentration of 3% is reported to be sufficient (Christiansen *et al.*, 1968).

In Britain, two types of smoked fish are sold as 'boil-in-the-bag' products. These are kippers and smoked haddock, both produced by a cold smoke process. There is evidence that, once contaminated with *Cl. botulinum* type E, toxin will develop in both these products after storage at a suitable temperature (Cann *et al.*, 1965b).

One of the advantages of 'boil-in-the-bag' vacuum packed fish is the ease of cooking in the pack by immersing in boiling water and this type of product is increasing in popularity. If there is a potential danger that such fish contain botulinum toxin, it is important that the cooking procedures recommended should be sufficient to destroy

this toxin. In the present investigation data is presented to show the boiling time required to destroy any preformed toxin in 'boil-in-the-bag' products.

Materials and methods

Chemical analyses

Fish were bought as frozen products from retail shops. The size and weight of each pack were recorded. Salt concentrations were estimated by Volhard's method and water contents by the method of Dean and Starke. In a few cases pH measurements were made on representative fish.

'Boil-in-the-bag' tests

These were performed on both retail frozen vacuum packed kippers and 100 g fillets vacuum packed at Torry. Each pack was inoculated with 10^6 viable spores of *Cl. botulinum* type E (strain FT8 isolated by the authors from North Sea herring). The method of vacuum packing and inoculation was that of Cann *et al.* (1965b). The fish were then incubated for 6 days at 30°C to produce toxin.

Boiling tests were carried out by immersing the packs of fish in an average sized domestic pan of water and cooking for varying times after the water was brought back to the boil. Temperatures at the centre of the packs were measured either with a Leeds and Northrup potentiometer adapted for use with a microthermocouple or with a microthermocouple attached to a Honeywell recorder. At least 12 cm of the microthermocouple was inserted longitudinally into the fillet to eliminate errors in readings due to thermal leakage along the thermocouple wire.

Toxin assay

Each sample was macerated with three times its weight of physiological saline and centrifuged for 10 min at 3000 rev/min. A comparison of the toxicity of trypsinized and non-trypsinized macerates was made. The procedure for trypsinization was that described by Cann *et al.* (1965b). Toxin titres were determined using doubling dilutions of macerate. Two mice were used at each dilution and observed for 48 hr.

Results

Previously published data on the relationship between toxin production, inoculum level and storage temperature for herring, kippers and smoked haddock are summarized in Table 1 (from Hobbs, 1967).

Salt and water contents of samples of kippers are presented in Table 2. The pH values found ranged from 6·15 to 7·3 with an average of 6·56.

Temperature measurements were made on herring, kippers and smoked haddock during immersion in boiling water. The fish were at varying temperatures before immersion. Fig. 1 summarizes the temperature measurements and shows the range of time and temperature relationships. Results of toxin tests on kippers are presented in Table 3.

TABLE 1. Some relationships between estimated shelf life of fish and toxin production by *Cl. botulinum* type E (inoculum: 10^5–10^7 spores/g)

	20°C (68°F)		10°C (50°F)		5°C (41°F)	
	Shelf life (days)	Toxin (days)	Shelf life (days)	Toxin (days)	Shelf life (days)	Toxin (days)
Herring	< 1	1–5	1–2	4–7	3–4	12–32
Kippers	5–6	2–8	6–9	8–20	7–10	> 28
Smoked haddock	2–5	3	5–8	28–30	10–12	> 28

TABLE 2. Salt and water contents of vacuum packed kippers

Manufacturer	No. of samples	Salt as % brine		% water	
		Range	Average	Range	Average
A	66	1·19–7·67	2·86	35–82	61·26
B	23	1·21–4·92	3·04	40–79	65·93
C	6	3·24–4·93	4·26	46–66	56
D	8	1·69–3·63	2·62	66–78	73·56
E	1	—	2·20	—	65
F	5	2·38–4·71	3·48	57·5–80·5	71·10
G	4	1·94–4·17	2·65	56–72·5	64·75
J	5	2·45–3·60	2·89	60·5–71·5	66·10
L	1	—	2·10	—	68
M	1	—	3·62	—	73·5
O	1	—	2·90	—	69
Q	2	4·76–4·83	4·80	63–69	66

TABLE 3. Destruction of toxin in kippers using the 'boil-in-the-bag' method of cooking

Boiling time (min)	Average final temperature (°C) (six samples)	Toxin strength (M.L.D./g)		
		Control sample		Test samples (six) after boiling
		Non-trypsinized	Trypsinized	
5	81·95	44	442	nil
7	91·6	553	88	nil
10	96·4	442	221	nil
12	95·7	885	44	nil
15	96·9	55	442	nil

The effect of trypsinization on toxicity varied in individual packs. Toxin titres were usually enhanced by trypsinization, but where proteolytic spoilage has occurred to any extent, inactivation of toxin may occur.

None of the fish tested were toxic after boiling for a minimum of 5 min, that is 5 min after the water returned to the boil.

Discussion

From the data available it is known that *Cl. botulinum* type E is present in vacuum packed smoked fish on sale in Britain, although the incidence is low. It is evident from our own work that much of the smoked fish on sale in Britain will support growth and toxin production, although we have data (unpublished) to show that this does not apply to all commercially available kippers. Salt concentration in commercially produced smoked fish is variable and, indeed, is difficult to control within fine limits. If salt is to be relied upon to prevent toxin production, any recommendations must take account of these variations, and the specification must be such that none of the fish has a salt content lower than 5% (w/w). This is clearly not achieved in commercially produced kippers, nor is it desirable from the point of view of palatability.

There is no opportunity for toxin to develop in frozen products. This can only occur if the fish is allowed to thaw out and is stored for some time in the unfrozen state. Freezing and cold storage will not inactivate any preformed toxin, nor will it kill a significant number of *Cl. botulinum* spores.

The results show that in none of the tests did any toxin remain after 5 min boiling. The total immersion time varied, depending on the size and initial temperature of the fish. The longest immersion time observed was $12\frac{1}{2}$ min, for fish at an initial temperature of -20°C.

The cooking experiments presented are not strictly comparable with other published data on heat inactivation of this toxin, since in our experiments the temperature was rising throughout. However, exposure to 65°C for 5 min as quoted by Abrahamsson, Gullmar & Molin (1966) is similar to the time temperature relationships found in the present experiments.

From the temperature measurements (Fig. 1) it is apparent that a small proportion of packs, the thicker ones, require more than 5 min boiling to achieve the heat treatment quoted by Abrahamsson *et al.* (1966). For a cooking specification to include a reasonable safety margin, a boiling time somewhat in excess of 5 min is, therefore, necessary. The majority of manufacturers specifications do now meet this requirement.

The situation, therefore, is that an outbreak of botulism could occur from 'boil-in-the-bag' smoked fish only if gross mishandling has occurred. The majority of such products in Britain are sold in the frozen state; therefore, the thawed out product would have to be stored above 5°C for an appreciable length of time, and then consumed without adequate cooking. It is, perhaps, significant that precisely such an

unlikely combination of circumstance has led to many outbreaks of botulism in the past.

One further situation which is worthy of comment is the increasing consumption of smoked fish without any further cooking. Products such as vacuum packed kipper fillets and smoked salmon consumed in this way represent the greatest potential danger.

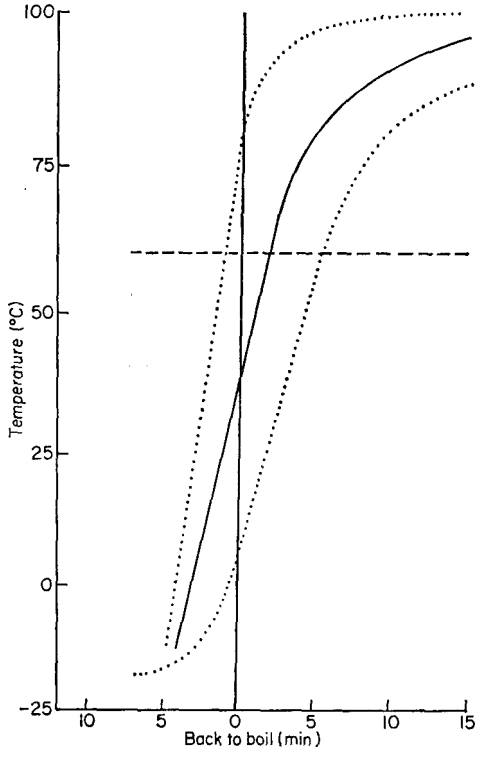

FIG. 1. Temperature measurement of fish during 'boil-in-the-bag' treatment. ———, Average temperature; . . . . ., range of temperature; - - -, thermal destruction of toxin after 5 min treatment.

Acknowledgment

The work described in this paper was carried out as part of the programme of the Torry Research Station. Crown Copyright reserved.

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Book Reviews

Surface-Active Lipids in Foods. S.C.I. Monograph No. 32.
London Society of Chemical Industry, 1968. Pp. 173. £2 10s. plus postage.

This is a small volume reproducing the papers given during a 2-day Joint Symposium, organized by the Food Group and the Oils and Fats Group of the Society, held in London in March 1968. The eleven papers are given in full. The discussions at the end of each of the four sessions have been competently summarized.

The first session included papers on molecular aggregation in phospholipid dispersions, structures of emulsifier–water phases, and the effects of emulsifiers on the crystallization of triglycerides. The discussion was mostly concerned with the effects of ultrasonication on structures, and on the nature of micelles. The effects of adding soap to monoglyceride–water systems were explained.

The second session dealt with properties of membrane lipids in model systems, the structure and formation of plant lipids particularly those containing acyl groups, and the mechanism of promotion of emulsification. The discussion turned on membrane permeability in bacteria and the effects on this of the temperature at which organisms were grown; the role of phospholipids in the transfer of fatty acids from the diet; the function of lipoxidase in the synthesis of traumatic acid; the biosynthesis of fats and waxes; the rigidity of elongated cylinders of oil in an emulsion; the influence of macromolecules such as gum acacia; and possible mechanisms in the formation or coalescence of emulsion drops.

The third session considered papers on surface-active lipids in milk and milk products, the lipoproteins and lipids of egg yolk, and surface active lipids in chocolate. The discussions which followed touched on the differences between the milk of ruminants and of monogastric animals; the fatty acid composition of egg lecithin; the methods by which lecithin modified the character of chocolate; effects of freeze-drying on whole hen egg; the effect of the diet of hens on the baking quality of their eggs; the wetting of milk powder particles; the synthesis of milk fat globules; the capacity of biological systems to control the physical properties of their surface-active lipids; and the effects of the increased surface of fat globules when milk is homogenized.

The fourth session dealt with surfactants in baked foods and the importance of the physical state in the application of fat-derived emulsifiers. In the discussion details were given of X-ray diffraction patterns of gels and coagels. It was noted that the iodine values of commercial monoglycerides were at variance with their expected structure.

There are over 300 references. Figures and tables are clearly legible and adequately captioned. This is a worthy addition to the Society's Monograph Series.

K. Bryce Jones

Biochemical and Biological Engineering Science. Vol. 2. Ed. by N. BLAKEBROUGH.
London and New York: Academic Press, 1968. Pp. 380 + xii. £5 5s.

This book on food engineering is the second half of a study of biological engineering, the first half being devoted to fermentation and related processes. It considers the properties of vegetable and animal materials, their preservation by drying, freezing and heat treatment and the engineering aspects of these processes. Finally, processing with ionizating radiations is considered.

In the sense that it tries to explain food science to the engineer and engineering to the food scientist, the book fails. As a food scientist, the reviewer found much of the food science too elementary to be useful and the engineering too advanced to be fully understood. It seems likely that the professional engineer will meet the same situation but in reverse. Nevertheless, it is a gallant attempt at an almost impossible task and is packed with useful information as well as containing a number of chapters of quite outstanding quality by well-known authorities.

As a book of general reference, it will be useful to postgraduate students in chemical engineering and food science and food technology, as well as to research and development departments in industry. The book is well illustrated, well indexed and remarkably free from printing errors. Its value is greatly enhanced by the excellent bibliography at the end of each chapter. Indeed, in a sense the serious student will find that a critical use of the bibliography goes a long way to meeting my general criticism that the book tends to fall between two stools.

JOHN HAWTHORN

Principles of Food Science. By GEORG BORGSTROM.
London: Collier-Macmillan, 1968. Vol. 1, pp. 397 + xii, £6; Vol. 2, pp. 473 + xi, £6.

Professor Borgstrom is an internationally-known food scientist who has brought distinction to the Department of Food Science at Michigan State University. The publication by him of a comprehensive general text on food science and technology is, therefore, an event of importance in a field which has only comparatively recently gained international acceptance as a scientific discipline in its own right. While there have been a number of general books dealing with the subject at various levels this is the first serious attempt to produce a text covering the whole field at about University Honours Degree level. As one would expect from an author of Dr Borgstrom's repute, the books are written with authority and represent one of the most important disciplinary contributions to the subject of food science to be made during the past twenty years. These two volumes will become an essential tool on the shelf of every food science library of repute.

The biggest intellectual problems facing the university teacher of food science is the

organization of his material, and those who have to attempt to impart this material systematically to students are faced with the major problem of deciding on the most logical approach to the subject. Teachers of food science often look at other subjects such as organic chemistry with a certain sense of envy. Since 1830 and piece by piece the jigsaw puzzle of organic molecular structure has been assembled and now the subject can be presented to the student systematically and logically. The whole building is divided into rooms of convenient sizes, each room connecting with its neighbour and after even quite a short period of study, the student having been led by the hand, as it were, from room to room can then walk round the outside of the building to see the whole structure in the grandeur of its concept and in the integrity of its inter-relationships.

The teacher of food science is in a very different position. His subject is more like the scattered units of a temporary army camp of vast size but still in the course of construction. The student is, therefore, faced with a rather shapeless structure with many uncompleted rooms and parts of the network of connecting roads under hasty and improvised repair. Through this maze, Borgstrom has chosen one particular pathway which has its own logic but in which the student must necessarily recross from time to time ground which he has briefly scanned on a previous occasion. In his journeyings he has been shown buildings with the scaffolding still in position and workmen busily engaged at little more than ground level. In others, he has seen completed structures.

If there must be criticism of these two volumes then it will lie more in the direction of the arrangement of the material, of the relative importance given to various subdivisions within the main subject and to the sequence in which topics are introduced. At the moment there is no sign of general agreement amongst professional food scientists as to how this should best be done. Until a consensus is reached there will always be criticism of any attempt to resolve the problems of presentation. There can be no denying that this is an important approach to the resolution of this dilemma. That the approach does not happen to coincide with this reviewer's opinion in no way detracts from the practical usefulness of these two volumes. For years to come they will be the essential part of the equipment for both student and teacher in this subject.

However, it must not be thought these volumes are for university use and that alone. They provide a reference text of value for every food processing or research laboratory. They merit the attention of public health authorities in all countries and at all levels. They will widen the eyes of receptive scientists from other disciplines, willing to explore unusual territory.

The collection of material upon which the work is based was begun 28 years ago. The preparation and writing took fifteen years. The result is a virtuoso display of erudition combined with the sustained effort of a dedicated enthusiast for his subject. From the viewpoint of the serious student, each chapter is supplemented with a comprehensive bibliography which enhances the value of the text. Despite the

long period of preparation and the ever-accelerating availability of new knowledge in recent years, the text is as up-to-date as the limitations of printing permit.

In a work as comprehensive as this it is not surprising to find the occasional loosely-written sentence which does not convey the author's intention. Nor are the tables and diagrams always as accurately labelled as one would wish. However, these are minor points which will doubtless be corrected in the successive editions through which the work will assuredly run. At £6 each these volumes are not cheap. They can be regarded as a capital investment.

JOHN HAWTHORN

Food and Society. By MAGNUS PYKE.
London: Murray, 1968. Pp. 178. £1 10*s*.

Dr Pyke is a writer whose books are never difficult to read. Here he takes nutrition – a subject on which he is well qualified to speak – as his theme and sets the modern science of nutrition, in a provocative and challenging manner, against the sociological background of the world in which nutritionists must operate. His opening chapter surveys the historical development of nutrition and states the dominant theme of the book – that what we eat is affected largely by the psychological conditioning to which we have been and are being submitted. This theme is extended in the subsequent chapter, which discusses the lack of scientific support for many food 'fads', old and new – while warning the reader not to disregard these without due consideration. Love potions, voodoo, the Doctrine of Signatures and the effects of custom and religion in controlling the eating habits of people throughout the world are then considered (even the lowly 'small port' does not escape – but we may take comfort that the restorative effect of a glass of whisky is, by omission, left unchallenged). The author emphasizes the important part played by these esoteric influences in achieving that 'state of complete physical, mental and social well-being' which the W.H.O. defines health to be. Both here, and in the final chapters which survey the part played by social, environmental and financial stress in the attainment of health, the author raises many important questions – often without attempting to provide solutions. As is pointed out in Chapter V, the adoption of a particular course of action implies the adoption of a philosophy of life which in turn arises not so much from scientific analysis as from religious faith (using the term in its widest sense). As a scientist, Dr Pyke can conclude from the evidence that 'man does not live by bread alone', but equally, on purely scientific grounds, he is unable to complete the quotation.

This wide-ranging book also includes a refreshing discussion of the 'safety' of foods, a chapter dealing with the hazards facing the Codex Alimentarius and others discussing the dangers of incomplete knowledge, self-deception and self-interest on the part of nutritional scientists. Throughout the book, the author illustrates his argument with examples that are invariably interesting and frequently entertaining, with page-foot references to inform the reader of his sources. This book should find a wide readership,

from students and teachers, from practising nutritionists, who will find much food for thought, and even from the eating public, who might benefit considerably—if only by collecting a few excuses for eating what they enjoy.

N. D. COWELL and A. E. V. LILLY

Books received

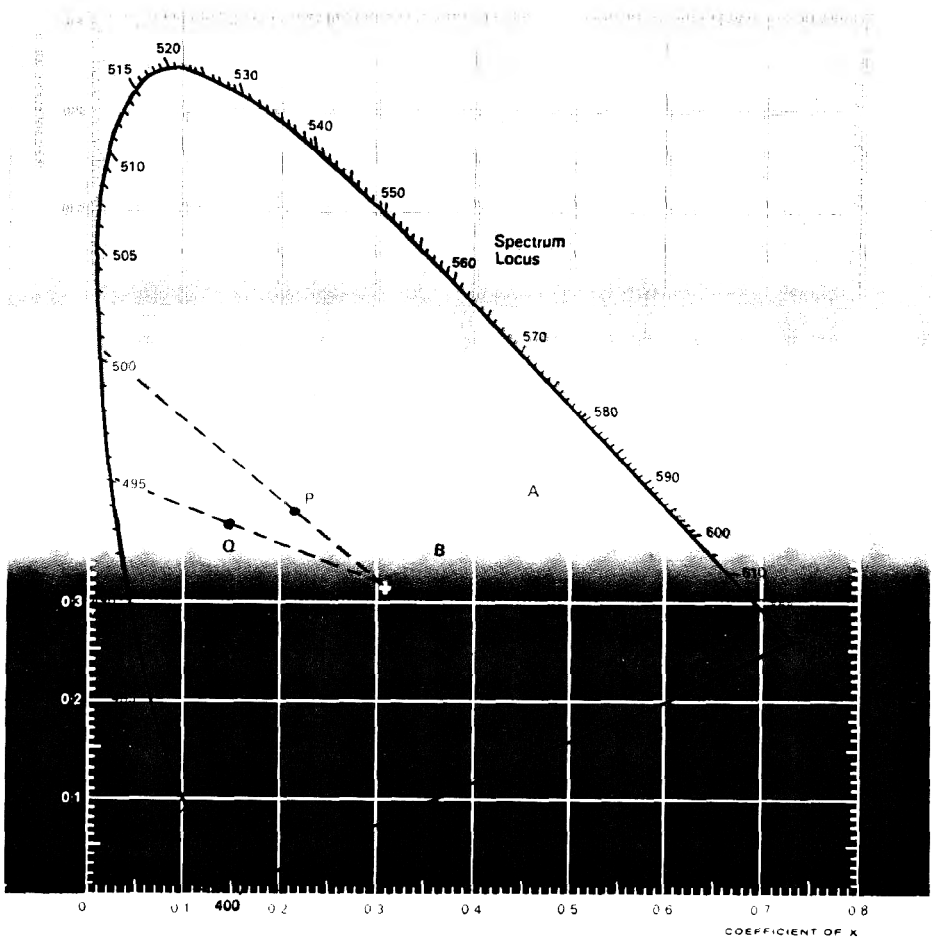
Freeze Drying of Foods and Biologicals. By ROBERT NOYES.
U.S.A.: Noyes Development Corp. Pp. 311. 1965. $35.

An Introduction to Industrial Mycology, 6th edn. By GEORGE SMITH.
London: Edward Arnold. Pp. x + 375. 1969. £4.

Beeinflussung des Stoffwechsels durch die Ernahrung. By J. C. SOMOGYI and H. D. CREMER.
Basel: Karger, Pp. 172. 1969. £6.

Dehydration Processes for Convenience Foods. By ROBERT NOYES.
U.S.A.: Noyes Development Corporation. Pp. 365. 1969. $35.

Food Guide to Europe, 1969. ANON.
U.S.A.: Noyes Development Corporation. Pp. 130. 1969. $20.



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

Abbreviations. Abbreviations for some of the commoner units are given below. The abbreviation for the plural of a unit is the same as that for the singular unless confusion is likely to arise.

gram(s)	g	second(s)	sec
kilogram(s)	kg	cubic millimetre(s)	mm^3
milligram(s)		millimetre(s)	mm
$(10^{-3}g)$	mg	centimetre(s)	cm
microgram(s)		litre(s)	l
$(10^{-6}g)$	µg	millilitre(s)	ml
nanogram(s)		pound(s)	lb
$(10^{-9}g)$	ng	gallon(s)	gal
hour(s)	hr	milliequivalent	mEq
minute(s)	min	R_F values	R_F

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

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

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