ISSN 0022-1163

Volume 17 Number 4 August 1982

Journal of Food Technology

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

JOURNAL OF FOOD TECHNOLOGY

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

Objects of the Journal. The Journal covers a wide field ranging from pure research in various sciences associated with food to practical experiments designed to improve technical processes. While the main object is to provide a forum for papers describing the results of original research, review articles are also included. The Editor welcomes manuscripts of original research or comprehensive reviews of specialized sections of food science or technology.

Contributions and editorial correspondence should be sent to Dr H. Liebmann, c/o Institute of Food Science and Technology (U.K.), 105–111 Euston Street, London NW1 2ED.

General correspondence should be sent to Dr P. Wix, Polytechnic of the South Bank, Borough Road, London SE1 0AA.

Business matters, including correspondence and remittances relating to subscriptions, back numbers, advertising and offprints, should be sent to the publishers: Blackwell Scientific Publications Ltd, Osney Mead, Oxford OX2 0EL.

The Journal of Food Technology is published bimonthly, six issues form one volume. The annual subscription is £68.00 (U.K.), £82.00 (Overseas), $195.00 (N. America, including cost of airfreight). Copies of the journal are dispatched by airfreight to New York for forwarding by surface mail throughout the Western Hemisphere, and by airfreight to Singapore for forwarding by surface mail to subscribers in India, Australasia and the Far East. Back volumes are still available.

U.S. Mailing Agent, Expediters of the Printed Word Ltd, 527 Madison Avenue, Suite 1217, New York, NY 10022. Second class postage paid at New York, NY.

J. Fd Technol. (1982) **17**, 417–430

Circulation cleaning of a plate heat exchanger fouled by tomato juice

I. Cleaning with water

C. S. CHEOW* AND A. T. JACKSON†

Summary

A diluted tomato paste, applied as evenly as possible to one side of plate heat exchanger plates, has been used to study the effect of three temperatures (20, 70 and 90°C) when using water as a circulation cleaning fluid. From the experimental results it would appear that 70°C is the optimum temperature for this duty. Increasing the temperature to 90°C caused protein denaturation of the deposited tomato soil and cleaning efficiency was reduced. The soiling technique used in this work was found to be simple and consistent in studying the cleaning-in-place of the plate heat exchanger, and the direct weighing method of measuring soil removal was found to be reproducible.

Introduction

CIP cleaning techniques use empirically determined time cycles for each phase of the cleaning operation. Considerable time, detergent and energy might be saved if a clear understanding of the principles involved, and a knowledge of the effect of certain variables were to be determined. Since evaluation of these variables requires the cleaning process to be stopped short of completion, processing plants are not well suited for such studies. The present work is directed to a better understanding of the effect of different process variables on circulation cleaning of tomato soil deposited on a laboratory plate heat exchanger.

Soil formed during food processing operations includes residues of one or more of the original ingredients together with deposited minerals, and is frequently impregnated with food particles (Abele, 1965). Morgan & Wassermann (1959) and Gordon, Hankinson & Carver (1968) found that soils from

Authors' addresses: * Mara Institute of Technology, Malaysia, and † Department of Chemical Engineering, University of Leeds, Leeds LS2 9JT.

† To whom correspondence should be addressed.

0022–1163/82/0800–0417 $02.00

Table 1. Food soil characteristics

Component on surface	Solubility	Ease of removal	Change on heating
Sugar	Water soluble	Easy	Caramelization: more difficult to clean
Fats	Water insoluble, alkali soluble	Difficult	Polymerization: more difficult to clean
Protein	Water insoluble, alkali soluble, slightly acid soluble	Very difficult	Denaturation: much more difficult to clean
Mineral salts	Water solubility variable, most are acid soluble	Easy to difficult	Unless interacting with other components generally easy to clean

grape juice, tomato juice and milk contain all components of the original fluid, but the distribution of components was markedly different. The soils were higher in protein and lower in moisture content than the original fluid, and Morgan & Wassermann found that the microscopic appearance of tomato soil was that of clotted tomato with unclotted juice adhering to the denser, sticky soiling film.

The mechanisms involved in cleaning soiled equipment are complicated and interrelated, and Jennings (1963, 1964) has extensively reviewed the literature in this field for both homogeneous and heterogeneous soil systems. Food soil constituents vary in their solubility characteristics and in their susceptibility to cleaning, the main characteristics being shown in Table 1 (Harper, 1974).

Food processors, equipment manufacturers and detergent producers have recognized a number of factors which influence the cleaning process, e.g. temperature, concentration, flow rate and time. Each of these can be varied independently to adjust the cleaning operation for a particular problem or plant operating practice. Optimization for cost-effective cleaning is complex due to the complex relationships existing between soil loads, time available, possible maximum flow rate, etc. (Tamplin, 1980). Considerable work has been carried out to establish the relative importance of these factors both in the laboratory and on pilot and full-scale plant.

Effect of temperature

Conflicting opinions exist regarding the effect of temperature on soil removal. Some workers imply that high cleaning temperatures encourage the 'burning-on' of residual soils, whilst others argue that higher temperatures yield better cleaning. Increasing the temperature of the cleaning fluid has been shown by Harper (1974) to have the effect of decreasing the soil–surface bond strength, decreasing viscosity (hence increasing turbulent action), increasing solubility of

materials and increasing chemical reaction rates. Below 35°C fat remains in a solid state and above 85°C heat-induced interactions take place, binding the protein more tightly to the surface and decreasing cleaning efficiency. For any food soil, the minimum effective temperature will be about 3°C above the melting point of fat in the soil. The maximum temperature will depend on the temperature at which the protein in the soil is denatured. Recommendations made by detergent and equipment manufacturers range from 40 to 90°C.

Effect of turbulence

In every case of cleaning, energy must be supplied to effect the final displacement of the soil deposit. For many years, a bristle brush has been the means of supplying this energy. In circulation and spray cleaning, this traditional energy source has been replaced by friction between deposited soil and the fluid flowing past it, the shear force, thus generated, being related to the turbulence of the fluid (Jennings, 1963). Timperley & Lawson (1980) showed that the same degree of cleaning of pipes of different diameters is obtained if the same mean flow velocity is used. Based on their results, the level of residual micro-organisms on the pipe surface is reduced to a minimum if the velocity of flow is about 1.5 m/sec. This conflicts with the work of Jennings, McKillop & Luick (1957), who stated that the Reynolds number of flow is a better criterion.

Effect of age

Cleaning recommendations always emphasize that surfaces should be cleaned as soon after soiling as possible. Smith (1969) pointed out that unheated sugar and fruit soils can be easily cleaned, provided they have not been allowed to dry. The ageing effect of oil-containing soils in the cleaning of textile fibres is well known (Untermohlen & Wallace, 1947; Durham, 1961). Bourne & Jennings (1961, 1963) showed that tristearin, deposited on stainless steel, exists in two chemically identical forms that are removed at different rates, and that the ageing effect involves transition of the fast-removal soil species to the more slowly removed form. Similar observations have been reported regarding milk soils (Burton, 1964).

Evaluation of cleanliness

Evaluation of cleaning procedures have been limited by the difficulty of determining the degree of soil removal. Various procedures and criteria to evaluate equipment cleanliness, and estimating of soil deposits have been developed to meet this demand. Advantages are claimed for each technique, but each has its limitations. Jennings (1964) has commented that there will probably never be a universally acceptable means of measuring cleaning (detergent) efficiency.

Methods which have been used for the evaluation of cleanliness and detergent efficiency include visual inspection (Armbruster, 1962), weighing (Hankinson

& Carver, 1968), turbidimetric methods (Maxcy & Shahani, 1961), bacteriological methods (Baldock, 1974) and the use of radioactive isotopes (Armbruster & Ridenour, 1952).

Soiling techniques

In order to evaluate cleaning performance, a standard soiling process needs to be established. The techniques of forming fouls and soil films for investigation of cleaning fall into two groups, (a) deposition of soil on equipment surfaces through normal routine processing, and (b) deliberate deposition of a soiling film on a test plate or disc. The first group represents conditions of fouling which occur in an operating plant, but the main disadvantage of this technique for laboratory investigation is that it can often take several days of operation to obtain a satisfactory film for study. The second group is the most popular for use in the laboratory since it is possible through careful technique to obtain a soil film which is consistent in terms of adequate soil thickness and tenacity.

Experimental cleaning circuit

The experimental rig was designed to enable water or detergent circulation cleaning of the heat exchanger. A schematic diagram of the cleaning circuit is

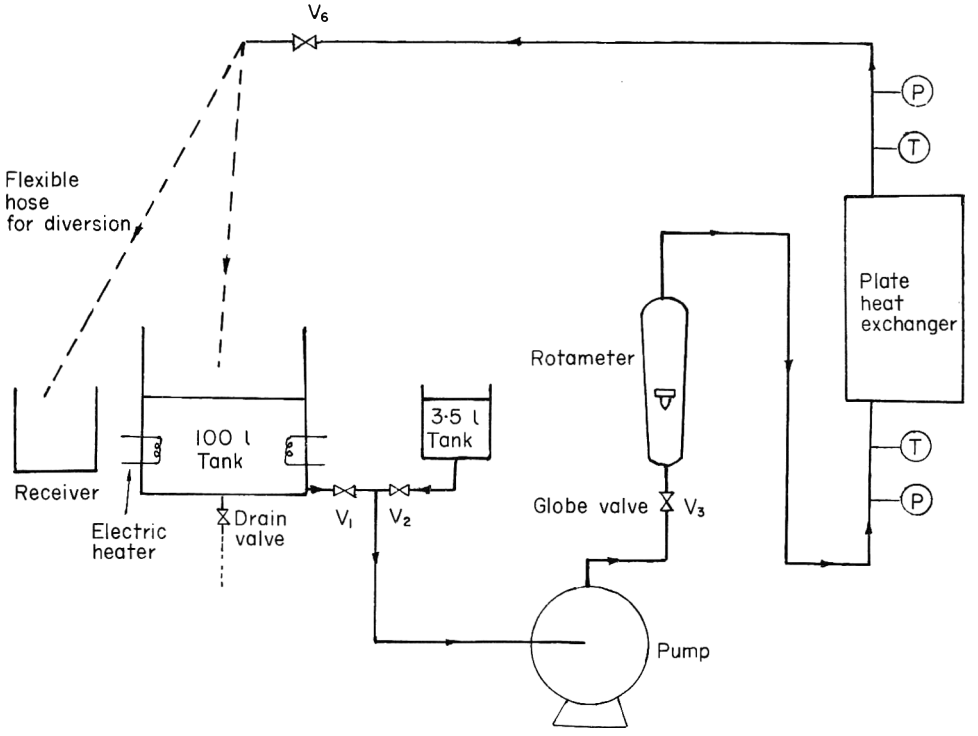


Figure 1. Schematic diagram of cleaning circuit. T = thermometer; P = pressure gauge.

shown in Fig. 1. The circuit consists of two feed tanks (100 l steel and 3.5 l plastic) connected together via a 6.0 mm bore stainless steel tee and valves V_1 and V_2 (both full bore stainless steel ball valves). The tee is connected to a Stuart Turner No. 12 centrifugal pump, the pump outlet being connected to the plate heat exchanger inlet via a Rotameter complete with stainless steel globe valve. The liquid leaving the heat exchanger can be discharged, through the flexible hose, back into the feed tank or into a separate receiver. Valve V_6 (stainless steel ball valve) can be used to increase the total back-pressure on the system. The liquid in the 100 l feed tank can be heated using two individually controlled 3 kW heaters, and this tank was intended to be used for the cleaning fluid. The 3.5 l feed tank is used for rinse water. The 100 l tank on installation was insulated with 50 mm thick polystyrene blocks.

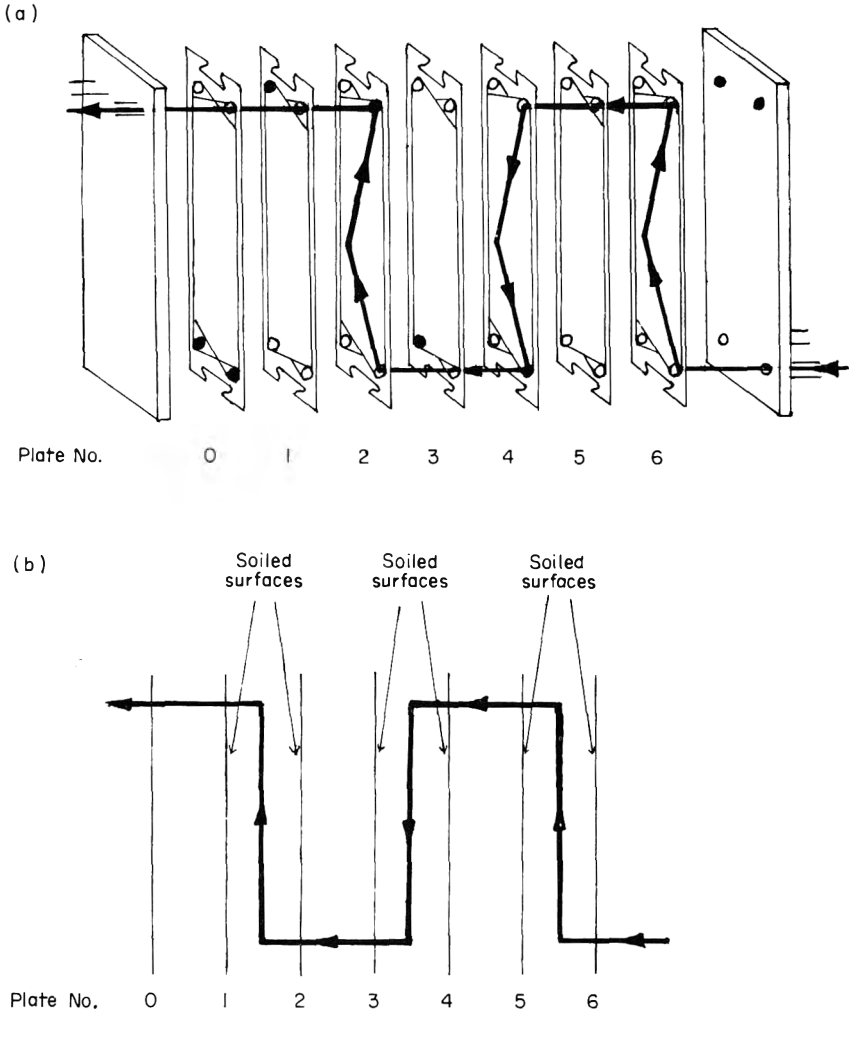


Figure 2a & b. Flow diagrams of cleaning solution.

The plate heat exchanger

The plate heat exchanger was supplied by the APV Co. Ltd, Crawley, and is a Junior Paraflow complete with thermometer fittings and pressure gauges. The plate arrangement is a series flow pattern with three passes per passage for both the service (heating/cooling) and product sides. The free-standing model unit consists of seven plates. Two sets of seven plates were used in this experimental work, so that it was possible to carry out two experimental runs per day. The plates were plates which had been used on other duties previously. Schematic flow diagrams for the cleaning solution passage through the heat exchanger are shown in Figs 2a and b.

In order to simulate flow velocities recommended for circulation cleaning of full-scale plate heat exchangers (typically APV type HMB) a flowrate of 109 l/hr is recommended for the Junior Paraflow (two times the minimum working flow rate) (Daoud, 1980). A flow rate of 2.0 l/min used for circulation cleaning with water on the experimental rig gives the following characteristics;

Flow velocity	0.304 m/sec
Shear rate	898 sec^{-1}
Reynolds number	3000

Materials and methods

Soiling operation

A 28–30% total solid content, double concentrate tomato paste was supplied in 5.0 kg tins by Libby, McNeill & Libby Ltd, Milnthorpe. Various combinations of tomato paste and distilled water were tested for adherence and consistency, and it was found that equal weights of tomato paste and water gave easy application on the plate by brush, and adherence of the juice during drying. Each individual plate was weighed before every experiment to check for accumulation of deposits or loss of gasket and plate materials during cleaning and scrubbing operations.

Tomato paste (100 g) and distilled water (100 ml) were mixed with a plastic spatula in a 25 ml beaker until a consistent composition was obtained. A 10 ml plastic syringe was used to transfer the juice to each plate surface, the plates being laid horizontally on the bench. The diluted paste was spread evenly over the whole corrugated surface of the plate using a 25 mm paint brush. The paint brush was pre-soaked with 2.0 ml of the diluted paste before using it to paint the first plate. The brush was used without washing for the rest of the plate surfaces. This technique was found to give uniform weights of soil on the six plates.

The plates, with the painted surfaces facing upwards, were placed in a pre-heated laboratory oven set at 112°C and dried for 45 min. The time of drying was chosen to give a consistent weight of soil without over-heating the tomato film. The plates were allowed to cool to room temperature before weighing.

Cleaning operation

The soiled plates were assembled in position with both sides of the three passes of cleaning fluid having the dried-on layers of tomato soil on their surfaces (Fig. 2).

The water used for cleaning was tap water (hardness 100–110 p.p.m.). The 100 l feed tank was half filled with water and heated to 2–5°C above the required temperature of cleaning to allow for heat loss through the pipework, header and follower of the plate heat exchanger. The water temperature was measured using mercury in glass thermometers. The system was warmed up prior to each experiment by circulating hot water for 10–15 min using unsoiled plates in the heat exchanger. The Rotameter was calibrated over the range of temperatures used by collecting the liquid in a vessel over a timed period.

For each experiment using the soiled plates, the heat exchanger was dismantled after a fixed circulation time and the plates placed in the drying oven at 112°C for 45 min. After drying the plates were cooled to room temperature and then re-weighed. The residual soil remaining on the plates after drying and weighing was completely removed using a wedge-edged plastic tube and sharp-edged plastic spatula under running hot water. This method of cleaning was adopted in order to avoid the possibility of scratching the surfaces of the plates, thus possibly affecting the adherence of the soiling film.

In all the experiments, fresh water was used for each cleaning run.

Results and discussion

The experimental results for water temperatures of 20, 70 and 90°C at a circulation flow rate of 2.0 l/min are plotted in Figs 3–5 The curves are best-fit smooth curves passing through the average values of the percentage of soil remaining on the plates for each circulation time. Individual points have been omitted for clarity, but each run was repeated at least three times randomly, not consecutively, to give the average figure. The broken curves represent the overall cleaning curve for the complete bank of six plates.

The shape of the curves is typical of most cleaning operations. There is an initial rapid rate of cleaning followed by a slowing rate of soil removal, and finally very little or no soil being removed. The percentage of soil remaining is the weight of final dried soil to initial dried soil on the plate. The Plate Nos 1–6 represent the label of the plates as shown in Figs 2a and b, where Plate No. 6 was nearest to the fluid inlet of the heat exchanger. All of the plates were arranged in numerical order for all the experiments, Plate Nos 2, 4 and 6 had soil painted on the gasket sides and Plate Nos 1, 3 and 5 had soil painted on the non-gasket sides.

It is clear, from Figs 3–5, that there was more soil remaining on the even plates than on the odd plates, and this is believed to be a result of some soil being caught at the edge of the gaskets on the even plates. The graphs also clearly

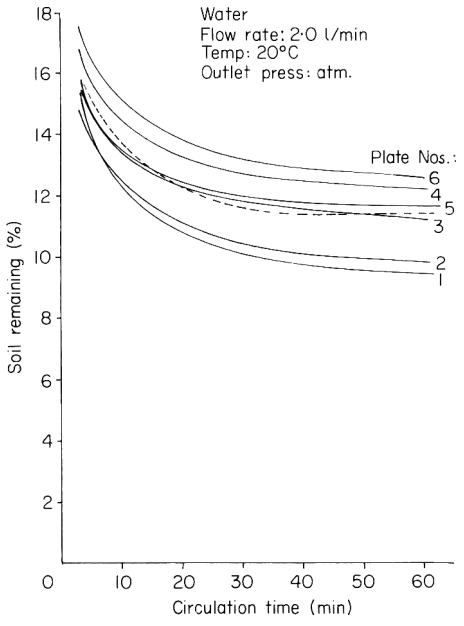


Figure 3. Cleaning curves of individual plates (20°C). – – –, overall.

indicate that there was more soil remaining on the plates in the entry section (higher plate numbers) than in the exit section, a phenomenon which has not been previously reported in the literature. Changes in position of the plates in the drying oven and preferential order of application of diluted paste on plates

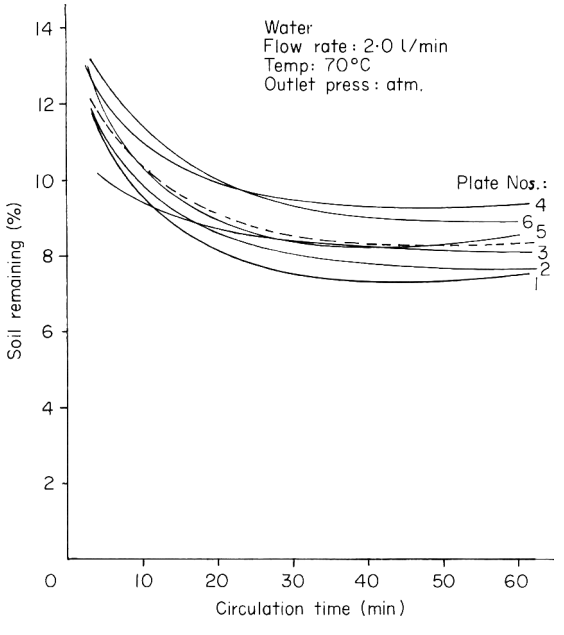


Figure 4. Cleaning curves of individual plates (70°C). – – –, overall.

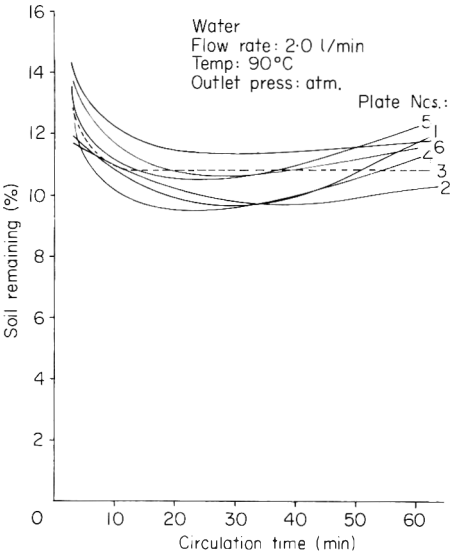


Figure 5. Cleaning curves of individual plates (90°C). – – –, overall.

did not affect the results. The probability of there being a wider gap between the inlet plates was slim because two different sets of plates were used. The possibility of different adhesion due to surface finish could also be discounted for both sets of plates. The 2–4°C higher temperature at the inlet end, compared to the exit end, should have improved cleaning in the inlet section. This phenomenon may be due to inlet effects as the fluid changes direction from the circular inlet piping to the rectangular flow channel of the plate heat exchanger. In a typical industrial situation, there is normally more soil in the product exit section (steam inlet section) than in the product inlet section (Burton, 1966) and therefore the feature of a cleaner exit end may not be obviously noticeable in the processing industry.

A very high proportion (82%) of the soil is removed in the first 3 min of circulation with water at all temperatures. The water dissolved the brownish soluble solids (caramelized sugar and some mineral salts) leaving a reddish, soft deposit which consists mainly of proteinaceous soil. Prolonged cleaning with water only removed another 4.0–6.0% of soil. The final level of soil remaining on the plates ranged from 7.0–10%. There was no further soil removal after 40 min for water temperatures of 20 and 70°C, and at 90°C, this occurred after about 10 min of circulation. It would appear that there is an optimum time of circulation for maximum removal of soil from the plates, and a longer period of circulation does not remove any further soil.

Statistical analysis of results

The results obtained for the overall percentage of soil remaining on the plate heat exchanger at various circulation times were analysed using linear regression

Table 2. Statistical analysis of results (log-log data). Circulation cleaning at 2.0 l/min, atmospheric pressure discharge

Temperature (°C)	Slope	Correlation coefficient (r)	Regression equation*	Circulation time ranges (min)
20	−0.12	−0.80	$y = 18.15t^{-0.12}$	3–60
70	−0.14	−0.75	$y = 14.06t^{-0.14}$	3–60
90	−0.04	−0.55	$y = 12.36t^{-0.04}$	3–60

*y = percentage soil remaining; t = time (min).

theory (Murdoch & Barnes, 1973). The results are best represented as a power law in log-log form. The slope and equation of the best fit regression line, coefficient of correlation and range of circulation times are shown in Table 2.

The magnitude of slope of the lines gives a measure of the rate at which the soil is removed, the greater the magnitude the faster the rate of soil removal.

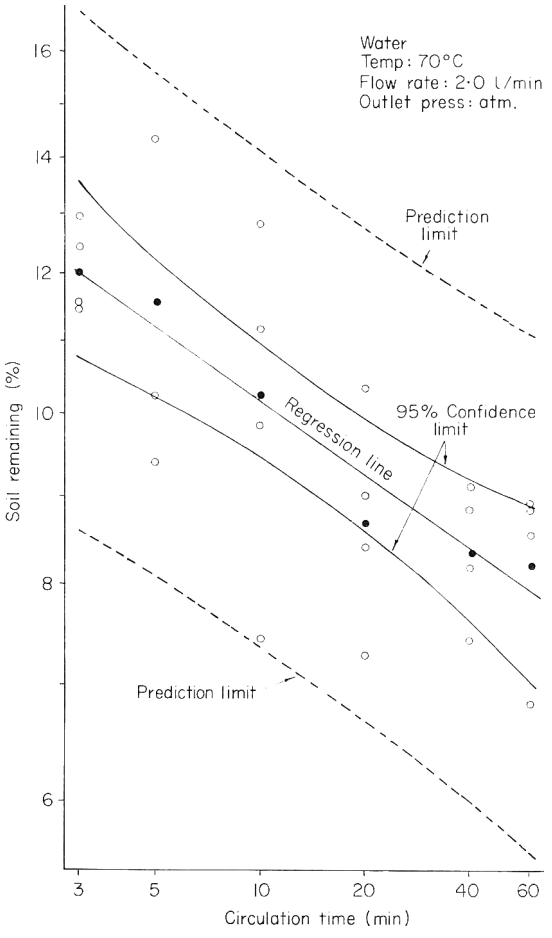


Figure 6. Log-log plot of overall cleaning curve. ○, Individual value; ●, average value.

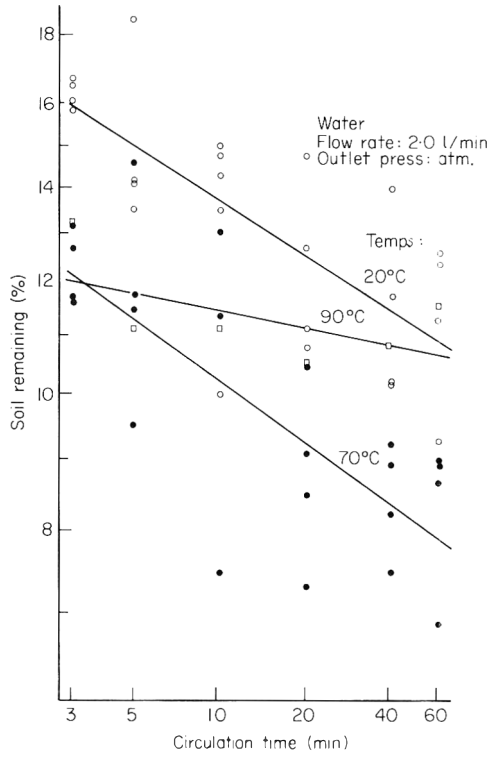


Figure 7. Effect of temperature on cleaning (water) (log-log plot). ○, 20°C; ●, 70°C; □, 90°C.

The negative sign is due to the fact that the longer the circulation time the less soil remains. The correlation coefficient value of regression is relatively high except for water at 90°C, probably due to the formation of tenaceous denatured protein which resists further removal. The values for the correlation coefficient at the other two temperatures indicate that the relationship between soil remaining and circulation time for each of the conditions is highly significant.

The scatter diagram, fitted regression line, 95% confidence limit and prediction limit for the results obtained when cleaning with water at 70°C are shown in Fig. 6 (using log-log scales). The results for all temperatures at a flow rate of 2.0 l/min are shown in Fig. 7 where it is apparent that 70°C is the optimum temperature when using water.

Weighing method

The weights of the plates with and without soil could only be measured to an accuracy of ± 0.01 g. A conservative estimate of the errors involved in the amount of residual soil for both individual plates and for the whole plate heat exchanger assembly was ± 1.2% irrespective of soil load. It was also found that most of the experimental points did not cross the smoothed curves between different cleaning temperatures (Fig. 8). The consistent results suggest that the

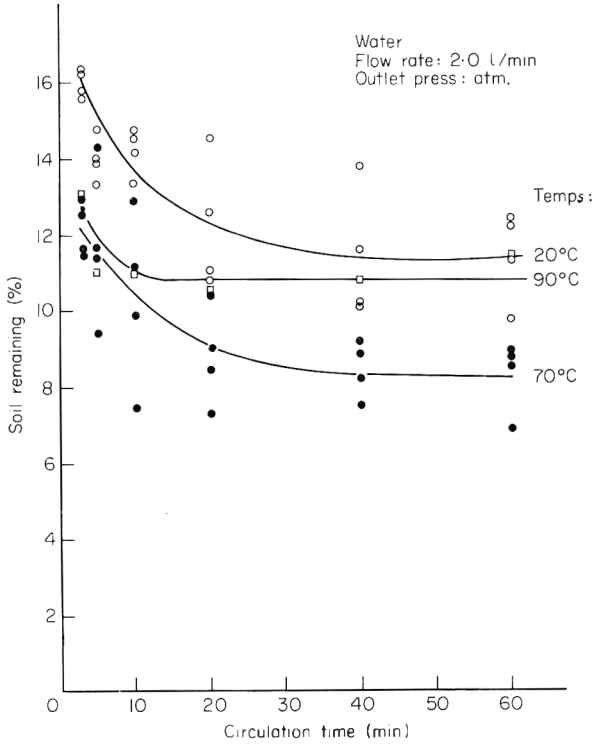


Figure 8. Effect of temperature on cleaning (water). ○, 20°C; ●, 70°C; □, 90°C.

errors in weighing are systematic errors. The amount of residual soil on the plates was small (0.20 g average) and it was confirmed in a number of experiments that the drying conditions of 112°C for 45 min was sufficient to produce dried soil of constant weight. The plates were left to cool in the atmosphere after drying, and since the accuracy of the balance used was ±0.01 g, the use of a desiccator was considered unnecessary.

Conclusions

In this investigation, an attempt has been made to study some of the factors affecting circulation cleaning of a plate heat exchanger fouled with tomato juice. Based on the results obtained, the following conclusions may be draw:

(1) the soiling technique used provides a simple and consistent method of studying circulation cleaning of plate heat exchangers. The dried-on tomato film after 3 min of rinsing with water shows characteristics similar to some encountered in commercial processing;
(2) the gravimetric method of measuring the soil on the plates was found to be effective in evaluating the cleaning efficiency and was found to be reproducible.

This gravimetric method coupled with visual inspection of the plates provides a very useful method of assessment;
(3) the final amount of residual soil becomes constant after a certain optimum circulation time, and extended circulation periods do not improve the cleaning efficiency of the plates; and
(4) the relationship between percentage of residual soil (y) and circulation time of cleaning fluid (t) can be represented by the expression $y = At^{-B}$, where A and B are constants for a specific cleaning condition.

It should be noted that the application of tomato juice which is then oven-dried represents a very severe soiling situation which will only be obtained in practice under severe fouling conditions (plant maloperation). Most plant-cleaning problems will be less severe and the absolute values obtained in this work for the constants in the expression above will differ from those found in practice.

The work is being extended to study the use of caustic solutions and the effects of other process parameters on the cleaning operation.

Acknowledgments

The authors would like to thank the APV Co. Ltd, Crawley, for providing the plate heat exchanger and Libby, McNeil & Libby Ltd, Milnthorpe, for providing the tomato paste. One of the authors would like to thank the Mara Institute of Technology, Malaysia, for providing a scholarship.

References

Abele, C.A. (1965) *J. Milk Fd Technol.* **28,** 257.
Anderson, R.M., Satanek, J. & Harris, J.C. (1959) *J. Am. Oil Chemists Soc.* **36,** 286.
Armbruster, E.H. & Ridenour, G.M. (1958) *Soap Sanitation Chem.* **28** (6), 83.
Armbruster, E.H. (1962) *The Sanitarian,* Jul/Aug.
Baldock, J.D. (1974) *J. Milk Fd Technol.* **37** (7), 361.
Bourne, M.C. & Jennings, W.G. (1961) *Fd Technol.* **15,** 495.
Bourne, M.C. & Jennings, W.G. (1963) *J. Am. Oil Chemists Soc.* **40,** 523.
Burton, H. (1964) *Report No. 109,* NIRD, Shinfield.
Burton, H. (1966) *J. Diary Res.* **33,** 317.
Daoud, I.S. (1980) APV Co. Ltd, Crawley (private comm.).
Durham, K. (1961) *Surface Activity & Detergency.* Macmillan, London.
Gordon, K.P., Hankinson, D.J. & Carver, C.E. (1968) *J. Diary Sci.* **51** (4), 520.
Hankinson, D.J. & Carver, C.E. (1968) *J. Dairy Sci.* **51,** 1761.
Harper, W.J. (1974) In: *Food Sanitation* (Ed. R. E. Guthrie) p. 130. AVI Ltd, Westport.
Jennings, W.G., McKillop, A.A. & Luick, J.R. (1957) *J. Dairy Sci.* **40,** 1471.
Jennings, W.G. (1963) *Fd Technol.* **17** (7), 53.
Jennings, W.G. (1964) In: *Advances in Food Research* (Eds C. C. Chichester, E. M. Mrak & G. F. Stewart) vol. 14, p. 325, Academic Press, London.
Maxcy, R.B. & Shahani, K.M. (1961) *J. Milk Fd Technol.* **24,** 122.

Morgan Jr, A.I. & Wassermann, T. (1959) *Fd Technol.* **13** (12), 691.
Murdoch, J. & Barnes, J.A. (1973) *Statistics: Problems & Solutions.* Macmillan, London.
Smith, H.N.W. (1969) *Candy Ind. Confectioners J.* **132,** 22 and 25.
Tamplin, C. (1980) In: *Hygenic Design & Operation of Food Plants* (Ed. R. Jowitt) p. 183, Ellis Horwood, Chichester.
Timperly, D.A. & Lawson, G.B. (1980) In: *Hygenic Design & Operation of Food Plants* (Ed. R. Jowitt) p. 79, Ellis Horwood, Chichester.
Untermohlen, W.P. & Wallace, E.L. (1947) *Text. Res. J.* **17,** 670.

(Received 3 August 1981)

J. Fd Technol. (1982) **17**, 431–440

Circulation cleaning of a plate heat exchanger fouled by tomato juice

II. Cleaning with caustic soda solution

C. S. CHEOW* AND A. T. JACKSON†

Summary

The effect on the circulation cleaning of a plate heat exchanger fouled with diluted tomato paste using a 2.0% w/w caustic soda solution has been studied at 70 and 90°C, and the effect of increased flow rate and overall pressure of the system has been investigated at a fluid temperature of 90°C.

Increasing the temperature of the 2.0% caustic soda solution increased the cleaning rate but did not significantly reduce the final amount of soil remaining on the plates. Increasing the circulation rate of the fluid increased the cleaning rate and also the final amount of residual soil.

An increase in pressure of the system decreased the overall cleaning efficiency.

Introduction

A typical cleaning cycle of a plate heat exchanger used in tomato juice processing consists of three phases, (1) pre-rinse, (2) alkaline wash and (3) post-rinse. The pre-rinse phase is accomplished by circulating cold tap water for 3–5 min, and removes the majority of tomato residue from the heat exchanger surfaces. In the alkaline wash phase, a 1.0–2.0% caustic soda-based detergent is circulated for 20–40 min and removes the remaining soil, particularly the proteinaceous soil. The final phase consists of circulation of clean water, possibly followed by dilute food grade acid solution, to ensure that all traces of alkali are removed. In-place cleaning of plate heat exchangers is always backed by strip-down inspection from time to time, since in cases of severe fouling CIP may not be completely effective and the plates may have to be manually scrubbed.

Authors' addresses: *Mara Institute of Technology, Malaysia, and †Department of Chemical Engineering, The University of Leeds, Leeds LS2 9JT.

†To whom correspondence should be addressed.

0022–1163/82/0800–0431 $02.00

Soil removal mechanisms

The mechanism of detergent action in soil removal has been attributed to many factors, e.g. surface tension, interfacial tension, contact angle, surface viscosity, foaming, electrostatic properties, adsorption, wetting, suspending action, emulsification, saponification and lubrication (Bourne & Jennings, 1963). If sodium hydroxide only is used as the detergent, such factors as solubilization, foaming and surface tension are virtually eliminated.

Fundamental studies involving model systems of homogeneous soils on homogeneous surfaces have assisted in understanding detergency processes, and show that the strong soil–substrate bond and weak soil–soil bond is involved in all cases. Using a single pass alkaline cleaning system with a small commercial high-temperature, short-time pasteurizer, Arnold & Maxcy (1970) found that saponification was not involved in the initial removal of lipids from the equipment surfaces. The lipids occurred in the cleaning solution as neutral fats rather than as fatty acids.

Harper (1974) viewed the fundamental phenomena involved in the cleaning process in terms of four essential steps:

(1) bringing the detergent solution into intimate contact with the soil to be removed by means of good wetting and penetrating properties;
(2) displacement of solid and liquid soils from the surface to be cleaned by saponifying of fats, peptizing of proteins and dissolving the minerals;
(3) dispersion of the soil in the solvent by dispersion, deflocculation or emulsification; and
(4) preventing redeposition of the dispersed soil back on to the clean surface by providing good rinsing properties.

Materials and methods

The experimental cleaning circuit consisted of an APV Junior Paraflow plate heat exchanger with seven plates, arranged to give three in-series passes for the cleaning fluid. The surfaces of the heat exchanger plates were soiled using diluted 28–30% double concentrate tomato paste painted on the plates, the plates then being dried in a laboratory oven at 112°C for 45 min. The details of the cleaning circuit and the soiling method, together with details of the determination of soil remaining on the plates after cleaning, has been described by Cheow & Jackson (1982).

The procedure for cleaning with caustic soda solution was similar to that used for water cleaning with the addition of a 3 min pre-rinse phase and a 3 min post-rinse phase using water at 20°C at a flow rate of 2.0 l/min.

Two per cent caustic soda solution prepared by diluting 50% A.R. solution was placed in the 100 l feed tank and heated to the required temperature. During the experimental runs, the pre- and post-rinse water was collected in a separate container prior to discharge to drain, and the caustic soda used in the cleaning cycle was recycled back to the 100 l feed tank. The concentration and

alkalinity of the solution was checked after each experiment by titration with standardized sulphuric acid solution, and fresh 50% solution or water was added to the tank to maintain the alkalinity. Scale or flake floating on the top or sinking to the bottom of the solution was removed before and after every experiment. The caustic soda solution was re-used in all the experimental runs in conjunction with a constant alkalinity check. The re-use of detergent solutions is common practice in industry and has been the subject of extensive research (Smith & Hedrick, 1967; Meyer, 1972; Asher, 1975).

A flow rate of 2.0 l/min was used to determine the effect on cleaning of temperatures of 70 and 90°C, and the effect of overall system pressure was studied at a temperature of 90°C. In addition, a flow rate of 3.0 l/min at a caustic soda temperature of 90°C was studied. In all the experimental runs the pressure at the outlet of the plate heat exchanger was atmospheric, except in the case of deliberately applied back-pressure.

Results

Temperature

The experimental results for circulation cleaning a a caustic soda flow rate of 2.0 l/min at temperatures of 70 and 90°C are plotted in Figs 1 and 2 as best-fit

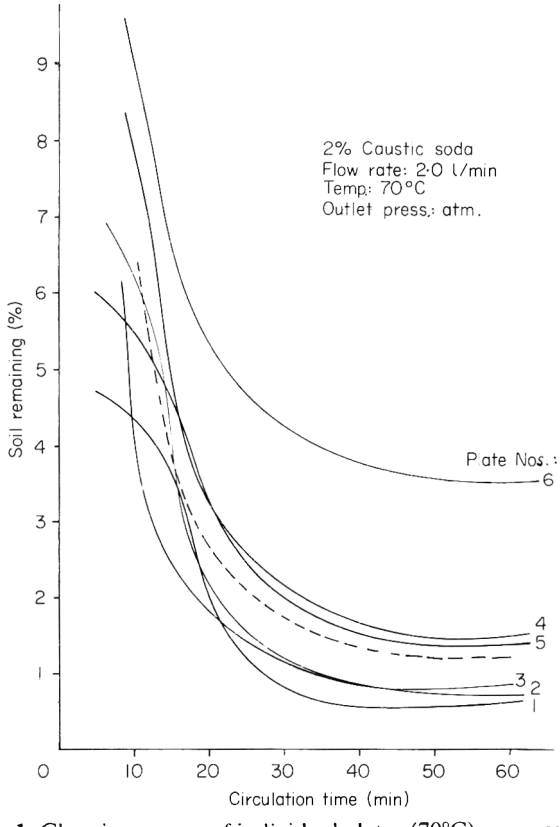


Figure 1. Cleaning curves of individual plates (70°C). ---, overall.

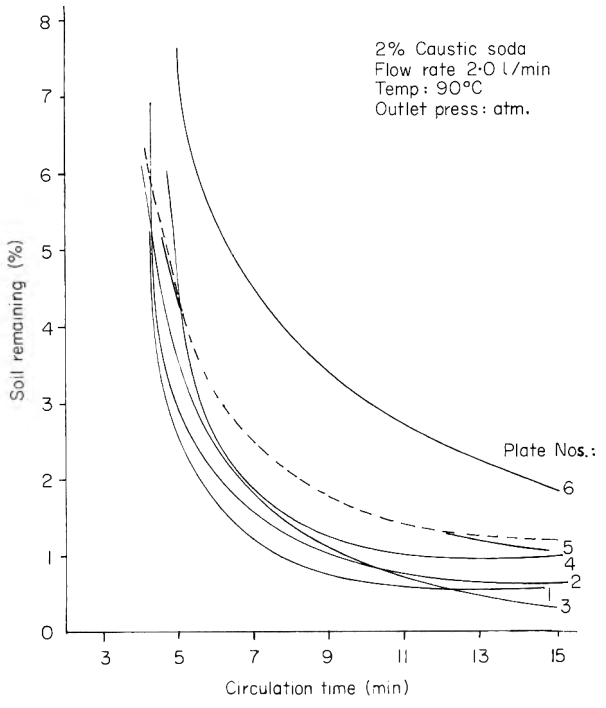


Figure 2. Cleaning curves of individual plates (90°C) (2.0 l/min). ----, overall.

smooth curves passing through the average of the values of percentage soil remaining on the plates for each circulation time, individual points have been omitted for clarity. The broken curves represent the overall cleaning curves for the complete bank of six plates.

The circulation time is the period of time for which the caustic soda solution was circulated, and excludes the 3 min pre- and post-rinse phases using cold water. The Plate Nos. 1–6 represent the label of the individual plates arranged in the plate heat exchanger in numerical order. Plate No. 6 was nearest the fluid inlet and Plate No. 1 nearest the fluid exit (Cheow & Jackson, 1982).

System pressure

The experimental results obtained for the overall circulation cleaning of the plate heat exchanger using 90°C caustic soda solution at a flow rate of 2.0 l/min with two different system pressures are plotted in Fig. 3. The pressure drop across the plate heat exchanger at this flow rate was found to be 55 kN/m^2, and by applying a back-pressure of 55 kN/m^2 at the outlet of the heat exchanger the inlet pressure rose to 110 kN/m^2 g.

Flow rate

The experimental results for circulation cleaning at 90°C using a caustic soda flow rate of 3.0 l/min are plotted in Fig. 4.

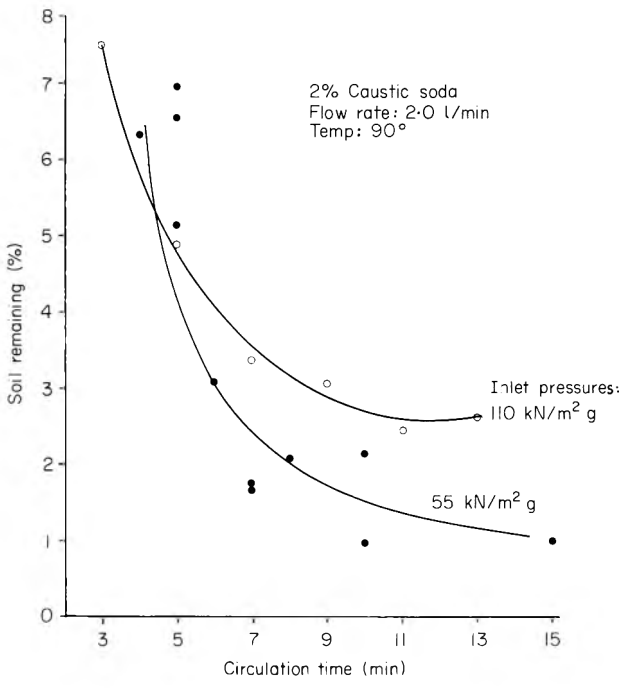


Figure 3. Effect of pressure on cleaning. ●, 55 kN/m² g; ○, 110 kN/m² g.

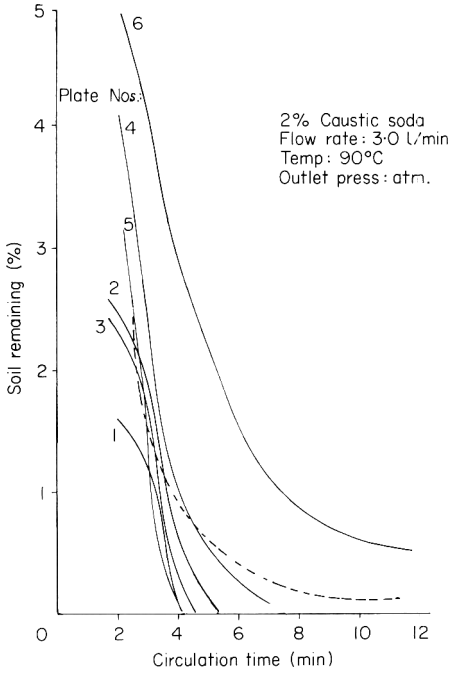


Figure 4. Cleaning curves of individual plates (90°C) (3.0 l/min). ---, overall.

Table 1. Statistical analysis of results (log-log data). Equation $y = At^{-B}$ (y = percentage soil remaining; t = circulation time, min). Two percent w/w caustic soda solution

Temperature (°C)	Flow rate (l/min)	Outlet pressure (kN/m²)	Slope (B)	Correlation coefficient (r)	Circulation time ranges (min)
70	2	Atmos.	−0.68	−0.84	5–60
90	2	Atmos.	−1.08	−0.81	4–30
90	2	55	−0.77	−0.98	3–13
90	3	Atmos.	−2.26	−0.96	2–11

Statistical analysis of results

The results obtained for the overall percentage of soil remaining on the plate heat exchanger after cleaning were analysed using linear regression theory (Murdoch & Barnes, 1973). The results are best represented by a power law in log-log form. The slope of the equation of best-fit regression line, coefficient of correlation and range of circulation times are given in Table 1. The overall effects of temperature, pressure and flow rate are plotted on log-log scales in Figs 5–7.

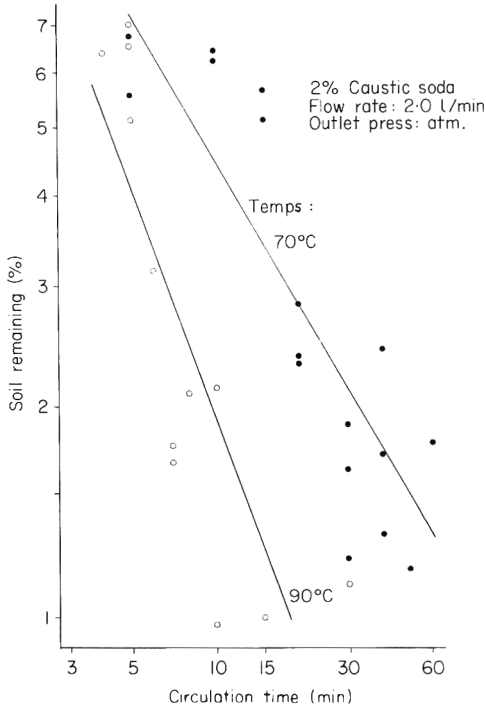


Figure 5. Effect of temperature on cleaning (caustic soda) (log-log plot). ●, 70°C; ○, 90°C.

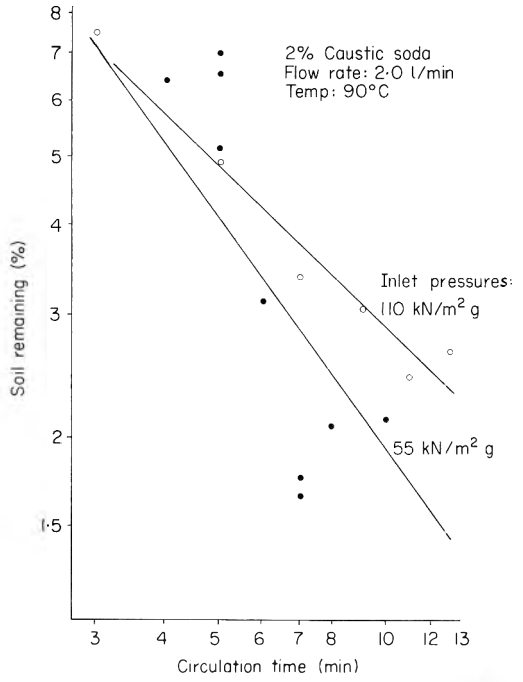


Figure 6. Effect of pressure on cleaning (log-log plot). ●, 55 kN/m² g; ○, 110 kN/m² g.

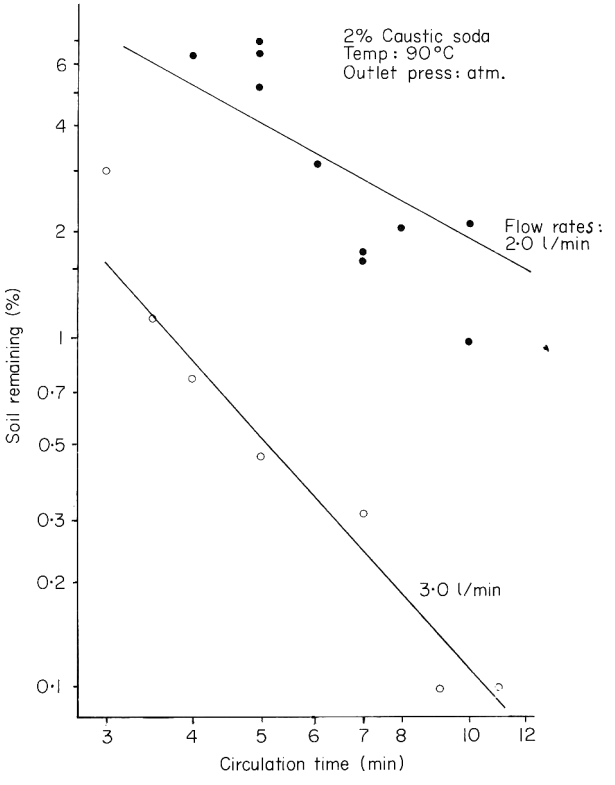


Figure 7. Effect of flow rate on cleaning (log-log plot). ●, 2.0 l/min; ○, 3.0 l/min.

Discussion

The influence of factors such as temperature, flow rate and pressure on the circulation cleaning of a plate heat exchanger was studied by comparison with a standard condition of 2.0% caustic soda solution at a temperature of 90°C and circulating at a flow rate of 2.0 l/min and at atmospheric outlet pressure. This condition is a typical cleaning condition for the APV Junior Paraflow heat exchanger scaled from commercial practice (Daoud, 1980; Robbins, 1980).

Temperature

It is clear, from Fig. 5, that a temperature of 70°C shows a decrease in the rate of soil removal compared with a temperature of 90°C. During this work, the circulation time required to reach a residual soil level of 1.0% was decreased from 40 to 15 min on increasing the temperature by 20°C. Jennings (1959) demonstrated that the removal of cooked-on milk films by caustic soda solution exhibited a Q_{10} value of 1.6, i.e. the rate of soil removal increased by a factor of 1.6 for every 10°C rise in temperature. This current work shows an approximate Q_{10} value of 1.58 for tomato film.

The final level of residual soil on the plates of the heat exchanger after a long circulation time (30 min) is only slightly greater at 70°C compared with 90°C, and it is apparent that temperature affects the circulation time required to achieve a certain result and not necessarily the final level of cleanliness. This also agrees with previous work on milk films by Jennings (1959).

No apparent protein denaturing effect on the soil at 90°C was observed when using caustic soda solution in contrast to cleaning with water (Cheow & Jackson, 1982).

System pressure

The high level of soil remaining on the inlet plates of the heat exchanger (high plate numbers), comparable with previous work (Cheow & Jackson, 1982), was thought to possibly be due to the lack of back-pressure at the outlet of the system. It was clear, however, from the experimental results that this was not the case and, moreover, an increase in total pressure of the system resulted in less efficient cleaning (Fig. 6), this result agreeing with the findings of Jennings (1960) which showed that cleaning is more efficient at reduced pressures, the efficiency increasing linearly.

Flow rate

The effect of an increase in flow rate from 2.0 to 3.0 l/min at a caustic soda temperature of 90°C is shown in Figs 4 and 7. The most significant effect of this increase in flow rate is that it is possible to achieve complete visual cleanliness at 11 min circulation time, a condition impossible to achieve even after 60 min circulation time at a flow rate of 2.0 l/min. This agrees with the work of

Jennings, McKillop & Luick (1957) who found that turbulence is more important than temperature in circulation cleaning of milk soils.

Conclusions

(1) An increase in the temperature of the 2.0% w/w caustic soda solution increased the cleaning rate at a flow rate of 2.0 l/min (fluid velocity 0.304 m/sec), but did not significantly reduce the final amount of residual soil on the plate heat exchanger at this flow rate;
(2) an increase in the flow rate of the caustic soda solution at a constant temperature of 90°C increased the cleaning rate as well as increasing the removal of final traces of residual soil;
(3) an increase in the system pressure reduced both the cleaning rate and efficiency; and
(4) the relationship between the percentage of residual soil (y) and the circulation time (t min) can be represented by the expression $y = At^{-B}$, where A and B are constants for a particular cleaning condition. The numerical value of the constant B gives a clear guide to the cleaning efficiency of the system, the higher the value, the better the cleaning obtained. The correlation coefficients ranged from 0.84 to 0.98, showing that the results fall well within the predicted errors of the experiments.

The poor cleaning performance of the first (inlet) section of the heat exchanger is not readily explicable, but visual inspection of the soil remaining on Plate Nos 5 and 6 show that the last area to be cleaned is the area on the top left of the section, the top right containing the outlet port. This would suggest channelling or uneven velocity in the flowing fluid in the first flow section. Holland *et al.* (1953) pointed out that most plate heat exchangers have not been designed specially for in-place cleaning, therefore some areas cannot be sufficiently cleaned since solution reaching these areas is not moving with sufficiently high velocity.

The soil system used in these experiments represents a very severe fouling situation, most plant-cleaning problems will be less severe.

Acknowledgments

The authors would like to thank the APV Co. Ltd, Crawley, for providing the plate heat exchanger and Libby, McNeil & Libby Ltd, Milnthorpe, for providing the tomato paste. One of the authors would like to thank the Mara Institute of Technology, Malaysia, for providing a scholarship.

References

Arnold, R.G. & Maxcy, R.B. (1970) *J. Dairy Sci.* **53,** 1540.
Asher, H. (1975) *Dairy Ind.* **40,** 341.

Bourne, M.C. & Jennings, W.G. (1963) *J. Am. Oil Chemists Soc.* **40,** 523.
Cheow, C.S. & Jackson, A.T. (1982) *J. Fd Technol.* **17,** 417.
Daoud, I.S. (1980) APV Co. Ltd, Crawley (private comm.).
Harper, W.J. (1974) In: *Food Sanitation* (Ed. R. E. Guthrie) p. 130. AVI Ltd, Westport.
Holland, R.F., Shaul, J.D., Theokas, D.A. & Widlan, H.M. (1953) *Fd Engng* **25** (5), 75.
Jennings, W.G., McKillop, A.A. & Luick, J.R. (1957) *J. Dairy Sci.* **40,** 1471.
Jennings, W.G. (1959) *J. Dairy Sci.* **42,** 1763.
Jennings, W.G. (1960) *Fd Technol.* **14,** 591.
Meyer, F. (1972) *Am. Soft Drink J.* **127,** 22.
Murdoch, J. & Barnes, J.A. (1973) *Statistics: Problems & Solutions*. Macmillan, London.
Robbins, H. (1980) Beecham Products Ltd, Coleford (private comm.).
Smith, G.A. & Hedrick, T.I. (1967) *J. Milk Fd Technol.* **30,** 256.

(*Received 3 August 1981*)

J. Fd Technol. (1982) **17**, 441–449

Overshooting of thermal processes due to temperature distribution at steam-off

I. J. KOPELMAN,* D. NAVEH† AND I. J. PFLUG†

Summary

The continued temperature rise after steam-off and during initial cooling that occurs in the slowest heating zone of conduction heating products during thermal processing has not been considered as a design parameter for the sterilization of low acid foods packaged in hermetically sealed containers in the existing mathematical methods.

This study demonstrates the 'overshooting' phenomenon and examines its significance utilizing a numerical approach. Preliminary results indicate that under certain conditions this phenomenon may contribute a significant portion of the lethality delivered to the slowest heating zone in the container.

Introduction

The computation of the sterilization value delivered during the thermal processing of low acid foods packaged in hermetically sealed containers requires knowledge of the time–temperature history at the slowest heating zone of the container and the temperature coefficient of the micro-organism to be killed in the process. Ideally, time–temperature history of the heating and cooling parts of the sterilization process would be expressed in an explicit function that would be integrated over process time, t_B (see Appendix for definition of symbols), using the temperature coefficient, z, to yield a sterilization value, F, eqn (1).

$$F = \int_0^{t_B} 10^{**} [T_1 - T(t)/z] \, dt \tag{1}$$

Authors' addresses: *Department of Food Engineering and Biotechnology, Technion – Israel Institute of Technology, Haifa, Israel, and †Department of Food Science and Nutrition, University of Minnesota, St Paul, Minnesota 55108, U.S.A.

*To whom correspondence should be addressed.

0022–1163/82/0800–0441

Since these functions are not readily available, other procedures have been developed. The General Method (Bigelow *et al.*, 1920; Patashnik, 1953), which is a graphical or numerical integration of eqn (1), uses the actual time–temperature data, yielding an accurate measure of the sterilization value of the thermal process.

The time–temperature heating and cooling data can be parameterized and then mathematical methods can be employed in relating the sterilization value, F, with processing conditions and the heating rate parameters. The accuracy of the mathematical methods is limited by the degree to which the temperatures at the slowest heating zone calculated from the parameters agree with the actual temperatures. The inability to accurately calculate the temperatures occurring during the initial cooling stages in conduction heating products has been a major problem of those developing these methods. In commenting on this problem, Ball & Olson (1957) wrote, 'If cooling curves had no initial lag . . . process calculation would be a relatively simple art and this book could be reduced to a mere pamphlet'.

In conduction heating products at steam-off, large temperature gradients exist across the container and lead to the subsequent temperature rise or 'overshooting' of the temperature at the slowest heating zone during the initial cooling phase. The mathematical methods of Ball (1923), Ball & Olson (1957), Hayakawa (1970), Stumbo (1973), and others do not include the added lethality due to this overshooting phenomenon. Hicks (1951) reported the phenomenon and calculated the lethality incurred during the early cooling phase and Lund (1975) alluded to it.

Though not included in the mathematical methods in use today, this lethality is taken into account in commercial practice and is incorporated into the recommended processing conditions by the National Food Processors Association, formerly the National Canners Association (NCA), and American Can Company (Pflug & Esselen, 1979). For example, using the recommended processing conditions and the heating parameters for cream-style corn, packed in No. 10 cans, an F_0 of 2.9 min is calculated (Ball method). This value is markedly lower than the design F_0 of 6 min recommended for this product when packed in No. 2 cans (NCA, 1962). Similarly, an F_0 of 12 min is calculated (Ball method) for dog food in No. 2 cans, whereas a target design F_0 of 6 min is used for the same product packaged in No. 10 cans (American Can Co.). The calculated lethality delivered to conduction heating products in larger size cans may be half of that calculated for smaller sized cans. However, the actual sterilizing value for large (i.e. No. 10) cans evaluated by the general-graphical method is much higher than the sterilizing value calculated by the Ball method and approaches the sterilizing value obtained for smaller (i.e. No. 2) cans (Alstrand & Ecklund, 1952).

The purpose of this study was to demonstrate and examine the significance of overshooting, utilizing a numerical approach.

Methods

Mathematical methods for thermal process calculations in common use

As was earlier noted, mathematical methods of process calculation are based upon integration of eqn (1) over the time–temperature conditions.

The procedure for the prediction of the time–temperature condition (normally taken at the slowest heating point) is commonly based on the product's heating rate parameters, j_h, f, and processing parameters, T_0, T_1, and t.

These parameters and the variable temperature, T, are all related in eqn (2).

$$\log (T_1 - T) = -\frac{t_B}{f_h} + \log [j_h (T_1 - T_0)] \tag{2}$$

The temperature, T_{t_B}, at steam-off, t_B, is the final and the highest temperature for any given location during the heating portion of the process. Based on this

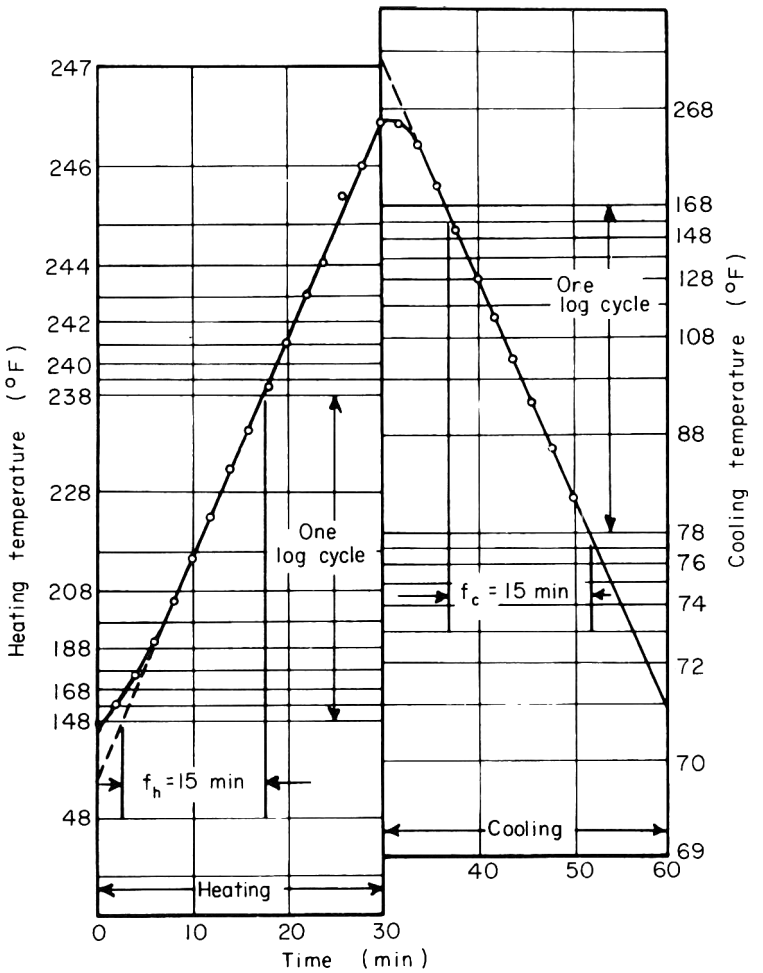


Figure 1. Semilogarithmic time–temperature responses for an entire heat sterilization process. f = Time required (min) for the asymptote of the heating or cooling curve to cross one log cycle, i.e. the time required for a 90% change in temperature on the linear portion of the curve.

temperature, the cooling portion is 'appended', completing the heating–cooling curve (Fig. 1). The integration process indicated in eqn (1) is carried out numerically yielding the sterilization value. The numerically obtained lethality values have been directly correlated (tables, graphs or formulae) with the heating parameters so sterilization values as a function of process parameters (T_0, j_h, f, T_1 and t_B) can easily be obtained (Ball & Olson, 1957).

The present approaches and their tabulated or graphically calculated results assume, therefore, that by steam-off (i.e. $t = t_B$) the temperature at any given location has reached its maximum value. In reality, this is only the case when there is little or no temperature distribution across the container at steam-off.

Computational method

The basic calculation method used in this analysis was a simple computer model based on the finite difference approximation to the unsteady-state conduction equation in one dimension. The example is a conduction heating food in a flexible package (pouch).

Using this model, the pouch is imaginarily divided into a number of thin layers, denoted Δx, in thickness. For each time step (Δt) the computer calculates the temperatures (T) in each layer according to the following recursion equation (Smith, 1965).

$$T(I, J) = T(I, J-1) + \frac{\alpha \Delta t}{\Delta x^2}\left[T(I-1, J-1) - 2T(I, J-1) + (I+1, J+1)\right]$$

where I is the layer number, $I = 1, 2, 3 \ldots$ (number of layers), and J is the time-step number, $J = 1, 2, 3 \ldots$ (number of time steps).

Assuming negligible surface resistance to heat transfer, the surface temperature is the externally imposed temperature condition. After each time step the average lethality in the layer is computed based on the computed temperatures in each layer.

Results and discussion

A quantitative evaluation of the overshooting phenomenon can be demonstrated by assigning numerical values to the model. A 1 in thick pouch, initially at 72°F, is exposed to a 260°F environment for 24 min, after which the environmental temperature is reduced to 160°F. The pouch is divided into ten layers, each 0.1 in thick. Temperatures in each layer are computed at intervals of 0.01 min. The product in the pouch is a typical food having a thermal diffusivity of 0.005 ft²/hr.

Temperature effect of overshooting

The temperature distribution within the body during heating, for time and position as a parameter, is shown in Figs 2 and 3 respectively. Temperature

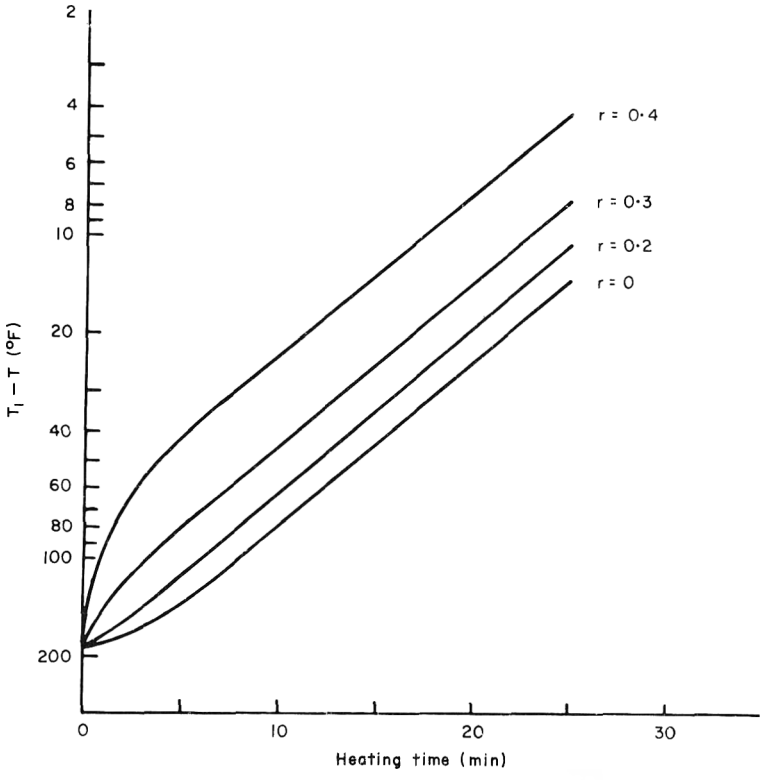


Figure 2. Heating profiles at different locations in pouch for T_1 = 260°F. T = Temperature; T_1 = temperature of coolant or heating media.

profile at steam-off ($t = t_B$) and during early cooling demonstrates the formation of a 'shoulder' (Fig. 4) due to heat flow to the cooled pouch surface and to the pouch centre. During this time the flow of heat towards the pouch centre causes a continued temperature rise at the centre – overshooting. The amount of the temperature rise (and decrease in g) is primarily a function of the temperature distribution at steam-off. In terms of the mathematical method parameter, g (g is the difference between the heating medium temperature, T_1, and the cold zone temperature, T), overshooting causes g to decrease to g' as shown in Fig. 5.

When operating at high retort temperatures where the designed sterilization value or F_0 can be delivered in a relatively short processing time (i.e. small t_B/f), large temperature gradients are expected within the container at steam-off (i.e. at $t = t_B$). However, significant temperature gradients may occur even under normal operating temperatures (i.e. 250°F) in large containers. For example, for still-processed, cream-style corn in No. 10 cans based on the processing conditions of NCA (1962), the g at steam-off can be as large as 22°F. Convection heating systems and conduction heating products where the value of t_B/f is large will have small temperature distributions, resulting in relatively small values of g (Fig. 3).

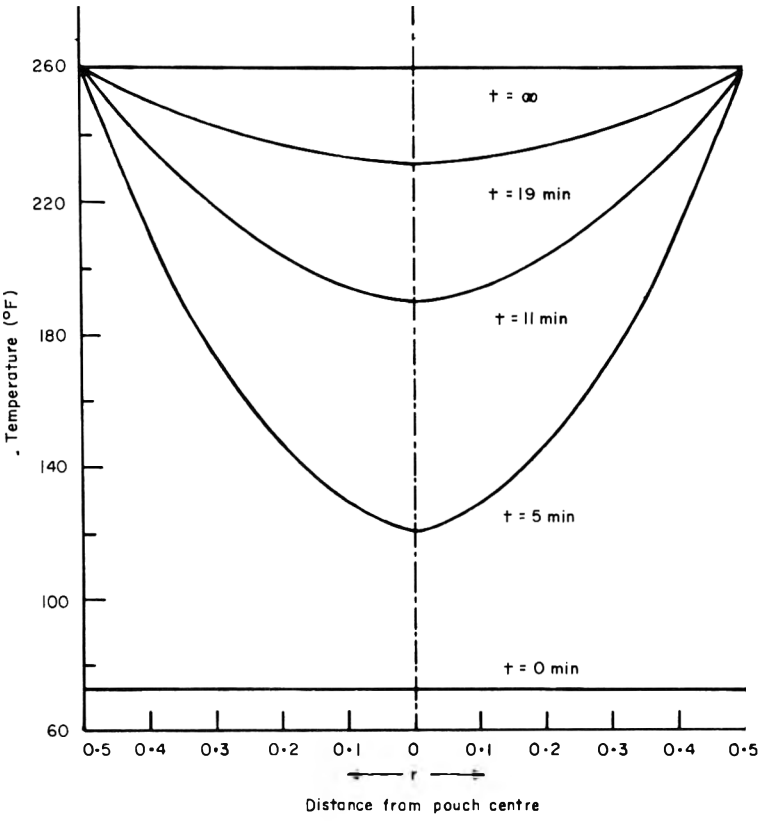


Figure 3. Temperature distribution in a pouch of 1 in thickness heated at 260°F after different times. t = Time.

Data presented in these figures are for a 1 in thick pouch; the data could have been transformed to arrive at dimensionless, more general graphs. Dimensionless temperatures can be expressed using the unaccomplished temperature form whereby

$$Q = \frac{T - T_1}{T_0 - T_1}$$

and time can be expressed using the dimensionless ratio of t/f, thus incorporating both the thermal diffusivity and the body geometry and dimensions.

Effect of overshooting on sterilization value

The cumulative F_0 values at the slowest heating zone were calculated (Fig. 6). The contribution of the overshooting is the integral, over the duration of the overshooting period, of the lethal rate of the overshooting temperatures minus the lethal rate of the temperatures at steam-off. In the case at hand, the overshooting period is from steam-off ($t = t_B = 24$ min; $T = T_{t_B} = 246.07$°F) until $t = 25.54$ min.

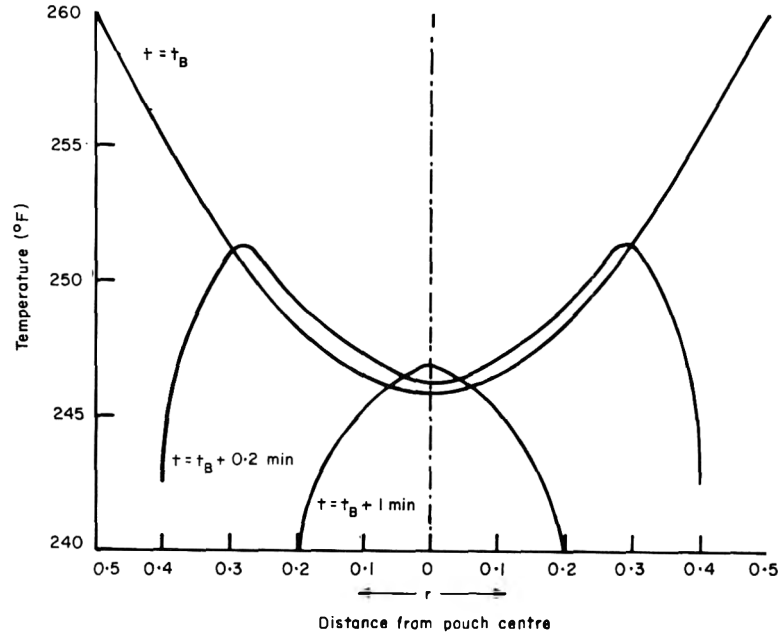


Figure 4. Temperature profiles at steam-off ($t = t_B$), 0.2 min after steam-off ($t = t_B + 0.2$), and 1 min after steam-off ($t = t_B + 1$) in a 1 in thick pouch heat at 260°F. t = Time; t_B = Ball heating time.

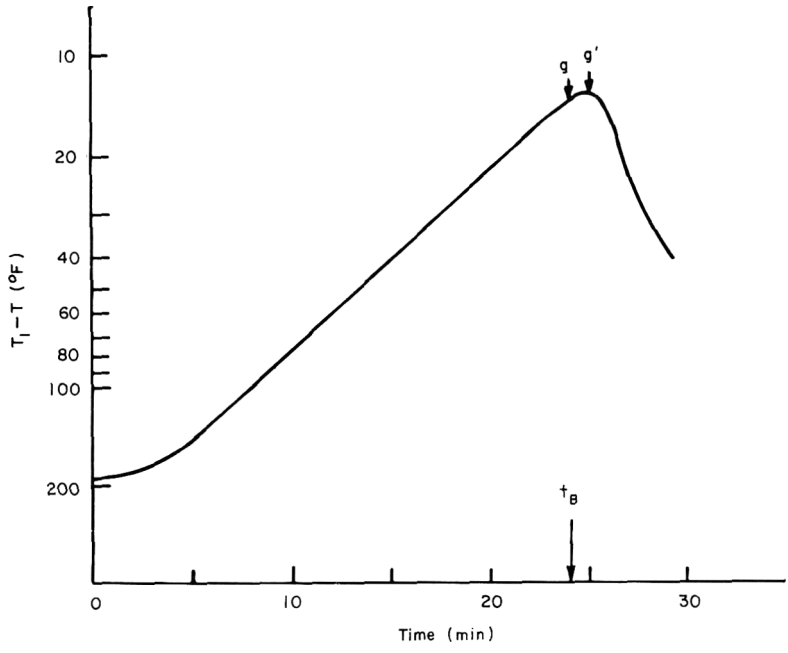


Figure 5. Temperature rise (g to g') due to the temperature distribution at steam-off. g = Difference between retort and product temperature in slowest heating zone at steam-off ($t = t_B$).

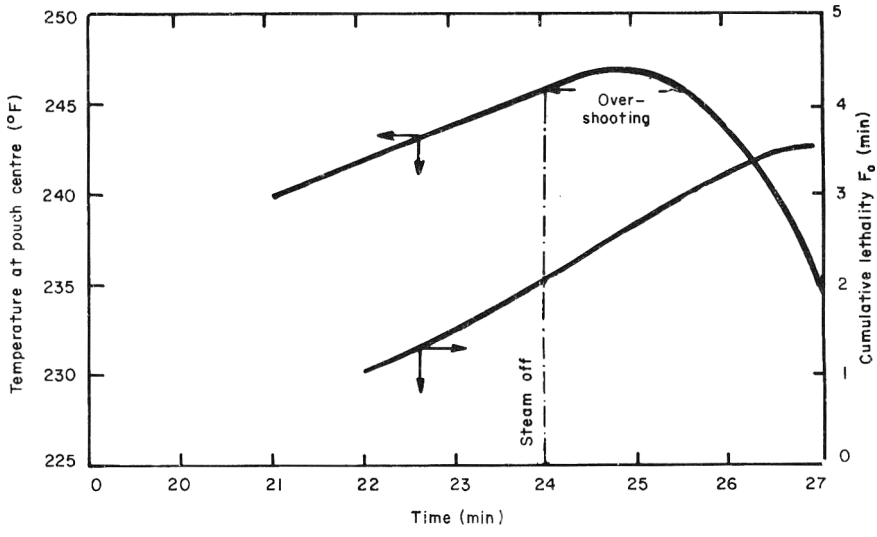


Figure 6. Temperatures and cumulative F_0 values at slowest heating zone of conduction heating product in a 1 in thick pouch before and immediately after steam-off exhibiting continued rise in temperature – the overshooting phenomenon.

For these conditions, the F_0 value delivered to the pouch calculated using the mathematical method of Ball (1923) is 3 min; of this, the F_0 contribution of the cooling part of the process was 1 min. When the time–temperature data are numerically integrated using the General Method, the F_0 value of the whole process is 3.8 min and the F_0 value of the cooling phase is 1.8 min. For the whole process the mathematically calculated F_0 is only 79% of the General Method F_0; for the cooling part the mathematically calculated F_0 (1 min) is only 56% of the cooling part of the General Method F_0 (1.8 min). These differences show the inability of the mathematical method, as presently used, to include the sterilization value due to overshooting.

Conclusion

In this paper the important contribution of overshooting to the lethality delivered in a thermal process has been demonstrated. Further investigations are being carried out, using numerical methods such as finite elements, to develop data that will make possible incorporation of the sterilization value due to overshooting.

Acknowledgments

This research was supported in part by a grant from the U.S.–Israel Bi-national Agricultural Research and Development Fund (BARD) and HHS/FDA Con-

tract 223–79–2258, Scientific Journal Series 12 017, Minnesota Agricultural Experiment Station, St Paul, Minnesota 55108.

Appendix

Symbols

f = Time required (min) for the asymptote of the heating or cooling curve to cross one log cycle; that is, the time required for a 90% change in temperature on the linear portion of the curve.

F_0 = The number of equivalent minutes at $T = 250°F$ delivered to a container or unit of product calculated using a z value of 18°F.

g = Difference between retort and product temperature in slowest heating zone at steam-off ($t = t_B$).

j = Lag factor $(T_a - T_1)/(T_0 - T_1)$.

t = Time; t_B, Ball heating time.

T = Temperature; T_0, initial temperature of product; T_1, temperature of coolant or heating media; T_a, apparent initial temperature as defined by the linear portion of the heating or cooling curve, i.e. the ordinate value of the origin of the asymptote of the curve; T_{t_B}, temperature of the product at $t = t_B$.

z = The temperature coefficient of microbial destruction. It is the negative reciprocal of the slope of the *TDT*, or phantom *TDT* curve. It is expressed in degrees of temperature.

α = Thermal diffusivity.

References

Alstrand, D.V. & Ecklund, D.F. (1952) *Fd Technol.* **6,** 185.

Ball, C.O. (1923) *National Research Council Bull.* **37,**

Ball, C.O. & Olson, F.C.W. (1957) *Sterilization in Food Technology.* McGraw-Hill Co. Inc., New York.

Bigelow, W.D., Bohart, G.S., Richardson, A.C. & Ball, C.O. (1920) *Res. Lab., Natl Canners Assoc. Bull.* **16–L.** Washington, DC.

Hayakawa, K.-I. (1970) *Fd Technol.* **24,** 1407.

Hicks, E.W. (1951) *Fd Technol.* **5,** 134.

Lund, D.B. (1975) *Heat Processing in Principles of Food Science Part II* (Ed. O. R. Fenmena). Marcel Dekker, New York.

National Canners Association (1962) *Natl Canners Assoc. Bull.* **26–L.**, Washington, DC.

Patashnik, M. (1953) *Fd Technol.* **7,** 1.

Pflug, I.J. & Esselen, W.B. (1979) In: *Fundamentals of Food Canning Technology* (Eds Jackson *et al.*) pp. 10–94. AVI Ltd, Westport.

Smith, G.D. (1965) *Numerical Solution of Partial Differential Equations.* Oxford University Press, London.

Stumbo, C.R. (1973) *Thermobacteriology in Food Processing,* 2nd edn. Academic Press, New York.

(*Received 12 August 1981*)

J. Fd Technol. (1982) **17**, 451–460

A calorimetric study of self-heating in coffee and chicory

ALOÏS RAEMY* AND P. LAMBELET

Summary

The technique of heat flow calorimetry was used to determine the specific heat of coffee and chicory products and to study their thermal behaviour above 20°C. Intensive exothermic reactions were particularly evident when measurements were made with sealed cells. The data obtained help in the understanding of how exothermic reactions (and self-heating) occurring in such foodstuffs can bring them above their minimum ignition temperature.

Introduction

The principle of heat flow calorimetry is well known (Calvet & Prat, 1956; Hemminger & Höhne, 1979); this is one of the many techniques of thermal analysis. Instruments based on this principle have been used for many investigations of thermal properties.

Our interest in this technique lies especially in the detection and characterization of exothermic phenomena in foods and also in the possibility of determining specific heat values above 20°C. Calorimetric information is important for designing or operating heating systems or machines. A better knowledge of these food properties should also help to understand how certain food products can be brought above their minimum ignition temperature and can ignite spontaneously.

Materials and methods

Instrument

The instrument we have used mostly for these measurements is a heat flow calorimeter (Setaram C80) of Calvet type. A schematic representation of the

Authors' address: Research Department, Nestlé Products Technical Assistance Co. Ltd, CH-1814 La Tour de Peilz, Switzerland.

*To whom correspondence should be addressed.

0022–1163/82/0800–0451 $02.00

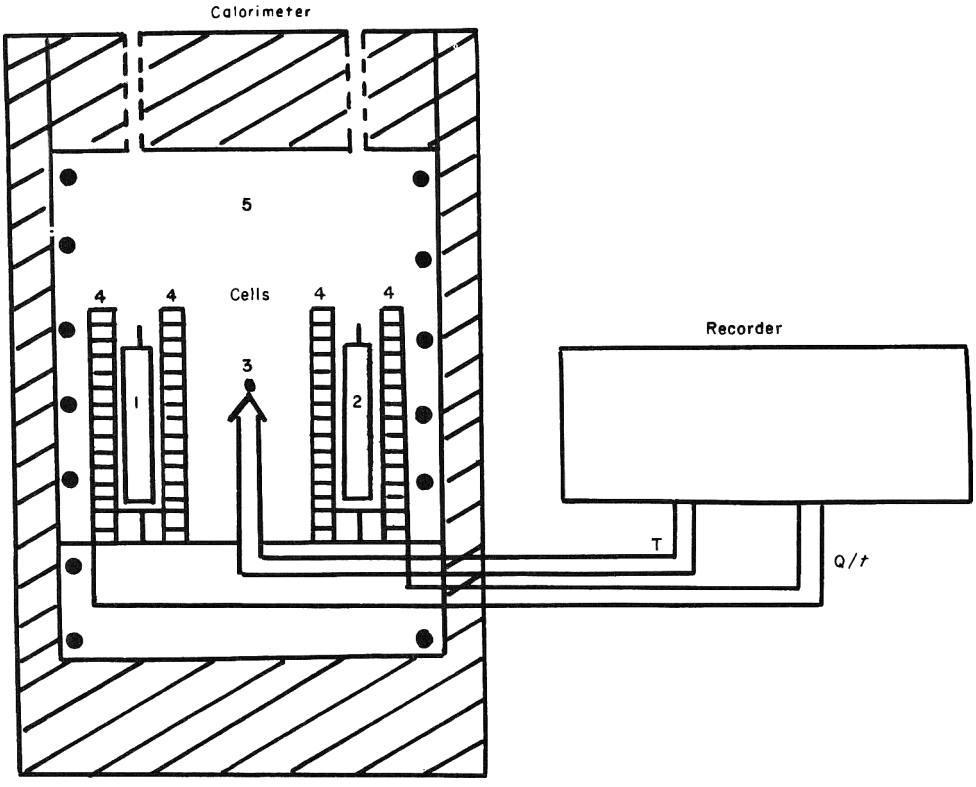


Figure 1. Schematic representation of the Setaram C80 heat flow calorimeter, 1 = Reference cell; 2 = sample cell; 3 = thermocouple; 4 = thermopiles; 5 = thermostatistically controlled enclosure.

Setaram C80 calorimeter is shown in Fig. 1. This instrument allows the analysis of relatively large quantities of non-homogeneous material (2.0–4.0 g whole coffee beans). The temperature range lies between ambient and 300°C. The samples are heated very slowly (generally at 1°C min^{-1}).

Specific heat measurements were performed in very thin-walled cells. For studying the thermal behaviour of the food products at high temperatures, different types of thick-walled cells, which support a pressure rise, were used. Calorimetric diagrams obtained with sealed cells were similar to those obtained when the samples were held under a constant pressure of 20–25 bar inert gas during heating; the reasons why are explained in detail elsewhere (Raemy, 1981).

For the measurements of coffee and chicory presented here, the samples were heated in sealed cells, the use of which is particularly easy; the products are analysed in presence of a fixed amount of oxygen.

Specific heat measurements

The calorimeter C80 allows the temperature to be increased by small amounts ΔT from any equilibrium temperature above ambient; the temperature rise is

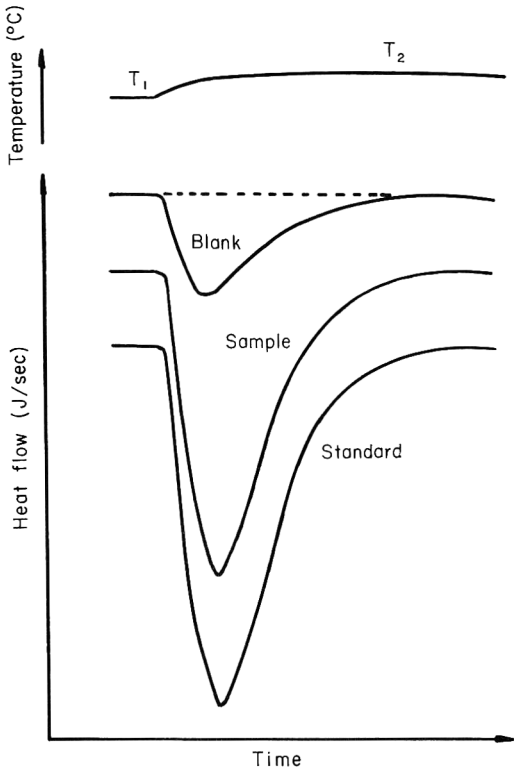


Figure 2. Specific heat determination by surface measurements.

reproduced very accurately. This allows the measurement of single values of the specific heat, for example at 30°C, by increasing the temperature from $T_1 = 30$°C to $T_2 = 33$°C. Two measurements (empty cell and sample) or three (empty cell, sample and standard) are necessary to determine the specific heat of a substance (Fig. 2). The reference cell is an empty piece of metal, built in such a way that all two or three curves produced have identical shapes.

One measurement of the sample and one of the empty cell allow the determination of the unknown specific heat value, at a fixed temperature. As ΔT is the same in both measurements, the following formula is obtained,

$$C_2 = \frac{Q_2 - Q_0}{m_2 \Delta T}, \tag{1}$$

where C_2 is the specific heat, under constant pressure, of the product, Q_0 is the required heat quantity (empty cell measurement), Q_2 is the required heat quantity (product measurement), m_2 is the mass of product and ΔT is the temperature increase from T_1 to T_2.

The heat quantity values corresponding to the surface between both equilibrium temperatures must be introduced in joules (or calories); it is therefore necessary to know the sensitivity curve (Fig. 3) of the calorimeter, determined by Joule effect.

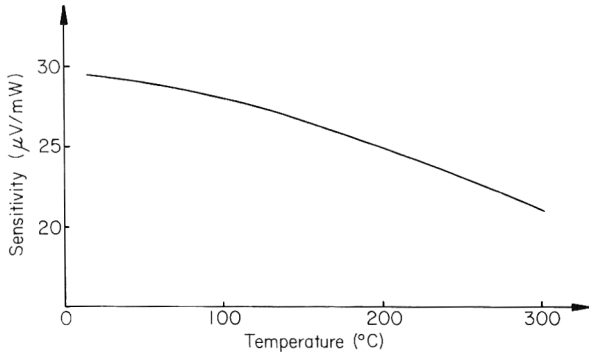


Figure 3. Sensitivity curve of the Setaram C80 heat flow calorimeter.

By performing a measurement of a standard, the specific heat value of the sample can be obtained directly by comparison,

$$C_2 = C_1 \frac{m_1}{m_2}\left(\frac{Q_2 - Q_0}{Q_1 - Q_0}\right), \tag{2}$$

where m_1 is the mass of standard substance, C_1 is the specific heat of standard substance and Q_1 is the required heat quantity (standard measurement) and where Q_0, Q_1, Q_2 can be introduced in any units (for example as surfaces in cm^2).

Although scientific tables and literature give specific heat values of such substances as KNO_3, Al_2O_3, benzoic acid, we use for our standard the Standard Reference Material 720 (synthetic sapphire) furnished by the National Bureau of Standards (U.S.A.).

Table 1. Specific heat values of coffee and chicory products at 30°C.

Sample description	Humidity (% wt)	C ($J\ g^{-1}\ °C^{-1}$)	C ($cal\ g^{-1}\ °C^{-1}$)
Green Arabica coffee beans from Mexico	7.5	1.85	0.44
Green Robusta coffee beans from Togo	4.5	1.46	0.35
Dried chicory roots from Austria	8	1.59	0.38
Roasted coffee beans	2.5	1.46	0.35
Roasted and ground coffee			
Low roast	2.6	1.46	0.35
Medium	2.4	1.42	0.34
High roast	2.2	1.42	0.34
Roasted and ground chicory	3.5	1.67	0.40
Soluble coffee powder	4.5	1.34	0.32
Soluble powder containing about 55% coffee and 45% chicory	2.5	1.46	0.35

Table 1 gives some specific heat values of coffee and chicory products obtained at 30°C: values between 1.2 J g^{-1} $°C^{-1}$ (or 0.3 cal g^{-1} $°C^{-1}$) and 2.0 J g^{-1} $°C^{-1}$ (or 0.5 cal g^{-1} $°C^{-1}$) were generally found for dry materials. For products like green coffee beans or soluble powders, small moisture differences have an appreciable influence on their specific heat values.

In a restricted temperature range, an increase of the specific heat values of solid foods is generally observed when temperature increases, as long as no physical or chemical changes occur. This can be controlled continuously by heating substance and empty cell at a constant rate: the deviation between both heat flow curves is proportional to the specific heat of the sample. Thus,

$$C_2 = \frac{\Delta U_2}{m_2 (\mathrm{d}T/\mathrm{d}t) S}, \tag{3}$$

where ΔU_2 is the calorimeter signal (between product and empty cell measurements), $\mathrm{d}T/\mathrm{d}t$ is the heating rate and S is the calorimetric sensitivity.

By performing a measurement of a standard as well, an equation similar to eqn (2) gives the unknown specific heat directly by comparing heights. Figure 4 shows such curves between 20 and 95°C; the specific heat of green Arabica coffee beans increases from 1.85 to about 2.4 J g^{-1} $°C^{-1}$; this represents a rate of about 30%.

For green coffee beans or coffee products there is also a weak endothermic phenomenon at about 60°C (fusion of one component?). It is clear that for food products which are in the form of fine powders, a more conventional differential scanning calorimeter like Mettler TA 2000B can also be used; methods are not very different and the results may even be obtained more quickly.

For liquid coffee or chicory extracts, specific heat values are much higher than for powders, due to the large amount of water. Following approximately the law of proportional additivity or Siebel's rule (Mohsenin, 1980), the

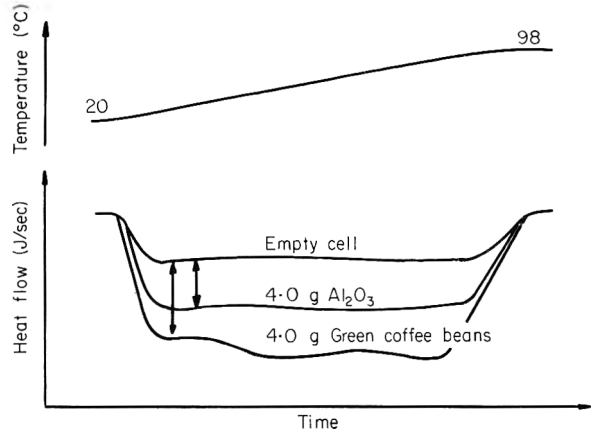


Figure 4. Specific heat determination by height measurements.

specific heat value, at 30°C, is about 3.0 J g^{-1} $°C^{-1}$ (0.7 cal g^{-1} $°C^{-1}$) for a concentrate extract (50% powder and 50% water) and approaches to 4.0 J g^{-1} $°C^{-1}$ (1.0 cal g^{-1} $°C^{-1}$) for very dilute solutions.

Specific heat values of liquid coffee extracts (Riedel, 1974) and of other foods (Riedel, 1977; Narain *et al.*, 1978; Putranon, Bowrey & Fowler, 1980; Hwang & Hayakawa, 1979; Levy, 1979; Keppeler, 1979) can be found in the literature: they were most often determined by calorimetric techniques. General compilation of thermal properties of foods and the methods used for their determination is given by Polley (1980) and Mohsenin (1980).

Results

Exothermic reactions

Samples of green coffee beans and of ground dried chicory roots were analysed by heating at constant rates, generally 1°C min^{-1}. The purpose of these measurements was to detect exothermic reactions, due to pyrolysis reactions.

With open cells at atmospheric pressure, the calorimetric curves showed an endothermic peak above 100°C (water phase transition) and at higher temperatures, an exothermic tendency. Measurements performed either under 20–25 bar pressure of inert gas (Raemy, 1981) or with sealed cells showed similar intensive exothermic phenomena. These reactions began at about 140°C for green coffee beans; the temperature range where they occurred was even lower for dried chicory. The measured enthalpies had values around 250–420 J g^{-1} (60–100 cal g^{-1}) for green coffee beans and values as high as 540–640 J g^{-1} (130–150 cal g^{-1}) for dried chicory. These exothermic phenomena correspond to roasting and carbonization of the products. Figure 5 presents the calorimetric curves of green Arabica coffee beans from Mexico and dried chicory from Austria.

Exothermic peaks were still evident in calorimetric diagrams of roasted coffee

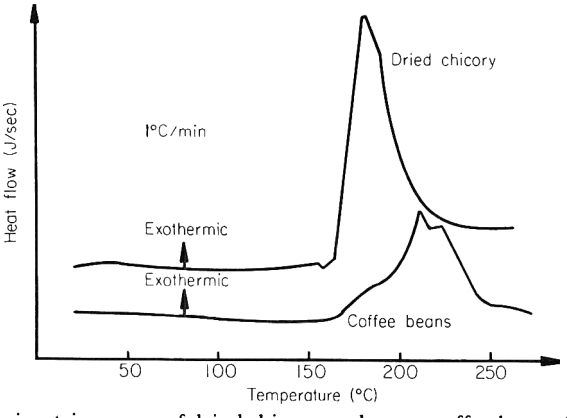


Figure 5. Calorimetric curves of dried chicory and green coffee beans (both heated in sealed cells).

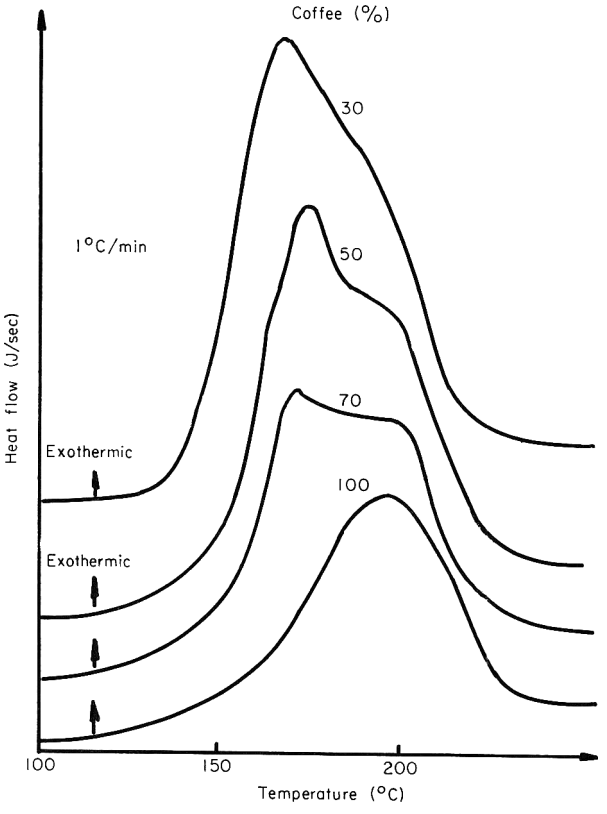


Figure 6. Calorimetric curves of soluble coffee and chicory powders (heated in sealed cells).

or chicory, and even those of soluble powders containing coffee, chicory or both components. Soluble powders (from pilot plant batches) with different percentages of coffee and chicory were compared (Fig. 6). When both components were present in the powder, the exothermic peak was a poorly resolved doublet; the first or left part is due primarily to chicory, the second to coffee. Enthalpies as high as 540–640 J g^{-1} (130–150 cal g^{-1}) were still measured for powders containing only chicory.

These exothermic phenomena are mainly attributed to the carbohydrates contained in both products. It is generally agreed that green coffee beans contain about 55% carbohydrate and that chicory contains about 75%. These values are little changed after roasting. Some of the carbohydrate is water soluble (about 40% of the total carbohydrate in coffee and 70% in chicory); this explains why exothermic reactions are still detected for the corresponding soluble powders. The remaining carbohydrate (60% for coffee) is found in the spent grounds. Figure 7 presents the calorimetric curves of a soluble coffee powder and of spent grounds, showing the highest peak for spent grounds. Such self-heating properties were also found with other foods having high contents of carbohydrate, e.g. most cereals. Figure 8 shows the calorimetric diagrams of wheat (variety: Scheppers) and whole rice (variety: Camolino).

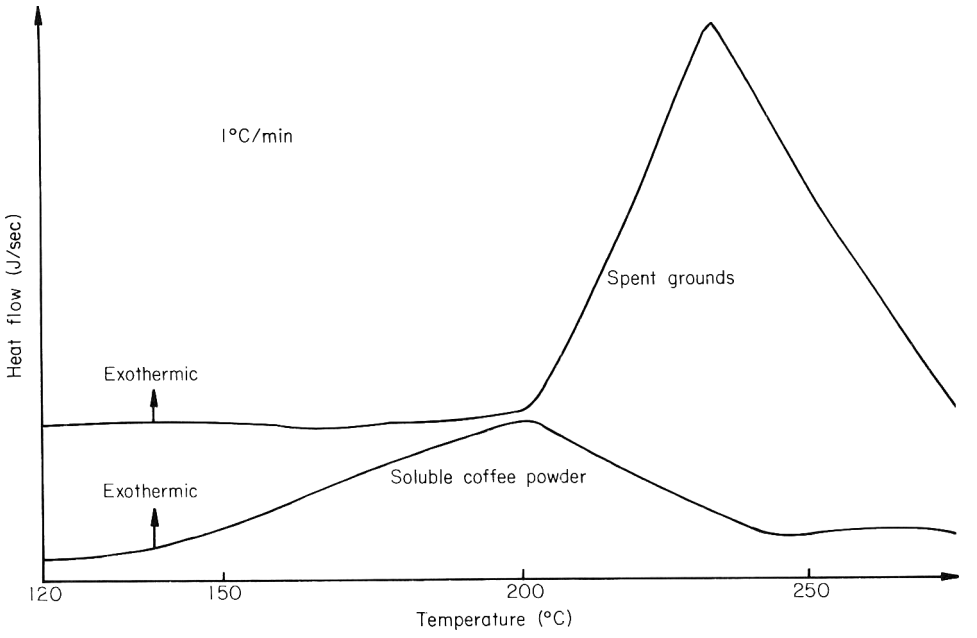


Figure 7. Calorimetric curves of a soluble coffee powder and of spent grounds (both heated in sealed cells).

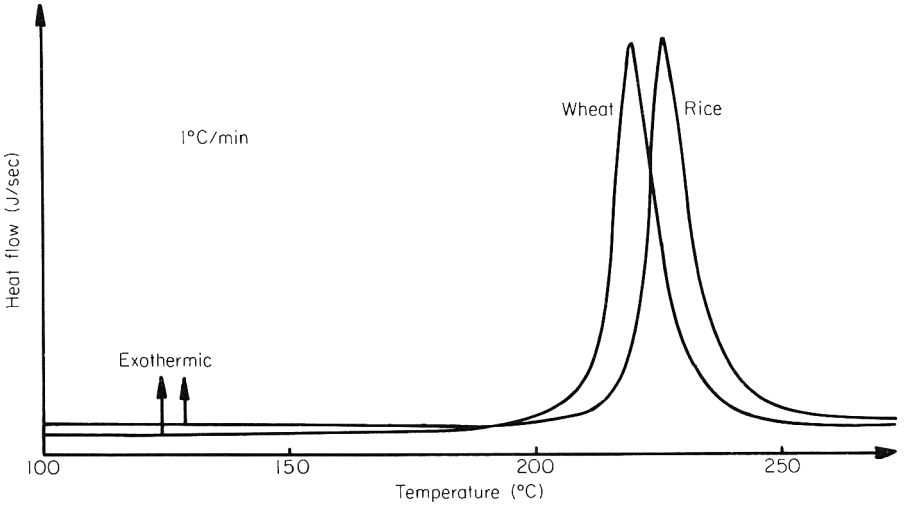


Figure 8. Calorimetric curves of wheat and whole rice (both heated in sealed cells).

Spontaneous heating and ignition

In an industrial operation, like high-temperature drying operations, exothermic reactions present a hazard. If heat generation always exceeds the heat loss, the material temperature will increase rapidly (this process is termed

self-heating or spontaneous heating) and the product will eventually ignite (spontaneous ignition). The following simple model, using our calorimetric data, should indicate how such phenomena could develop, for example, in a soluble chicory powder. If, for example, 1.0 g chicory powder is maintained at a temperature higher than the threshold of exothermic reactions, its temperature will increase due to spontaneous heating. The total amount of heat the powder can give up is 640 J g^{-1} (or 150 cal g^{-1}). The specific heat of this powder (at elevated temperatures) is about 2.0 J g^{-1} $°C^{-1}$ (or 0.5 cal g^{-1} $°C^{-1}$).

The maximum temperature rise which can occur is now obtained by calculating

$$\Delta T = \frac{Q}{mC} = \frac{640}{1\,2} = 320°C.$$

Even if a part of the heat is lost, because of other phenomena or non-adiabatic conditions, it seems possible that some of the powder can be brought above its minimum ignition temperature (Forsyth, 1980; Palmer, 1973) and that it will eventually ignite.

In the presence of water, a reduced heating rate can arise for three reasons: (1) the specific heat of the product is higher than in the absence of water; (2) heat is absorbed by water evaporation; and (3) in the case of an already smouldering fire, the reduction in dispersability, caused by the water, hinders flame propagation.

It is evident that more sophisticated models (Bowes, 1976) can be developed and that adiabatic calorimetry techniques (Townsend & Tou, 1980; Hub, 1981) could better show how, and how far, temperature will rise in cases of spontaneous heating (thermal runaway). These alternative methods would bring us interesting complementary data.

Conclusion

The analysis of coffee and chicory products by heat flow calorimetry has given information on the thermal behaviour of these foods. Such knowledge of food properties allows a better understanding of spontaneous heating, particularly in which temperature range this phenomenon can occur and to which consequences it can lead. This is of great importance for many high-temperature processing operations.

Acknowledgments

The authors are grateful to J. Loeliger and C. Desarzens for discussions, to Miss P. Clerc and J. P. Marquet for helping to perform the measurements and to R. F. Hurrell for improving their English.

References

Bowes, P.C. (1976) *Inst. Chem. Eng. Symposium Series No.* **47**.
Calvet, E. & Prat, H. (1956) *Microcalorimétrie*. Masson et Cie, Paris.
Forsyth, V.G. (1980) *Fire Prevention No. 135,* p.25.
Hemminger, W. & Höhne, G. (1979) *Grundlagen der Kalorimetrie*. Verlag Chemie, Weinheim.
Hub, L. (1981) *Instit. Chem. Eng. Symposium Series No.* **68,** 3.
Hwang, M.P. & Hayakawa, K.I. (1979) *J. Fd Sci.* **44,** 435.
Keppeler, R.A. (1979) *J. Dairy Sci.* **62,** 1340.
Levy, F.L. (1979) *J. Fd Technol.* **14,** 549.
Mohsenin, N.N. (1980) *Thermal Properties of Foods and Agricultural Materials*. Gordon and Breach, New York.
Narain, M., Bose Siripurapu, S.C., Jha, M. & Dwivedi, V.K. (1978) *J. Fd Sci. Technol.*, **15,** 18.
Palmer, K.N. (1973) *Dust Explosions and Fires*. Chapman and Hall, London.
Polley, S.L., Snyder, O.P. & Kotnour, P. (1980) *Fd Technol.* **34,** 76.
Putranon, R., Bowrey, R.G. & Fowler, R.T. (1980) *Fd Tech. Aust.* **32,** 56.
Raemy, A. (1981) *Thermochimica Acta,* **43,** 229.
Riedel, L. (1974) *Chem. Mikrobiol. Technol. Lebensm.* **4,** 108.
Riedel, L. (1977) *Chem. Mikrobiol. Technol. Lebensm.* **5,** 118.
Townsend, D.I. & Tou, J.C. (1980) *Thermochimica Acta,* **37,** 1.

(Received 26 August 1981)

J. Fd Technol. (1982) **17**, 461–466

The use of low temperatures and radiation to destroy Enterobacteriaceae and Salmonellae in broiler carcasses

R. W. A. W. MULDER

Summary

Experiments were carried out to determine the effect of a radiation dose of 2.5 kGy (250 krad) on the Enterobacteriaceae and Salmonellae contamination of broiler carcasses when applied at two radiation temperatures (+5°C and −18°C). After the treatments the carcasses were stored for 4 months at −18°C.

The storage period proved to have a considerably lethal effect on Enterobacteriaceae and Salmonellae. Salmonellae could be detected in the starting samples material as well as in the carcasses after 1 month of storage at −18°C. After a 1 month storage period no *Salmonella* was found in one of the two groups of low temperature and radiation-treated carcasses.

Introduction

The initial contamination of broilers with *Salmonella* is not removed by the slaughtering process. The end product, chilled or frozen, will remain contaminated. The major part of the Dutch broiler production is sold as frozen products. It has been shown that *Salmonella* can survive during frozen storage (Enkiri & Alford, 1971). Chilled poultry carcasses are also frequently found contaminated with these organisms (Gunnarson *et al.*, 1974; Mulder, Notermans & Kampelmacher, 1977; Van Schothorst *et al.*, 1974).

By applying a radiation treatment to chilled or frozen poultry carcasses a considerable reduction of the number of contaminated carcasses can be achieved. A dose of 6.0 kGy (600 krad) was found quite effective in this respect (Mossel, Van Schothorst & Kampelmacher, 1968), but changes in odour, colour and taste of the chilled products were observed. These side effects can be eliminated by performing the radiation treatment when the products were frozen (Brasch & Huber, 1948; Coleby *et al.*, 1961; Hannan & Shepherd, 1959; Wierbicki *et al.*, 1970).

Author's address: Spelderholt Institute for Poultry Research, Ministry of Agriculture and Fisheries, 7361 DA Beekbergen, The Netherlands.

0022–1163/82/0800–0461 $02.00

Because of the absence of oxygen during a radiation treatment under frozen conditions, radical formation is inhibited or at least delayed in the products. So no reactions causing the above-mentioned changes of the product take place.

In order to achieve an equivalent lethal effect, higher radiation doses are required for frozen products than for chilled products (Matsuyama, Thornley & Ingram, 1964). The ice layer in frozen products provides protection of the bacteria against the radiation. Presence of proteins and polysaccharides also may provide protection (Anon., 1968).

In this study, experiments were carried out to determine the effect of a radiation dose of 2.5 kGy (250 krad) on the Enterobacteriaceae and *Salmonella* contamination of broiler carcasses applied at two temperatures (+5°C and −18°C).

This radiation dose was chosen because of the expected approval by the Dutch Government to irradiate poultry at this level. According to results from literature (Bolder, Germs & Mulder, 1981) a storage period of a few months at a temperature of −18°C can have a considerable lethal effect on Enterobacteriaceae, but had little effect on the number of samples infected with Salmonellae. Notermans *et al.* (1975) found in their experiments a five-fold reduction in the numbers of Salmonellae present as a result of the freezing procedure applied. Therefore, the irradiated carcasses were stored at −18°C for a 4 month period.

Materials and methods

The experiments were carried out with 186 commercially slaughtered, hard-scalded, water-cooled broiler carcasses, which were kept at chill temperatures (+5°C). The broilers were slaughtered at 6 weeks of age; the average live weight was 1400 g. After slaughtering the carcasses were packed in polyethylene bags. The broiler flock proved *Salmonella* positive at 5 weeks of age.

The experimental set-up is given in Fig. 1. Skin samples from the neck, the breast and the cloaca area aseptically excised from the carcasses with sterile

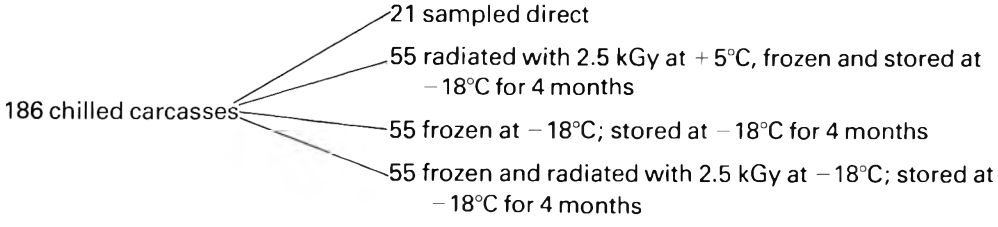


Figure 1. Estimation of the effect of additional storage at −18°C on the combined effect of radiation at +5°C, freezing and radiation at −18°C on Enterobacteriaceae and Salmonellae in broiler carcasses.

instruments (approx. 100 cm^2 or 10 g per carcass) were homogenized in a Colworth Stomacher with peptone saline solution (1:9) for 1 min and from this homogenate 10 ml was used in further examinations. Conventional procedures were used in the sampling of the drip water released after thawing the carcasses. The thawing of the carcasses was carried out under standardized conditions for 24 hr at +7°C. Every month of storage eleven carcasses were examined.

Salmonella isolation methods

Skin and drip water samples were resuscitated and pre-enriched (1:9) in 90 ml tryptone soy broth for 2 hr at 37°C and in buffered peptone water for 18–24 hr at 37°C respectively.

Subsequently 10 ml of the broth was transferred to two bottles each containing 90 ml tetrathionate brilliant green broth (Müller–Kauffmann) and to two bottles each containing 90 ml selenite cystine broth according to Stokes & Osborne (1955). The enrichment media were incubated for 24 and 48 hr at 37 and 43°C. Subcultures were made on the following media:

(1) Brilliant green phenol red agar (OXOID) (BG);
(2) BG + 0.1 $g/10^{-3}$ m^3 sulphadiazine + 2.5 $g/10^{-3}$ m^3 sodium-deoxycholate;
(3) BG sulphapyridine agar (DIFCO);
(4) XLD medium (OXOID);
(5) Hektoen Enteric agar (OXOID).

Incubation was at 37°C for 18–24 hr. Suspect colonies were subcultured on to McConkey agar (OXOID) and examined biochemically and serologically. Enterobacteriaceae were estimated in violet red bile glucose agar (OXOID) by using serial dilutions from the homogenate mentioned before. Layered poured plates were incubated during 18–24 hr at 37°C.

Radiation treatment

The broiler carcasses were packed in polystyrene boxes. Each box contained five frozen or chilled carcasses, which were individually packed in polyethylene bags. All carcasses were irradiated at the Proefbedrijf Voedselbestraling, Wageningen, The Netherlands. The source was cobalt-60 which had an intensity of 1.5 kGy (150 krad)/hr. The dose applied was 2.5 kGy (250 krad). During the radiation treatment the temperature of the carcasses increased by 1°C.

Results

Table 1 gives the results obtained with the treated carcasses with respect to the Enterobacteriaceae. A considerable decrease in the number of Enterobacteriaceae could be detected on carcasses which were irradiated in the chilled or frozen condition. On frozen carcasses there was a decrease in numbers estimated on the skin and also in the drip water.

Table 1. Enterobacteriaceae counts (log N) on broiler carcasses after different treatments and storage at −18°C

Storage period (months)	Chilled carcasses	Frozen carcasses				Frozen carcasses + 2.5 kGy (250 krad) and 2.5 kGy (250 krad) + frozen carcasses
	Skin sample	Skin sample	s.d.	Drip water	s.d.	
0	5.43	6.37	(0.26)	5.96	(0.43)	All counts
1		5.69	(0.77)	4.27	(0.35)	were less
2		5.69	(0.64)	5.52	(0.22)	than 1.00
3		5.45	(0.72)	3.85	(0.32)	
4		5.02	(1.11)	3.73	(0.58)	

s.d. = standard deviation.

Table 2 gives the results with respect to the *Salmonella*-positive carcasses after the same treatment and storage period. More Salmonellae were isolated from the drip water than from the skin samples. Irradiation has also with respect to Salmonellae an extra effect above freezing only. During the 4 months storage period the frozen carcasses proved positive every month, whereas the irradiated

Table 2. *Salmonella*-positive broiler carcasses after different treatments and storage at − 18°C

Samples	Storage period at −18°C (months)				
	0	1	2	3	4
Chilled carcasses (n = 21)*					
Skin samples	+(10)				
Drip water					
Frozen carcasses (n = 11)†					
Skin samples	+(11)	+(11)	+(11)	+(5)	+(6)
Drip water	+(11)	+(6)	+(11)	+(5)	+(11)
Frozen carcasses + 2.5 kGy (250 krad) (n = 11)‡					
Skin samples	—	+(5)	—	—	—
Drip water	+(10)	+(10)	+(1)	—	—
2.5 kGy (250 krad) + frozen carcasses (n = 11)§					
Skin samples	+(5)	—	—	—	—
Drip water	+(5)	—	—	—	—

* Serotypes: *agona, infantis, montevideo, münchen.*
† Serotypes: *agona, anatum, enteritidis, infantis, montevideo.*
‡ Serotypes: *heidelberg, montevideo, thompson.*
§ Serotypes: *montevideo, thompson.*

carcasses, irradiated in the chilled or frozen condition, were negative after 3 months. No *Salmonella* could be detected with any of the *Salmonella* isolation methods described.

Discussion and conclusions

The effect of irradiation on chilled and frozen carcasses with respect to the Enterobacteriaceae is evident. A dose of 2.5 kGy (250 krad) proved sufficient to reduce the numbers of these organisms to fewer than ten cells per gram or millilitre. With Salmonellae the results were less evident. Among carcasses a relatively low percentage positives was estimated. This could be caused by the small number of carcasses used every sampling time. It is also possible that with chilled carcasses the bacteria are more firmly attached to the skin and so they are not detected during the procedure for *Salmonella* isolation. It is evident that more *Salmonella* are isolated from drip water than from skin samples. This can be caused by the fact that the drip water samples were examined undiluted whereas the skin samples were diluted in peptone saline solution. Another reason may be the attachment of bacteria to the skin not detaching during enrichment of the samples, although this is not likely because of the bile salts present and the high temperature (43°C) applied during enrichment. Another factor may be the fact that the presence of a competitive microflora consisting of *Escherichia coli, Citrobacter, Proteus* and *Pseudomonas* species interferes with the detection of *Salmonella* on chilled carcasses.

The irradiation of the carcasses under chill conditions seems to give better results than applying the same process under frozen conditions. Radiation at chill temperatures resulted in *Salmonella*-negative samples after 1 month of storage, whilst irradiation after freezing gave *Salmonella*-negative samples after a 3 month storage period at −18°C.

From the literature where the influence of temperature on the radiation sensitivity has been described this is not surprising (Matsuyama *et al.*, 1964). However, the differences found in these experiments had no statistical significance and can be attributed to the relatively small number of carcasses used for each sampling in this experiment.

Irradiation and subsequent storage of the carcasses at − 18°C leads to a *Salmonella*-free product within 3 months of storage. Therefore, this process could be applied to broiler carcasses in attempts to reduce the spread of *Salmonella*.

References

Anon. (1968) *Radiation Preservation of Foods*. Publication no. **1273,** NAS/NRS, Washington D.C.

Bolder, N.M., Germs, A.C. & Mulder, R.W.A.W. (1981) In: *Quality of Poultry Meat* (Eds R. W. A. W. Mulder, C. W. Scheele & C. H. Veerkamp) p. 473. Spelderholt Institute for Poultry Research, Beekbergen, The Netherlands.

Brasch, A. & Huber, W. (1948) *Science,* **108,** 536.
Coleby, B., Ingram, M., Shepherd, H.J., Thornley, M.J. & Wilson, S.M. (1961) *J. Sci. Fd Agric.* **12,** 483.
Enkiri, N.K. & Alford, J.A. (1971) *Appl. Microbiol.* **21,** 381.
Gunnarson, A., Hurvell, B., Nordblom, B., Rutquist, L. & Thal, E. (1974) *Nord. Vet. Med.* **26,** 499.
Hannan, R.S. & Shepherd, H.J. (1959) *J. Sci. Fd Agric.* **10,** 286.
Matsuyama, A., Thornley, M.J. & Ingram, M. (1964) *J. appl. Bact.* **27,** 110.
Mossel, D.A.A., Van Schothorst, M. & Kampelmacher, E.H. (1968) IAEA, Vienna.
Mulder, R.W.A.W., Notermans, S. & Kampelmacher, E.H. (1977) *J. appl. Bact.* **42,** 179.
Notermans, S., Van Schothorst, M., Van Leusden, F.M. & Kampelmacher, E.H. (1975) *Tijdschr. Diergeneesk.* **100,** 648.
Stokes, J.L. & Osborne, W.E. (1955) *Appl. Microbiol.* **3,** 217.
Van Schothorst, M., Van Leusden, F.M., Edel, W. & Kampelmacher, E.H. (1974) *Tijdschr. Diergeneesk.* **99,** 815.
Wierbicki, E., Annelis, A., Killoran, J.J., Johnson, E.L., Thomas, M.H. & Josephson, E.S. (1970) *Proceedings 3rd International Congress Food Science and Technology,* Washington, 9–14 August.

(*Received 4 September 1981*)

J. Fd Technol. (1982) **17**, 467–470

Effect of simultaneous high intensity ultraviolet irradiation and hydrogen peroxide on bacterial spores

CATHERINE E. BAYLISS* AND W. M. WAITES

Summary

High intensity (1.8×10^3 mW/cm^2) ultraviolet light irradiation of spores of *Bacillus subtilis* in the presence of 2.5% (w/v) hydrogen peroxide resulted in a rapid kill. Higher concentrations of hydrogen peroxide protected spores against the effects of irradiation. The results are discussed in relation to the use of simultaneous ultraviolet irradiation and hydrogen peroxide treatment as a method of sterilization.

Introduction

Ultraviolet (u.v.) light irradiation of spores of *Bacillus subtilis* in the presence of hydrogen peroxide produces a rapid kill which is up to 2000-fold greater than that produced by irradiation alone (Bayliss & Waites, 1979a). This increased kill is slightly reduced in the presence of concentrations of H_2O_2 above 1.0% (w/v) and higher concentrations of H_2O_2 markedly reduced the kill (Bayliss & Waites, 1979b). We have reported that the synergism occurs at low u.v. (254 nm) doses (up to 10^2 mW/cm^2), although for some more resistant strains, such as *B. subtilis* NCDO 2130, mild heating was needed to produce the kill required for sterilization of packaging used in aseptic filling. Higher u.v. doses are available by using UV–C lamps (Bachmann, 1975) and in this study we have determined the spore kill produced by u.v. doses up to 1.8×10^3 mW/cm^2 in the presence or absence of H_2O_2 in order to see if such doses will increase the kill at higher concentrations of H_2O_2.

Methods

Organisms, maintenance of cultures, spore preparation and enumeration

The use of *B. subtilis* NCDO 2129 and NCDO 2130, which produce spores especially resistant to H_2O_2, has been described previously (Bayliss & Waites,

Authors' address: Agricultural Research Council, Food Research Institute, Colney Lane, Norwich NR4 7UA.

*To whom correspondence should be addressed.

0022–1163/82/0800–0467 $02.00

1979b). The cultures were maintained on nutrient agar (Oxoid) slopes. Spores were produced on *Bacillus* spore agar (BS) (NCDO 2129) or supplemented nutrient medium (NCDO 2130) as described by Bayliss, Waites & King (1981). Spore preparations contained at least 95% free, phase-bright spores. Viable spores were enumerated by diluting in glass-distilled water and plating on BS using a Spiral Plate Maker (Spiral Systems, Cincinnati, U.S.A.) (Bayliss & Waites, 1980). Cultures were incubated at 30°C and colonies counted after 16 and 40 hr.

Ultraviolet irradiation of spores

Spores were added to 0.1 M phosphate buffer pH 7.0 containing H_2O_2 (Analar, BDH) at concentrations up to 20% (w/v), which had been equilibrated at 20°C. Samples, at a total microscopic count of about 3×10^8/ml phase-bright spores, were exposed in 4.0 ml aliquots in 9.0 cm Petri dish bases to a high intensity u.v. source (UV–C 13–70, Brown, Boveri & Co. Ltd, 5401 Baden, Switzerland) for 20 sec and agitated during treatment to reduce shielding. Samples (2.0 ml) were heated to 80°C over 60 sec by adding to pre-heated, screw-capped bottles before adding catalase (2.0 ml) at a concentration sufficient to break down residual H_2O_2, plunging into ice and storing in ice for 5 min prior to enumeration.

Results

In the absence of H_2O_2 approximately 0.01% of spores of *B. subtilis* NCDO 2129 and NCDO 2130 survived irradiation at a dose of 1.8×10^3 mW/cm^2 (Fig. 1). In the presence of H_2O_2, higher kills were achieved but these were reduced at concentrations of H_2O_2 above about 2.5% (w/v); with *B. subtilis* NCDO 2129 spores more than 1.0% of the population survived irradiation in the presence of 20% (w/v) H_2O_2. Examination of Table 1 shows that exposure to high intensity u.v. irradiation, H_2O_2 or heat separately do not consistently produce the four log cycle kill which is the minimum required for aseptic packaging (Swartling & Lindgren, 1962). However, results obtained with spores of *B. subtilis* NCDO 2130 using combined u.v. and 2.5% (w/v) H_2O_2 followed by a short heat treatment demonstrate that a minimum five log cycle kill is obtainable (Table 1).

Discussion

The increased kill observed when spores of *B. subtilis* are exposed to mild u.v. (254 nm) irradiation and H_2O_2 is only evident when the treatments are simultaneous, not successive. In this study we have shown that a ten-fold increase in u.v. dose, compared to the maximum dose previously used (Bayliss & Waites, 1979b), increased the kill by u.v. plus H_2O_2 by ten-fold. The protective effect

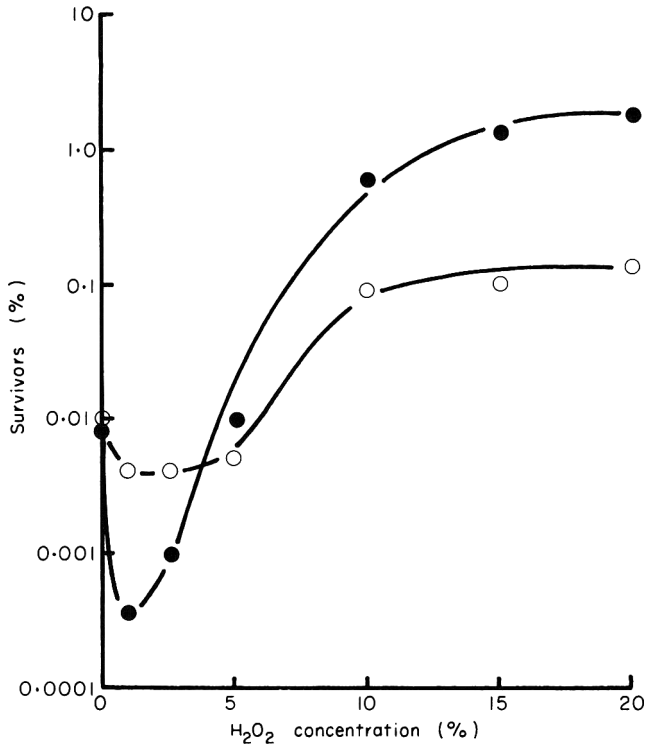


Figure 1. Effect of simultaneous ultraviolet irradiation and hydrogen peroxide on the viability of spores of *Bacillus subtilis*. Suspensions of spores of *B. subtilis* NCDO 2129 (●) and NCDO 2130 (○) were irradiated for 20 sec (total u.v. dose 1.8×10^3 mW/cm^2) in the presence of increasing concentrations of hydrogen peroxide as described in Methods.

Table 1. Effect of high intensity u.v. irradiation, hydrogen peroxide and heat on spores of *Bacillus subtilis*

	Survivors (%)	
Treatment	NCDO 2129	NCDO 2130
Ultraviolet irradiation*	0.01	0.008
Hydrogen peroxide†	81	97
Ultraviolet simultaneously with H_2O_2	0.005	0.001
Heat‡	145	127
H_2O_2 followed by heat	0.007	0.74
Ultraviolet + H_2O_2 followed by heat	0.00002	0.0003

* 4.0 ml samples were exposed to 1.8×10^3 mW/cm^2 u.v. irradiation over 20 sec.

† Samples were exposed to 2.5% w/v H_2O_2 for 60 sec.

‡ 2.0 ml samples were transferred to pre-heated bottles and heated to 80°C over 60 sec.

which occurs in the presence of higher concentrations of H_2O_2 (Bayliss & Waites, 1979a; see Fig. 2b) was also observed in these experiments, although the level of H_2O_2 which protected was increased slightly from 1 to 2.5% (w/v). It is surprising that such low concentrations of H_2O_2 protect spores against u.v. irradiation. However, H_2O_2 absorbs u.v. and if the action of u.v. itself on spores is important in their destruction, then at high H_2O_2 concentrations fewer spores will be killed. In addition, irradiation of H_2O_2 produces hydroxyl radicals (Symons, 1960) which may be responsible for the damaging effects of the combined treatment. Killing probably occurs more efficiently when free radicals are produced within spores (Baylis & Waites, 1979a) or vegetative cells (Repine, Fox & Berger, 1981). As the concentration of H_2O_2 is increased hydroxyl radicals will still be formed, but at higher H_2O_2 concentrations, decomposition will be terminated by reaction of breakdown products with H_2O_2 molecules (Symons, 1960) without spores taking part in the reaction.

These results have important consequences for combining u.v. and H_2O_2 for sterilization of surfaces since they show that a strict *upper* limit in H_2O_2 concentration must be observed for the synergism to be effective. With low doses of u.v. heat is required to kill the most resistant fraction of the spore population (Bayliss & Waites, 1979b; Cerf & Metro, 1977). If a high intensity u.v. lamp is used with a concentration of 2.5% (w/v) H_2O_2, consistent kills of greater than four log cycles of the most resistant bacterial spores can be achieved without heating although heating can provide an even bigger safety factor. We have also shown that fungal spores can be inactivated with u.v. and H_2O_2 (Peel & Waites, 1979). It is apparent, therefore, that the use of a high intensity u.v. lamp combined with low levels of H_2O_2 will allow rapid sterilization of packaging surfaces contaminated with fungal or bacterial spores.

Acknowledgments

We wish to record our thanks to Brown-Boveri U.K. for their generous loan of the UV–C lamp and M. J. Tatton and W. H. Worts for technical assistance.

References

Bachmann, R. (1975) *Brown Boveri Review*, **62**, 206.
Bayliss, C.E. & Waites, W.M. (1979a) *FEMS Microbiol. Letters*, **5**, 331.
Bayliss, C.E. & Waites, W.M. (1979b) *J. appl. Bact.* **47**, 263.
Bayliss, C.E. & Waites, W.M. (1980) *J. appl. Bact.* **48**, 417.
Bayliss, C.E., Waites, W.M. & King, N.R. (1981) *J. appl. Bact.* **50**, 379.
Cerf, O. & Metro, F. (1977) *J. appl. Bact.* **42**, 405.
Peel, J.L. & Waites, W.M. (1979) *Patent Specification U.K. 7901091.*
Repine, J.E., Fox, R.B. & Berger, E.M. (1981) *J. biol. Chem.* **256**, 7094.
Swartling, P. & Lindgren, B. (1962) *Rep. Milk Dairy Res., Alnarp, Sweden*, **66**.
Symons, M.C.R. (1960) In: *Peroxide Reaction Mechanisms* (Ed. J. O. Edwards) p. 137. Interscience, New York.

(Received 6 October 1981)

J. Fd Technol. (1982) **17**, 471–482

Factors controlling the growth of *Clostridium botulinum* types A and B in pasteurized cured meats

IV. The effect of pig breed, cut and batch of pork

ANGELA M. GIBSON, T. A. ROBERTS AND A. ROBINSON*

Summary

Pork slurries were prepared from leg or shoulder muscle from three animals from each of three breeds of pig (Pietrain, Gloucester Old Spot and Large White × Landrace cross). Slurries (pork:water, 1:1.5) contained $NaNO_2$ (100 μg/g), NaCl (2.5, 3.5 or 4.5% w/v on the water), were subjected to one of three heat treatments (unheated, 80°C for 7 min, 80°C for 7 min plus 70°C for 1 hr) and stored at 35, 20, 17.5 or 15°C for up to 6 months to determine the relative effects of the above factors on growth (spoilage) and toxin production by *Clostridium botulinum* types A and B at 10^3 spores per bottle.

Increasing salt or heat treatment, or decreasing storage temperature or inoculum level all reduced spoilage and toxin production. Both 'animal' and 'cut' significantly affected spoilage and toxin production. More spoilage and toxin production occurred in meat from the shoulder cut than from the leg cut. In both cases there was considerable variation between animals within breed, but there was no systematic difference between breeds. There is no obvious explanation for the variation in meat between animals, but it should be borne in mind when planning and assessing results of large multifactor experiments. Although there was more spoilage and toxin production after 6 months' than 3 months' storage, the statistical analyses yielded essentially similar conclusions.

Introduction

The possible formation in cured meat products of carcinogenic nitrosamines (Status Report, 1972) led to pressures to reduce drastically the amount of nitrite used and considered by the industry essential for colour (Fiddler *et al.*, 1973; Fujimaki, Emi & Okitani, 1975), flavour (Cho & Bratzler, 1970; Wasserman & Talley, 1972; Kueper & Trelease, 1974; Mottram & Rhodes, 1974) and bacteriological stability and safety, particularly against *Clostridium botulinum* (Steinke

Authors' addresses: Agricultural Research Council, Meat Research Institute, Langford, Bristol BS18 7DY, and *School of Mathematics, University of Bath, Claverton Down, Bath BA2 7AY.

0022–1163/82/0800–0471

& Foster, 1951; Blanche–Koelensmid & van Rhee, 1968; Greenberg, 1972; Ingram, 1974, 1976). Several factors in cured meat products combine to prevent the growth of spores of *Cl. botulinum* which survive pasteurization, and experiments sufficiently large to study most of these factors demand relatively large quantities of bacteriological media or pork. Although bacteriological media can be standardized, at least within a manufactured batch, similar experience using pork as a growth medium is generally lacking, but there have been several reports that experimental variation could be attributed to differences between batches of pork (Rhodes & Jarvis, 1976) even in meat formulations (Tompkin, Christiansen & Shaparis, 1977; Sofos *et al.*, 1979).

In our previous studies the large amount of pork needed for 'low' pH slurries, and the anticipated duration of that work – several years – precluded stockpiling and mixing in one operation all the meat required (Roberts, Gibson & Robinson, 1981a). Similar experiments on 'high' pH slurries were reduced in size and pork was stockpiled and mixed in a single batch (Roberts, Gibson & Robinson, 1981b). In neither study was any attempt made to use pork from a single breed.

This paper reports an attempt to assess the importance in such large experiments of variation between batches of pork, by studying two 'cuts' of meat (leg and shoulder) within and between three breeds of pig.

Materials and methods

Pork muscle was obtained from three animals from each of three breeds: Gloucester Old Spot, Pietrain and Large White × Landrace cross. Animals were numbered 1–9, three from each breed, and leg and shoulder muscles taken from each animal. The pork was defatted by hand, minced and stored, and slurries prepared as described by Roberts *et al.* (1981a).

Experimental plan

NaCl (% w/v on the water)	2.5, 3.5, 4.5
$NaNO_2$ (μg/g slurry)	100
Heat treatment	0, 80°C/7 min
	80°C/7 min + 70°C/1 hr
Inoculum (spores per bottle)	10^3

Table 1 lists the slurries prepared, and Table 2 summarizes the chemical analyses of slurries at time of preparation.

Three salt levels were used to give a range of probabailities of spoilage and toxin production. Other variables were kept to a minimum, i.e. nitrite was only used at 100 μg/g level, and nitrate, isoascorbate and polyphosphate were not included. Toxin tests were carried out as described by Roberts *et al.* (1981a).

Table 1. List of slurries prepared

	All slurries contained 100 μg/g $NaNO_2$			
Slurry	Animal No.	Breed	Meat cut	NaCl (% w/v on water)
181	1	Pietrain	Leg	2.5
182				3.5
183				4.5
190			Shoulder	2.5
191				3.5
192				4.5
187	2	Pietrain	Leg	2.5
188				3.5
189				4.5
184			Shoulder	2.5
185				3.5
186				4.5
193	3	Pietrain	Leg	2.5
194				3.5
195				4.5
196			Shoulder	2.5
197				3.5
198				4.5
202	4	Gloucester	Leg	2.5
203		Old Spot		3.5
204				4.5
199			Shoulder	2.5
200				3.5
201				4.5
205	5	Gloucester	Leg	2.5
206		Old Spot		3.5
207				4.5
208			Shoulder	2.5
209				3.5
210				4.5
211	6	Gloucester	Leg	2.5
212		Old Spot		3.5
213				4.5
214			Shoulder	2.5
215				3.5
216				4.5
217	7	Large White ×	Leg	2.5
218		Landrace		3.5
219				4.5

Table 1 – *continued*

	All slurries contained 100 μg/g $NaNO_2$			
Slurry	Animal No.	Breed	Meat cut	NaCl (% w/v on water)
220			Shoulder	2.5
221				3.5
222				4.5
223	8	Large White ×	Leg	2.5
224		Landrace		3.5
225				4.5
226			Shoulder	2.5
227				3.5
228				4.5
229	9	Large White ×	Leg	2.5
230		Landrace		3.5
231				4.5
232			Shoulder	2.5
233				3.5
234				4.5

Table 2. Chemical analysis of slurries immediately after preparation

Slurry	pH value	NaCl (% w/v)		Nitrite (μg/g)		Fat (%)	Water (%)
		Added	Detected	Added	Detected		
181	5.60	2.5	2.40	100	53	1.60	87.86
182	5.58	3.5	3.40	100	52	1.59	87.05
183	5.57	4.5	4.00	100	50	1.56	86.33
184	6.01	2.5	1.82	100	73	2.13	87.74
185	6.06	3.5	2.90	100	66	2.19	86.70
186	6.03	4.5	3.76	100	64	2.02	86.17
187	5.50	2.5	2.13	100	53	1.37	87.74
188	5.55	3.5	3.18	100	56	1.29	87.03
189	5.56	4.5	4.07	100	57	1.21	86.52
190	5.72	2.5	2.30	100	68	1.94	88.26
191	5.89	3.5	3.11	100	61	2.15	87.29
192	5.86	4.5	4.02	100	55	1.98	87.33
193	5.76	2.5	2.30	100	59	1.56	87.21
194	5.74	3.5	3.09	100	52	1.64	87.17
195	5.66	4.5	4.11	100	57	1.58	86.26
196	5.86	2.5	2.31	100	66	2.02	88.20
197	5.86	3.5	2.99	100	71	1.74	87.82
198	5.87	4.5	4.24	100	69	1.71	86.89
199	5.87	2.5	2.21	100	48	1.58	89.51
200	5.89	3.5	2.84	100	54	1.83	87.10

Table 2 – *continued*

Slurry	pH value	NaCl (% w/v) Added	NaCl (% w/v) Detected	Nitrite (μg/g) Added	Nitrite (μg/g) Detected	Fat (%)	Water (%)
201	5.84	4.5	4.24	100	55	1.76	86.70
202	5.78	2.5	2.44	100	60	1.05	89.32
203	5.66	3.5	2.99	100	60	0.97	87.98
204	5.65	4.5	4.11	100	58	0.95	87.15
205	5.66	2.5	2.32	100	52	1.15	88.38
206	5.65	3.5	3.28	100	59	1.05	87.81
207	5.71	4.5	4.53	100	58	1.07	86.77
208	5.88	2.5	2.81	100	65	1.96	87.57
209	5.86	3.5	3.93	100	71	2.06	86.95
210	5.83	4.5	4.51	100	73	1.67	86.69
211	5.71	2.5	2.43	100	53	1.32	87.65
212	5.70	3.5	3.39	100	52	1.13	87.17
213	5.68	4.5	4.17	100	50	1.08	86.76
214	5.88	2.5	2.51	100	53	1.94	87.42
215	5.75	3.5	3.06	100	49	1.83	86.83
216	5.75	4.5	4.24	100	51	1.83	86.14
217	5.76	2.5	2.53	100	56	0.89	88.09
218	5.60	3.5	3.05	100	61	1.01	87.21
219	5.60	4.5	4.24	100	61	0.97	86.47
220	5.85	2.5	2.42	100	63	1.85	87.49
221	5.85	3.5	3.57	100	66	1.77	86.82
222	5.81	4.5	4.29	100	57	1.74	86.59
223	5.81	2.5	2.52	100	58	0.74	88.77
224	5.81	3.5	3.34	100	62	0.78	88.14
225	5.81	4.5	4.31	100	59	0.85	87.02
226	6.05	2.5	2.49	100	59	1.37	88.20
227	5.68	3.5	3.26	100	64	0.94	87.97
228	5.63	4.5	4.15	100	57	1.13	87.06
229	5.55	2.5	2.53	100	59	1.35	87.47
230	5.54	3.5	3.47	100	59	1.33	86.78
231	5.55	4.5	4.27	100	65	1.40	85.94
232	5.63	2.5	2.40	100	78	2.00	87.45
233	5.60	3.5	3.32	100	79	1.99	86.23
234	5.62	4.5	4.35	100	73	1.79	85.99

Statistical analyses

The analyses classified factors into animals (Nos 1–9), cut ('leg'/'shoulder') and heat treatment (unheated; LOW, 80°C/7 min; HIGH, 80°C/7 min + 70°C/1 hr), and variables: salt level and storage temperature. There were nine animals in three groups of three breeds (Table 1).

Spoilage data. Analysis of variance on the area under the 'step function' were carried out (Roberts *et al.*, 1981a) on data collected after storage for 3 and 6 months.

Toxin data. The proportion of samples containing toxin at each treatment combination was analysed using a logistic regression (Roberts, Gibson & Robinson, 1981c) on data after storage for 3 and 6 months.

Results

Analysis of variance of spoilage data (Tables 3.1–3.5)

In the 3 and 6 month analyses both 'cut' and 'animal' were significant (Tables 3.1 and 3.2). Heat treatment, salt level and storage temperature were all significant (Table 3.3), as in earlier work (Roberts *et al.*, 1981a, b, c). Storage

Table 3.1. Analysis of variance on spoilage data. 1. Comparison of leg (mean pH 5.66) and shoulder (mean pH 5.9) cuts

	3 months		6 months		
Cut	Mean	LSD* ($P = <0.01$)	Mean	LSD ($P = <0.01$)	Replicates
Leg	140†		338		
Shoulder	165	4	395	8.4	324

*Means are significantly different when they differ by more than the least significant difference (LSD).

†Figures given are the means of the 'area under the step function' (Roberts *et al.*, 1981a), i.e. the smaller the number the less spoilage.

Table 3.2. Analysis of variance on spoilage data. 2. Comparison of animals and breed

		3 months			6 months			
Breed	Animal No.	Mean	Breed mean	LSD* ($P = <0.01$)	Mean	Breed mean	LSD ($P = <0.01$)	Replicates
Pietrain	1	146†	140	8.4	344	337	18	72
	2	128			321			
	3	147			345			
Gloucester Old Spot	4	151	158	8.4	373	381	18	72
	5	147			352			
	6	175			417			
Large White × Landrace	7	179	159	8.4	427	381	18	72
	8	154			378			
	9	143			339			

*Key as in Table 3.1.

Table 3.3. Analysis of variance on spoilage data. 3. The effect of increasing salt, heat treatment or storage temperature

Treatment	3 months		6 months		
	Mean	LSD* ($P = <0.01$)	Mean	LSD ($P = <0.01$)	Replicates
Salt (%)					
2.5	172†		404		
3.5	150		364		
4.5	135	5	331	10	216
Heat treatment					
UH	155		377		
LOW	167		394		
HIGH	136	5	327	10	216
Storage temperature (°C)					
15	97		226		
17.5	104		247		
20	135		352		
35	273	6	640	12	162

* Key as in Table 3.1.

temperature was the most significant single factor, with 'cut' the next most significant. In both the 3 and 6 month analyses there was significantly more spoilage in the shoulder cut than the leg (Table 3.1), probably because the pH level of the shoulder muscle was consistently higher (mean 5.9) than the 'leg' (mean 5.66). Overall, pork from the Pietrain breed spoiled significantly less than that from the other two breeds, but there was considerable variation between animals within a breed (Table 3.2). The rank order of the spoilage means for each animal was similar after 3 and 6 months' storage (Table 3.2).

The three most significant interactions all involved the 'cut' of meat:

Cut × *animal.* There was a greater difference in spoilage between the shoulder and the leg cuts from the Pietrain breed (Animal Nos 1–3) than between the cuts from the other two breeds (Table 3.4).

Cut × *heat treatment* (Table 3.4). At both 3 and 6 months, there was significantly more spoilage in the 'shoulder' cut after LOW heat treatment, and significantly less spoilage in the 'leg' after HIGH heat treatment.

Cut × *storage temperature.* The difference between spoilage of the shoulder and the leg cuts, 'shoulder' always being the greater, increased with increasing storage temperature (Table 3.4).

Heat treatment × *storage temperature.* Spoilage was significantly less after HIGH heat treatment followed by storage at 15 or 17.5°C, at both 3 and 6 months' storage. Irrespective of heat treatment, spoilage at 35°C was greater than at 20°C, and greater at 20°C than at 17.5 or 15°C (Table 3.5).

Other interactions although statistically significant were small compared with the single main effects.

Table 3.4. Analysis of variance on spoilage data. 4. Significant interactions

Interaction		3 months Leg	3 months Shoulder	LSD* ($P = <0.01$)	6 months Leg	6 months Shoulder	LSD ($P = <0.01$)	Replicates
Cut × animal								
PIE	1	112†	180		275	412		
	2	110	146		265	377		
	3	131	163		314	375		
GOS	4	141	161		355	391		
	5	130	164		315	389		
	6	170	180		401	433		
L × L	7	180	179		434	421		
	8	147	161		464	393		
	9	137	149	12	312	365	26	36
Cut × heat treatment								
UH		152	157		367	387		
LOW		153	180		364	425		
HIGH		115	157	7	281	373	15	108
Cut × storage temperature (°C)								
15		94	100		213	239		
17.5		99	109		226	268		
20		122	148		317	388		
35		244	302	8	594	686	17	81

Key as in Table 3.1.
PIE = Pietrain breed.
GOS = Gloucester Old Spot.
L × L = Large White × Landrace cross.

Table 3.5. Analysis of variance on spoilage data. 5. Significant interaction: heat treatment × storage temperature

Storage temp. (°C)	3 months UH‡	3 months LOW	3 months HIGH	LSD* ($P =$ <0.01)	6 months UH	6 months LOW	6 months HIGH	LSD ($P =$ (<0.01)	Replicates
15	100†	104	87		228	244	205		
17.5	107	116	90		255	268	218		
20	132	147	127		372	355	330		
35	280	300	239	10	653	712	555	21	54

Key as in Table 3.1.
‡Heat treatment (°C).

Logistic regression analysis of toxin data

All single factors were significant, i.e. 'animal', 'cut', storage temperature, salt level and heat treatment, paralleling the analyses of variance on the spoilage data. The analyses of deviance (Table 4) illustrate the relative importance of the factors and interactions, the greater the tabulated value of 'deviance' the greater the contribution of that source of variation (factor) to the overall control of toxin production. Storage temperature and salt level were the two

Table 4. Analysis of deviance on factors controlling toxin production by *Cl. botulinum* in pork slurries

Source of variation	Degrees of freedom		Deviance*		Mean squares		Significance ($P = <0.01$)	
	6†	3	6	3	6	3	6	3
Animal (Nos 1–9)	8	8	55	53	7	7	S‡	S
Cut (leg/ shoulder)	1	1	142	104	142	104	S	S
Salt	1	1	170	169	170	169	S	S
Storage temperature	1	1	439	709	439	709	S	S
Heat treatment	2	2	103	120	52	60	S	S
Cut × animal	8	8	46	48	6	6	S	S
Salt × heat treatment	—	2	—	10	—	5	NS§	S
Heat × storage temperature	2	2	78	63	39	32	S	S
Salt × storage temperature	—	1	—	0	—	—	NS	NS
Animal × storage temperature	8	8	83	80	10	10	S	S
Animal × heat treatment	16	16	83	81	5	5	S	S
Cut × salt	—	1	—	6	—	6	NS	(S)¶
Cut × storage temperature	1	1	9	24	9	24	S	S
Heat treatment × salt × storage temperature	—	2	—	22	—	11	NS	S
(storage temperature)^2	1	1	414	535	414	535	S	S
(salt)^2	1	1	11	8	11	8	S	S

*The relative importance of factors or interactions significantly affecting toxin production are reflected by the magnitude of figures in the deviance column.

†Months.

‡S = significant.

§NS = not significant.

¶Significant when $P = <0.05$, but not significant when $P = <0.01$.

most significant factors controlling toxin production, while 'cut' was again more significant than 'animal'. Significantly more samples containing toxin were obtained from the 'shoulder' than the 'leg'. Relatively more toxin production occurred in the Large White × Landrace cross than in Pietrain or Gloucester Old Spot. Although statistically significant, these differences between 'animal' (and breed) were small in comparison with 'cut', salt and storage temperature, and are unlikely to be relevant commercially. In the logistic regression there was a large quadratic effect of storage temperature and a significant, but small quadratic effect of salt (Table 4).

Generally the analyses of deviance at 3 and 6 months agree well. The relative order of significance of the factors remained similar although the salt × heat treatment, salt × cut and heat treatment × storage temperature interactions which were just significant at 3 months were not significant at 6 months ($P = < 0.01$). There was also a small but significant salt × heat treat ment × storage temperature interaction.

Discussion

There was good agreement between the analyses of variance of the spoilage data and analyses of the toxin data by logistic regression. All five factors studied (i.e. salt level, heat treatment, storage temperature, cut of meat ('leg' or 'shoulder') and animal (Nos 1–9)) were significant in both analyses although the relative order of significance differed. Storage temperature was always the most significant single factor affecting spoilage or toxin production and 'animal' the least significant. Salt level, 'cut' and heat treatment varied in their order of significance but 'cut' was always more significant than 'animal'. Significantly more spoilage and toxin production always occurred in the shoulder cut (mean pH 5.9) than the leg cut (mean pH 5.66). In earlier work, pH was one of the most significant variables affecting spoilage and toxin production (Roberts, Gibson & Robinson, 1982), and most probably accounts for this difference.

Although the effect of 'animal' was small compared with the other main effects it was still highly significant ($P = < 0.01$). When the overall means of each animal were compared (Table 3.2) the rank of those means were not ordered according to breed, and it is therefore concluded that the variation between animals of the same breed was greater than the variation between breeds. In this study only nine animals (three from each breed) were compared. Although the lowest mean was in the Pietrain breed (Animal No. 2) means for Pietrain animal Nos 1 and 3 were not significantly different from those of Animal Nos 4 and 5 of Gloucester Old Spot, and 8 and 9 of Large White × Landrace ($P = < 0.01$). A larger number of animals from each breed would have to be compared to establish firmly any effect of breed.

Probabilities of toxin production for all combinations of factors were calculated separately for each animal for 3 and 6 months' storage. One of the six tables so produced is shown as an example (Table 5). There was reasonably

Table 5. Predicted probabilities (calculated as a percentage) of toxin production for Animal Nos 1–9; 4.5% salt (w/v on the water), 100 μg/g nitrite after 3 months' storage

Cut	Animal No.	UH*				LOW				HIGH			
		15†	17.5	20	35	15	17.5	20	35	15	17.5	20	35
Leg	1	0‡	0	1	15	0	3	24	79	0	2	15	33
	2	0	2	13	45	1	14	58	87	1	7	36	38
	3	0	3	25	69	1	10	50	85	1	10	45	51
	4	0	5	26	36	1	12	49	56	1	13	45	20
	5	0	1	9	36	0	5	33	69	1	9	42	44
	6	0	4	28	83	1	18	70	96	2	18	64	82
	7	0	7	44	91	2	24	76	97	3	31	78	90
	8	1	9	45	73	1	11	49	72	1	8	35	26
	9	1	15	61	88	2	19	66	88	0	5	28	25
Shoulder	1	0	3	23	84	4	41	88	99	3	34	81	93
	2	0	3	20	62	2	20	70	93	1	11	48	54
	3	1	9	49	88	2	23	74	95	2	24	69	77
	4	0	5	30	45	1	14	54	65	2	15	50	27
	5	0	4	29	72	1	17	65	91	3	28	74	78
	6	0	8	48	93	3	34	84	99	3	34	81	93
	7	0	6	40	91	1	20	73	97	2	26	75	90
	8	2	22	70	90	2	25	73	90	2	19	61	54
	9	2	28	78	95	3	33	81	95	1	11	46	47

* Heat treatments

† Storage temperature (°C).

‡ Percentages calculated to nearest whole number.

good agreement for probabilities of toxin production between animals, when all other factors were constant, but occasional large differences occurred. There is no obvious explanation for such variation, assuming it to be the consequence of differences in meat from different animals, even within the same breed.

There is little published information on the effect of fat level on toxin production by *Cl. botulinum* in pork slurries or pork products. Tompkin, Christiansen & Shaparis (1978a) stated that fat content did not influence botulinal inhibition in their canned, comminuted pork product but offered no supporting data. Fat levels of *ca.* 4 and 12%, compared in a pasteurized pork slurry (Rhodes, 1979), did not significantly affect toxin production by *Cl. botulinum*. Hence variation in toxin production is unlikely to be the consequence of the small differences in fat concentration (0.75–2.19%) (Table 2). Excess iron has also been reported to reduce the antibotulinal effect of nitrite (Tompkin, Christiansen & Shaparis, 1978b, 1979). Iron content was not monitored throughout this work and it would be prudent, in future, to analyse for iron to establish whether differences in iron content of the meat could be responsible for observed differences in toxin production in meat from different animals.

The quadratic effect of salt and temperature also were high significant in these analyses confirming the effect first apparent in our study on the effect of potassium sorbate (Roberts *et al.*, 1982). Currently the massive data base from the 'low' and 'high' pH studies (Roberts *et al.*, 1981a, b) is being studied to seek similar effects.

There was considerably more spoilage and toxin production after 6 months' than after 3 months' storage but the good agreement between the two sets of results indicate that the shorter storage period would be adequate for future experiments, without losing important information and reducing costs, effort and time.

Acknowledgments

This work was funded by the Department of Health and Social Security. The authors wish to thank Mr. R. A. Pinches and Mr. G. E. Andrew for technical assistance.

References

Blanche-Koelensmid, W.A.A. & van Rhee, R. (1968) *Antonie van Leeuwenhoek,* **34,** 287.
Cho, I.C. & Bratzler, L.J. (1970) *J. Fd Sci.* **35,** 668.
Fiddler, W.J.W., Pensabene, J.W., Kushnir, I. & Piotrowski, E.G. (1973) *J. Fd Sci.* **38,** 714.
Fujimaki, M., Emi, M. & Okitani, A. (1975) *Agric. Biol. Chem.* **39,** 371.
Greenberg, R.A. (1972) *Proc. Meat Ind. Res. Conf., Chicago (1973),* p. 25. Am. Meat Inst. Found., Arlington, Va.
Ingram, M. (1974) *Proc. Int. Symp. Nitrite in Meat Prod.* (Eds B. Krol & B. J. Tinbergen) Zeist, 1973, p. 74. PUDOC, Wageningen.
Ingram, M. (1976) In: *Microbiology of Agriculture, Fisheries and Food,* Society of Applied Bacteriology Symposium Series No. 4 (Eds F. A. Skinner & J. G. Carr) p. 1. Academic Press, London.
Kueper, T.V. & Trelease, R.D. (1974) *Proc. Meat Ind. Res. Conf. Chicago,* p. 69. Am. Meat. Inst. Found., Arlington, Va.
Mottram, D.S. & Rhodes, D.N. (1974) *Proc. Int. Symp. Nitrite Meat Prod.* (Eds B. Krol & B.J. Tinbergen) Zeist, 1973, p. 161. PUDOC, Wageningen.
Rhodes, A.C. (1979) The effect of curing and processing parameters on the survival of *Clostridium botulinum* in cured meat and fish. Ph.D. Thesis, University of Reading.
Rhodes, A.C. & Jarvis, B. (1976) *J. Fd Technol.* **11,** 13.
Roberts, T.A., Gibson, A.M. & Robinson, A. (1981a) *J. Fd Technol.* **16,** 239.
Roberts, T.A., Gibson, A.M. & Robinson, A. (1981b) *J. Fd Technol.* **16,** 267.
Roberts, T.A., Gibson, A.M. & Robinson, A. (1981c) *J. Fd Technol.* **16,** 337.
Roberts, T.A., Gibson, A.M. & Robinson, A. (1982) *J. Fd Technol.* **17,** 307.
Sofos, J.N., Busta, F.F., Bhothipaksa, K. & Allen, C.F. (1979) *J. Fd Sci.* **44,** 668.
Status Report (1972) *J. Fd Sci.* **37,** 989.
Steinke, P.K.W. & Foster, E.M. (1951) *Fd Res.* **16,** 477.
Tompkin, R.B., Christiansen, L.N. & Shaparis, A.B. (1977) *J. Fd Sci.* **42,** 1046.
Tompkin, R.B., Christiansen, L.N. & Shaparis, A.B. (1978a) *Appl. Environ. Microbiol.* **35,** 886.
Tompkin, R.B., Christiansen, L.N. & Shaparis, A.B. (1978b) *J. Fd Technol.* **13,** 521.
Tompkin, R.B., Christiansen, L.N. & Shaparis, A.B. (1979) *Appl. Environ. Microbiol.* **37,** 351.
Wasserman, A.E. & Talley, F. (1972) *J. Fd Sci.* **37,** 336.

(25 October 1980)

J. Fd Technol. (1982) **17**, 483–498

The preparation and assessment of spun fibres from fish proteins using a wet spinning process

I. M. MACKIE* AND B. W. THOMSON

Summary

A process is described for preparing spun protein fibres from fish muscle proteins using as raw material filleting waste of cod (*Gadus morhua*) and whole blue whiting (*Micromesistius poutassou*). The flesh from these species was extracted with alkali at 0°C and the proteins, after isolation by isoelectric precipitation, were redissolved in alkali to give the spinning solution. The fibres were obtained by extrusion into an acetate precipitation bath and were analysed chemically and assessed by taste panels. The data obtained are discussed in relation to the potential application of this process as one way of upgrading fish waste and under-utilized species.

Introduction

An examination of the utilization of the world's fish catch (FAO, 1978) shows that over 25% of the total is used for non-human outlets, the most important of which is fish meal for animal feeding. Although a considerable amount of this 'industrial fish' is unsuitable or unacceptable for human consumption, it none the less contains protein equivalent in all respects to that in fish acceptable as food and, it can be argued, as potential raw material for protein products for food use.

The concept of isolating proteins from fish with acceptable properties as a complete product itself or as an ingredient for general use in foods has been an objective of scientists for many years but it has been plagued by the combined problems of extreme susceptibility of fish lipids to oxidation and of fish proteins to denaturation on drying (Finch, 1970). Dried protein preparations, of which Fish Protein Concentrate (FPC) is typical, usually lack the important functional properties of water-holding and gel-forming capacities and confer little other than improved nutritional quality when added to foods. Such products are

Authors' address: Torry Research Station, 135 Abbey Road, Aberdeen AB9 8DG, Scotland.
* To whom correspondence should be addressed.

0022–1163/82/0800–0483 $02.00

usually dried by the simultaneous removal of water and lipids by organic solvents such as isopropanol and, although tasteless and odourless, they have had no success commercially (Pariser *et al.*, 1978). It is now clear that FPCs have limited application (Pariser *et al.*, 1978) and that proteins from fish must retain gel-forming, emulsifying and other functional properties if they are to have wide acceptance for food use.

Alternative approaches to the problem of loss of functional properties are to isolate proteins by conventional extraction and precipitation techniques and either to modify them by chemical treatments acceptable for foods (Spinelli *et al.*, 1975) or to retain them in the wet state throughout the process and subsequent storage, prior to consumption.

As the feel of the food as it is eaten is known to be an important organoleptic requirement for fish, we have investigated the spinning process as a means of forming textures from alkaline protein extracts of fish flesh (Mackie, 1977). Spinning has the added attraction over alternative extrusion systems in having various preliminary washing and extraction procedures which remove some of the undesirable flavours and blood pigments associated with minced flesh from fish waste materials.

A considerable amount of information is available on procedures for spinning vegetable proteins, such as those of soya for the manufacture of simulated meats (Burke, 1971), but there is little on spinning proteins from non-vegetable sources. Proteins from meat industry waste, lung and stomach (Young & Lawrie, 1975) and from blood plasma (Swingler & Lawrie, 1977) have been shown to form acceptable fibres as has casein from milk (Schmandke *et al.*, 1976).

To some extent the non-fish proteins lose their functional properties on drying and, because of this, the mild process of wet spinning (Boyer, 1954) has been the basis of the processes which have since been developed (Gutcho, 1973) for preparing protein fibres for food use. In the wet-spinning process, proteins in either low or high pH extracts are extruded through narrow orifices (spinnerettes) into a buffered solution with a pH near the isoelectric point of the proteins. The system offers the possibility of altering texture of the fibres by a number of variables such as concentration of the protein in the alkaline extract, the dwell time in the precipitation bath, the composition of the salts in the bath itself, or the degree of stretch introduced into the fibres. These and other factors have been evaluated during the development of the process for fish muscle proteins.

Materials and methods

Raw materials

Three sources of fish protein were used. The first was blue whiting (*Micromesistius poutassou*) an under-utilized species which abounds off the north-western coast of the U.K. These fish were caught by the Station's research vessel

the *G. A. Reay* during 1979–80, frozen on board in blocks and stored at −30°C until required. The second source of protein was the skeletal waste or residue remaining after filleting operations on cod (*Gadus morhua*). In this instance the fish were kept in ice for periods up to 12 days prior to filleting. Finally, minced fillets of cod were used for protein extractability studies.

Preparation of fish minces

(1) *Blue whiting mince.* The frozen blocks were allowed to thaw at room temperature for a sufficient length of time to allow the fish to be separated from the frozen mass. Thawing was then allowed to continue overnight in a refrigerated room at 2.2°C.

The thawed blue whiting were headed and gutted before being passed through a Baader 694 deboning machine fitted with a drum with 5.0 mm holes. The separated mince was stored near 0°C prior to the protein extraction.

(2) *Deboned skeletal mince.* Cod skeletons (headless) were passed through the Baader 694 deboning machine and the separated mince stored as above prior to the protein extraction.

Pre-treatment of minces

The blue whiting mince was used as obtained from the deboning machine as it was found that the dark pigments could not be removed by washing with dilute salt solution. The pigments in the mince from the cod skeletons, however, could be largely removed by washing twice with 0.05 M sodium chloride solution in the ratio 1:2, i.e. one part mince to two parts of the salt solution, to give an almost white product.

During these and subsequent operations the temperature was maintained at 5°C or less.

Protein extraction

Mince (100 g) was blended with 900 ml 0.04 M sodium hydroxide solution in an MSE 'Atomix' blender at half full speed for 30 sec. A Perspex baffle, which just covered the surface of the liquid, was used to avoid excessive frothing of the protein solution.

The homogenate was then stirred using an H-shaped stainless steel stirrer for 15 min prior to centrifuging at 3000 ***g*** for 15 min.

Effect of pH on the extractability of protein

The effect of pH on the extractability of muscle proteins was determined by carrying out the above procedure on cod muscle mince. Hydrochloric acid or sodium hydroxide were added as appropriate to obtain the desired pH, and water was added to make a final volume of 1000 ml.

Preparation of protein isolate

The pH of the supernatant solution obtained was adjusted to pH 5.0 by the addition of 1.0 M hydrochloric acid and the precipitated protein recovered by centrifugation at 3000 *g* for 15 min.

Preparation of the spinning dope

The protein isolate obtained as described above was used for preparing the spinning dope as follows. To c. 400 g protein isolate was added sufficient distilled water to give a final volume of 1.0 l. The protein suspension was then blended to a fine slurry and the pH adjusted to 11.5 by the addition of 1.0 M sodium hydroxide. Usually 40–45 ml was found to be sufficient and it was also found that a protein:alkali ratio of 20:1 gave a suitable spinning dope with a pH of 11.5. Deaeration of the dope was achieved by centrifuging at 3000 *g* for 15 min.

Measurement of the viscosity of the spinning dope

The rheological flow behaviour was determined using a Contraves Rheomat 15T–Fc Viscometer in conjunction with the MS A—E measuring systems. The viscosity was measured over a variety of shear rates at a temperature of 5°C.

Spinning apparatus

The spinning apparatus (Fig. 1) is shown in diagrammatic form in Fig. 2. It consists of a reservoir (A) which has an outer jacket through which coolant at 5°C can be passed. Linked to (A) is a geared metering pump (B) (Slack & Parr, Derby, England) which in turn is connected to the sandwich filter (C) containing two nylon meshes of mesh size 200 and 100 μm. On emerging from the filter the dope passes into tube (D) and then into the spinnerette (E) (Courtaulds Engineering Ltd, Coventry, England, type SD/127) which has either 200 or 100 holes each of 100 μm diam. The bath (F) contains the coagulant solution of 1.0 M sodium acetate, 0.5 M acetic acid, pH 5.0. The spun fibre passes under the glass guide (G) and is taken up on reel (H).

Operation

After transferring the dope to the reservoir and screwing the lid firmly into place, a pressure of 18.18 kg/m^2 was applied and the speed of the pump adjusted to 70 rev/min. When dope was seen emerging from tube (E), the spinnerette holder assembly was screwed into place and when it emerged from the spinnerette, the bath (F) containing the coagulant was raised into place. The fibre 'tow' was then pulled away from the spinnerette face and under the glass guide (G) onto the take-up reel (H) which was set at a speed of 60 rev/min. The fibres

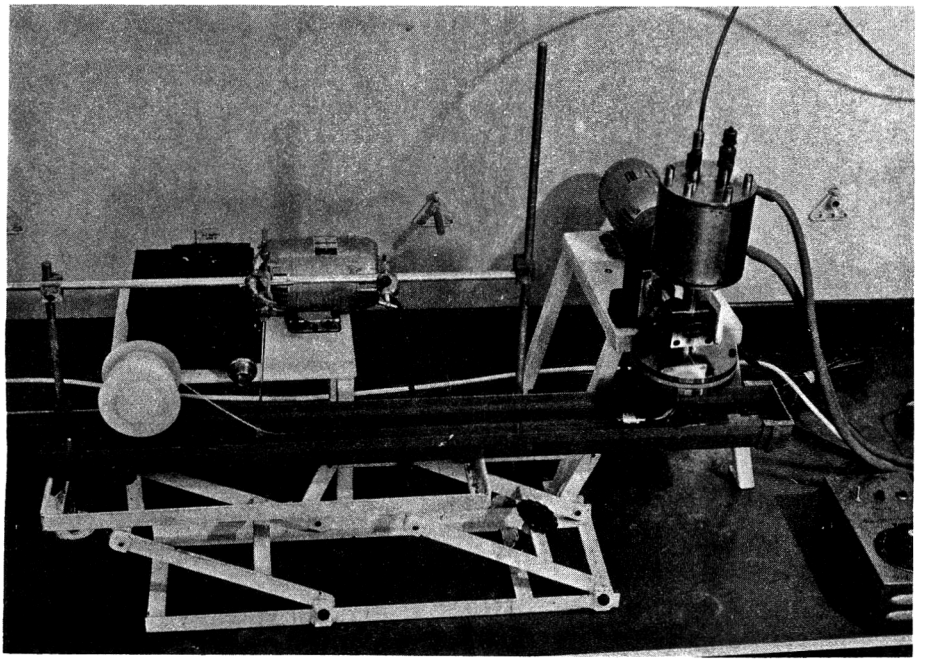


Figure 1. The spinning apparatus.

were collected as bundles by slipping the 'hank' off the roller. The fibre hank was immersed in distilled water for 24 hr in order to remove excess coagulant prior to carrying out proximate analyses.

Proximate analyses

Water and ash contents were carried out using standard methods. Nitrogen determinations were carried out by the micro-Kjeldahl procedure.

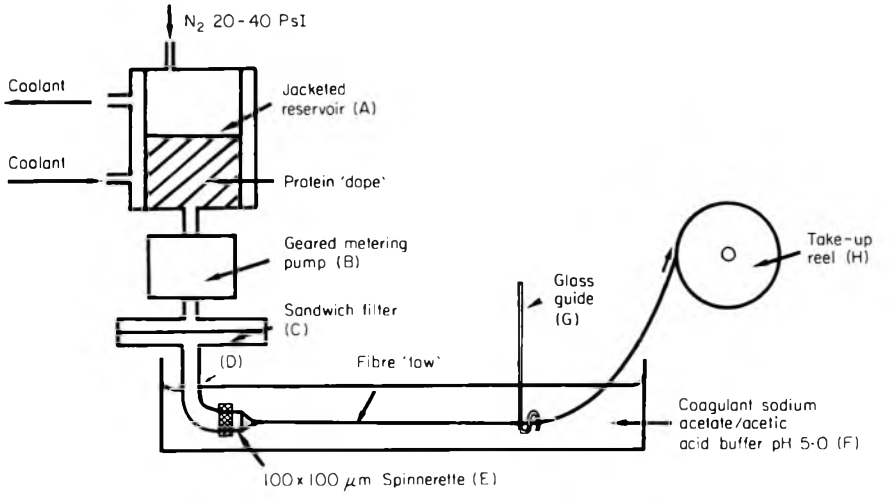


Figure 2. Flow diagram of spinning apparatus.

Amino acid analyses

Approximately 1.0 g of mince or fibres was hydrolysed in 250 ml 6.0 N HCl for 24 hr. The hydrolysate was made to 1.0 l, filtered and 10 ml evaporated to dryness in a rotary evaporator. This residue was taken up in 1.0 ml of lithium citrate buffer (0.3 M, pH 2.2). An aliquot (0.4 ml) was used for amino acid analyses on a Locarte amino acid analyser (Mackie & Ritchie, 1974).

Preparation of fibres for scanning electron microscope

Fish protein fibres were fixed overnight at 4°C in 2% glutaraldehyde in veronal-acetate buffer, dehydrated through a graded series of ethanol/water mixtures of absolute alcohol (approximately 20 min in each mixture), mounted on aluminium stub and coated with gold palladium in a sputter coater. The coated fibres were examined in a Cambridge Stereoscan 5600 at 15 kV accelerating voltage and magnification of 200 ×.

Results and discussion

Extraction of proteins from fish flesh

Provided that the ratio of extractant to flesh is at least 10:1, as much as 85% of the proteins of fish flesh are extractable in dilute solutions of acid (pH 2.0) or alkali (pH 11.5) and as the pH is altered towards the isolectric point of pH 5.0 the solubility decreases to reach a minimal value of 10–15% (Fig. 3). Similar findings have been obtained by Dyer, French & Snow (1950). It has thus been possible to obtain most of the skeletal muscle proteins in solution under mild conditions of low ionic strength and at temperatures near 0°C, an important consideration when there is the possibility of lysinoalanine formation under harsher conditions in alkaline solutions (Annan & Manson, 1981). The proteins remaining insoluble are believed to be largely connective tissue proteins and, as these are of lower nutritional value, their removal in this initial extraction step can be seen as being beneficial. This applies particularly to the extraction of proteins from the flesh obtained from skeletal processing waste, as it is known that it contains relatively large amounts of connective tissue protein (Thomson & Mackie, 1982).

While a high yield of non-connective tissue proteins is obtained on extracting with weak alkali under the conditions specified above, the solutions are not concentrated enough for spinning, as any fibres which form are soft and generally too weak to handle. It was, therefore, necessary to introduce a concentration step prior to the spinning stage to give fibres of adequate strength and which were at the same time acceptable to taste panels. The simplest and most effective procedure was to precipitate the proteins by adjusting the pH to the isoelectric point, pH 5.0, and to blend the isolate thus obtained in dilute alkaline solution at pH 11.5. De-gassing of the extracts was necessary and was readily carried out by centrifugation at 3000 ***g*** for 15 min. The alternative

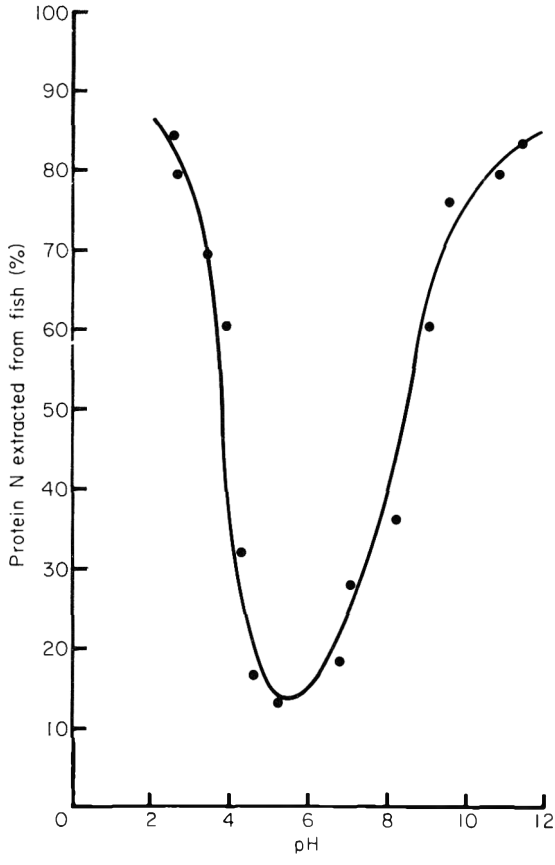


Figure 3. Variation of solubility of proteins of cod flesh with pH.

procedure of applying reduced pressure was not successful because of froth formation. Using this procedure viscous gels suitable for spinning were obtained in yields of *c.* 70% based on the recovery of nitrogen. The reduction in yield from the initial extraction can be attributed to partial solubility in the salt solution produced on neutralizing the initial alkaline extract.

Rheological properties of the protein extracts or 'dopes'

The alkaline extracts of fish proteins had to be sufficiently concentrated for spinning and at the same time fluid enough to be pumped through the filters and spinnerette. It was found in practice that these requirements were met when the concentration of the protein in the extract lay between 3 and 4%.

As expected for solutions of predominantly high molecular weight linear proteins, the alkaline spinning solutions show non-Newtonian behaviour, that is a non-proportional relationship between shear rate (D) and shear stress (τ) (Fig. 4). This flow behaviour was found to fit Casson's equation (Charm, 1963) which relates shear stress and shear rate as follows:

$$\sqrt{\tau} = K\sqrt{D} + \sqrt{C} \qquad (1)$$

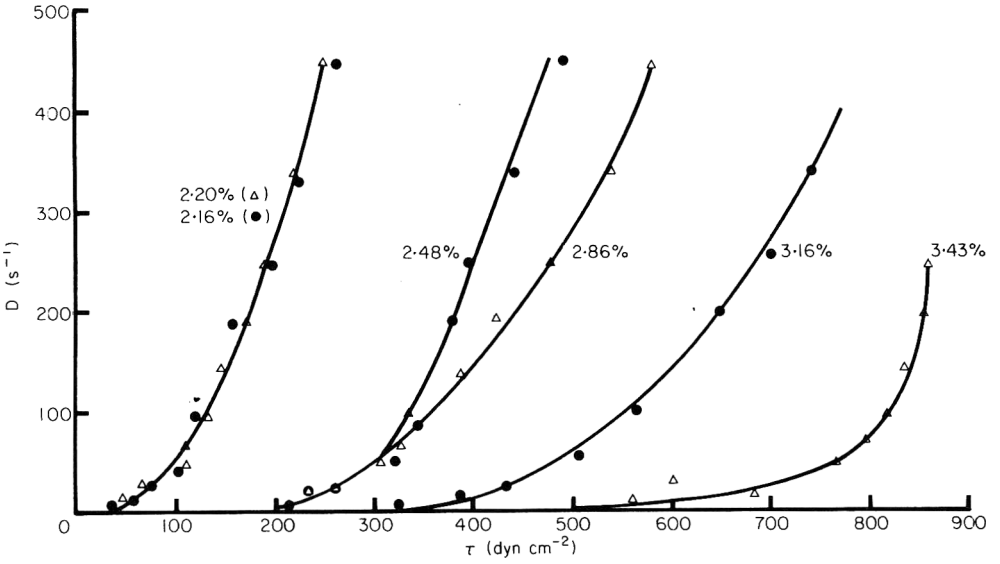


Figure 4. Rheological flow curves for fish protein isolates from blue whiting and cod skeletal mince respectively. Relationship between shear rate and shear stress at different concentrations of protein. △, Blue whiting; ●, cod skeletal mince.

where K and C are constants. C is termed the yield stress and can be determined from a plot of $\sqrt{D}$ *versus* $\sqrt{\tau}$, the intercept of which is $\sqrt{C}$ (Fig. 5). This value of C may then be used to derive the power law constants from eqn (2)

$$\tau - C = bD^s \tag{2}$$

by plotting log $\tau - C$ against log D (Charm, 1963). A straight line of slope s and intercept b was obtained (Fig. 6). It was found that the exponent s varied between 0.5 and 0.7 while b was dependent upon concentration (Table 1).

It is evident that the non-Newtonian flow properties of such extracts, in particular the marked concentration dependence of the yield stress, severely limits the range of concentration over which the requirements for both spinning and pumping can be met. In addition, the flow properties are affected by the length of time the fish has been stored in ice prior to the extraction of the proteins (Fig. 7). Fish iced for 1 day, for example, can give very viscous gels which can be difficult to pump at a protein concentration of 3.0% but fish stored for 9 or 12 days in ice can give an extract of lower viscosity and better quality of fibres are formed on spinning. It was found in practice, however, that viscosities of the protein extract varied considerably even when fish of the same nominal age in ice were used. The reasons for this variation in viscosity have not been established, but contributory factors are likely to be a variable degree of contamination with proteolytic enzymes of viscera, fluctuation in temperature during removal of flesh from bones and variation in the relative amount of myofibrillar proteins in the final extract. The myofibrillar proteins are also known to lose solubility in salt solutions during storage of fish in ice (Crupkin *et*

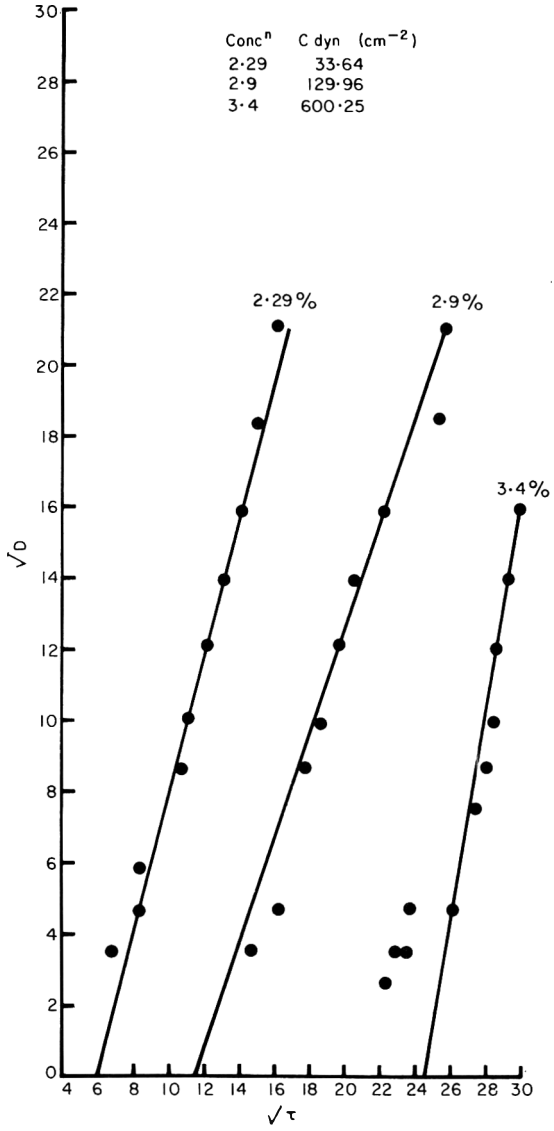


Figure 5. Plot of $\sqrt{D}$ *versus* $\sqrt{\tau}$ at different concentrations of protein (isolate from blue whiting mince).

al., 1979) and it is likely that the mechanisms responsible are also of importance in determining the viscosity of the alkaline extracts.

Stability of alkaline extracts

The viscosity of the alkaline extracts showed little change over several hours provided that the temperature was kept between 0 and 5°C. This was satisfactorily achieved during the spinning process by fitting a cooling jacket to the reservoir (Figs 1 and 2) and circulating water at 5°C through it. It was not,

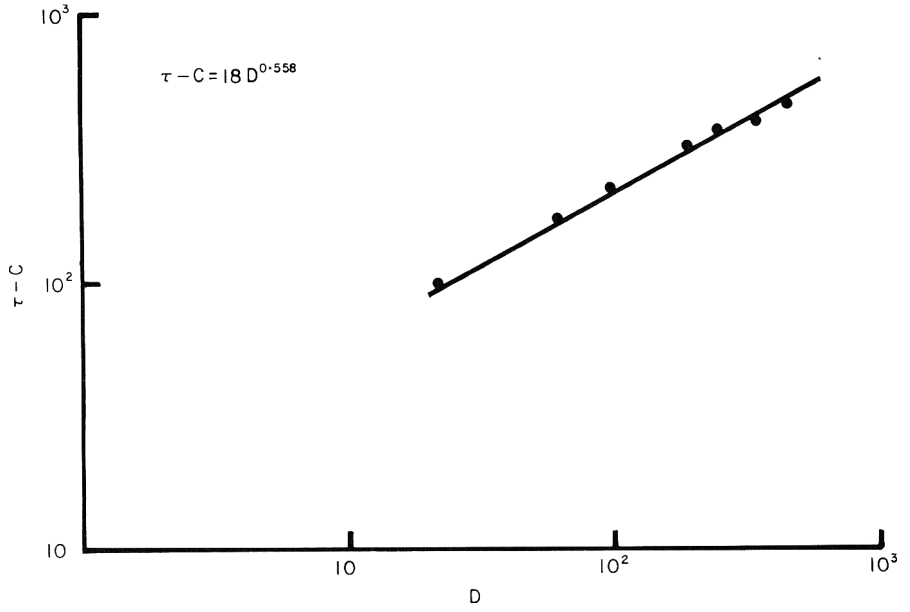


Figure 6. Plot of log $\tau - C$ *versus* log D at 3.1% protein (isolate from cod skeletal mince).

however, practicable to keep either the isolate or the alkaline spinning solution for more than 24 hr as viscosities often fell below the optimal range for spinning over this period (10–20 poise at 5°C and shear rate of 38 sec^{-1}). Throughput of the dope was of the order of 0.5 l in 20 min, and so for any particular batch the viscosity for all practical purposes was constant and did not affect the spinning process.

Optimal conditions for fibre formation

Although, in theory at least, it should be possible to form fibres from any protein precipitating from a viscous solution, the formation of fibre is favoured if one or all of the following criteria are met (Hartmann, 1978):

Table 1. Values for yield stress and corresponding power law constants at different concentrations of protein from cod skeletal minces

Protein in alkaline extract (%)	C yield stress (dyn cm^{-2})	Power law constants	
		b	s
2.10	104.4	3.9	0.633
2.48	268.96	7.0	0.524
3.16	342.25	18.0	0.558

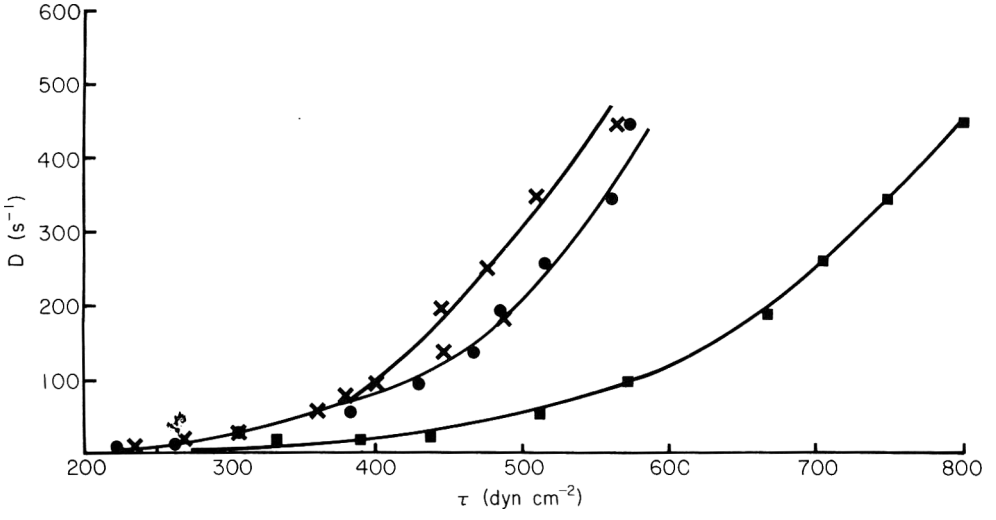


Figure 7. Effect of ageing on rheological properties of alkaline extracts of cod skeletal minces, washed with 0.5 M salt prior to extraction. ■, 1 day, $C = 3.0\%$; ●, 9 days, $C = 3.1\%$; ×, 12 days; $C = 3.1\%$.

(1) molecular weight $< 10\,000$;
(2) long linear chain;
(3) linear symmetry; and
(4) high degree of polarity.

The proteins from fish muscle readily meet the above requirements and, as they exist in their natural state in linear filaments, spinning can be regarded as a

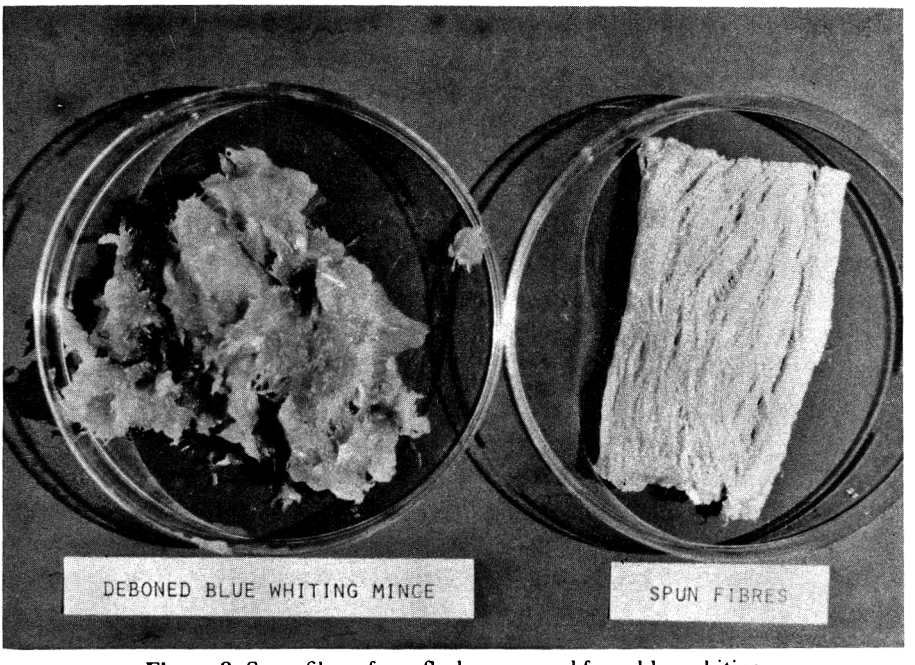


Figure 8. Spun fibres from flesh recovered from blue whiting.

Table 2. Proximate analyses of minces and fibres.

Material	Percentage		
	H_2O	Ash	N × 6.25
Cod skeletal waste			
Unwashed mince	82.8	1.34	15.86
Washed mince	85.70	1.03	13.27
Fibre	82.31	0.20	17.50
Blue whiting			
Mince	80.25	1.41	17.56
Fibre	79.52	0.15	20.21

favoured process. Compared with other solutions of proteins used for spinning (Young & Lawrie, 1975), the viscosities are high and maximal concentrations are limited to 4% because of the gel formation at higher concentrations. Concentrations of the order of 20% are required for plasma proteins (Swingler & Lawrie, 1977).

The most stable fibres were obtained using 1.0 M sodium acetate, 0.5 M acetic acid, pH 5.0 as the precipitant, using either 100 or 200 μm holes in the spinnerette. Under those conditions, fibres were readily produced on a continuous basis from minces from both deboned blue whiting and from cod skeletal waste. In both cases much of the pigment was removed in the extraction and spinning processes (Fig. 8).

The fibres thus prepared had protein contents between 17.5 and 20.0% (Table 2), similar to those for fish flesh, but the ash contents were markedly less.

The effect of reducing the pH in the precipitation bath was to decrease the protein content of the fibres. At pH 4.0, for example, protein contents between 6 and 8% were obtained. Similarly, as the concentration of sodium acetate was increased from 0.2 to 2.0 while the pH was held at 5.0, the protein content of the fibres decreased from 21 to 16% (Table 3).

These results contrast with those of blood plasma proteins (Swingler & Lawrie, 1977) which formed acceptable fibres at low pH. Swingler & Lawrie

Table 3. The effect of molarity of sodium acetate on the protein content of fibres

Molarity of sodium acetate	Protein in fibre (%)
0.2	21.3
0.5	20.1
1.0	19.0
2.0	16.0

Table 4. Effect of residence time in the precipitation bath on protein content of fibres. Gear pump output (23.0 g extract min^{-1} (29.3 m min^{-1}) of fibre from 100 μm hole)

Distance in bath before pick-up (cm)	*T* (sec)	Protein in fibre (%)
34	0.7	16.3
50.6	1	15.8
100	2	18.5

(1977) also investigated the composition of the coagulant bath and concluded that, provided the acetic acid concentration was high enough, plasma proteins coagulated adequately in solutions where the concentration of sodium chloride varied from 5.0 to 20% and the acetic acid concentration from 1.0 to 10%. The residence time in the precipitation bath also affected the protein content of the fibres (Table 4), the optimal distance being 100 cm at a pump output of 23.0 g extract min^{-1}.

It was found, in agreement with Swingler & Lawrie (1977), that the extrusion rate through the spinnerettes essentially determined the speed of the pick-up reel which was adjusted to maintain the fibres under tension as they were drawn from the bath. It was not possible, however, to stretch the fibres by increasing the speed of the pick-up reel. This observation is in agreement with those of Swingler & Lawrie (1977) and Balmaceda & Rha (1974).

Chemical and sensory evaluation of the fibres

The fibres obtained under optimal conditions were white, even when prepared from the skeletal waste of cod and they had the appearance and gel-like property associated with natural fibres of fish flesh (Fig. 8). Their appearance in the scanning electron microscope (Fig. 9) was that of a homogeneous dispersion of protein with a skin-like surface similar to that obtained for soya protein fibres (Aquilera, Kosikowski & Hood, 1975).

Amino acid analyses (Table 5) again confirmed that there was little, if any, change in the amino acid composition resulting from the extraction and spinning processes. The relatively lower value for proline for the fibre from the mince from cod skeletal waste again confirmed previous observations that connective tissue proteins are selectively removed during the extraction process. While no lysinoalanine could be detected in the fibres with the analytical system used, its formation cannot be excluded entirely, as it is known that it is formed in detectable concentrations in soya protein at temperatures above ambient (Annan & Manson, 1981). However, as it is present in many foods which have not been subjected to treatment with alkali but to heat only, its significance as a health hazard has yet to be established.

Figure 9. Scanning electron micrographs of fish protein fibres (200 ×).

Taste panel evaluation of the fibres was encouraging both with regard to appearance and texture. The fibres as prepared by precipitation at pH 5.0, followed by washing in water, tended to be too tough and stringy but, by allowing them to soak in 0.5 M sodium citrate/citric acid (pH 6.5) or in 3% sodium pyrophosphate solution (pH 6.5–7.0), they became softer and more acceptable. For sensory assessment the fibre 'tows' were chopped into suitable lengths and then cooked as for steamed fish or as battered products of the fish finger type. It was not necessary to use a binder as the fibres themselves had adequate adhesion.

Our main conclusions are that fibres with textures normally associated with fish can be produced by the spinning process and that they can be made into acceptable but not highly rated fish finger type of products. Their storage life at 0°C is no more than that of fish and, for the time being, at least, they cannot be stored successfully in the frozen state because of toughening and drying out. The reasons for the severe deterioration on frozen storage are not understood but it is likely that mechanisms similar to, but more rapid than those associated with frozen storage deterioration of fish flesh are operating.

The spun fibres can, however, be kept satisfactorily in acid preservative of the marinade type. In many ways they are highly suited for this type of product as the operation requires no more than a transfer from the precipitation bath to the marinading liquor. Very acceptable products were prepared and it would seem that they have potential value in semi-preserve products in general.

Table 5. Amino acid analyses of minces and fibres g amino acid/100 g protein

Amino acid	Skeletal-frame waste			Blue whiting		
	Unwashed mince	Washed mince	Fibres	Mince	Fibres	Cod mince
Aspartic acid	10.13	9.52	10.74	8.84	10.39	9.32
Threonine	5.05	4.20	3.97	4.12	4.70	4.46
Serine	5.16	4.19	4.94	3.68	4.20	4.38
Glutamic acid	16.05	16.15	18.01	13.16	15.36	13.55
Proline	4.28	3.18	2.25	2.16	3.02	4.14
Glycine	6.93	5.03	3.61	4.79	3.58	7.39
Alanine	6.78	5.74	5.84	5.35	5.45	6.08
Cystine/2	1.72	1.00	1.12	1.97	2.35	1.97
Valine	5.40	4.79	5.17	4.45	5.20	4.35
Methionine	3.44	3.13	3.45	2.66	3.42	2.58
Isoleucine	4.67	4.41	4.95	4.06	4.89	3.70
Leucine	8.13	7.71	8.69	7.34	8.57	6.98
Tyrosine	4.08	3.48	4.00	3.22	3.82	3.03
Phenylalanine	4.47	3.47	3.76	3.63	3.81	3.53
Histidine	1.57	1.16	2.00	1.29	3.76	1.65
Lysine	6.48	7.08	9.96	7.12	6.50	7.53
Arginine	3.72	3.43	6.48	4.8	5.87	5.77

Further development work is, however, necessary before any of the above products could be considered for commercial processes. Our aim has been to demonstrate the possibility of preparing fibres for food use from fish waste material.

Acknowledgments

Mr. A. Johnston advised on the design of the spinning apparatus and Mr. N. Houston examined the spun fibres in the scanning electron microscope. The authors are also indebted to Dr. G. C. East, Department of Textile Industries, University of Leeds, for helpful discussions and demonstrations.

References

Annan, W.D. & Manson, W. (1981) *Food Chem.* **6** (3), 255.
Aquilera, J.M., Kosikowski, F.V. & Hood, L.F. (1975) *J. Texture Studies*, **6**, 549.
Balmaceda, E. & Rha, C. (1974) *J. Fd Sci.* **39**, 226.
Boyer, R.A. (1954) U.S. Patent, 2682466.
Burke, C.S. (1971) *Textured Vegetable Proteins. II. Survey of the UK and US Patent Literature.* BFMIRA, Leatherhead, Surrey.
Charm, S.E. (1963) *J. Fd Sci.* **28**, 107.

Crupkin, M., Barassi, C.A., Martone, C. & Trucco, R.E. (1979) *J. Sci. Fd Agric.* **30,** 911.
Dyer, W.I., French, H.V. & Snow, J.M. (1950) *J. Fish. Res. Bd Can.* **7,** 585.
Finch, R. (1970) *C.R.C. Rev. Food Technol.* **1,** 519.
FAO (1978) *Yearbook of Fisheries Statistics,* **47,** 2.
Gutcho, M. (1973) In: *Textured Foods and Allied Products.* Noyes Data Corp., Park Ridge, New Jersey, U.S.A.
Hartmann, W.E. (1978) *J. Texture Studies,* **9,** 125.
Mackie, I.M. (1977) *Spectrum,* **152,** 9.
Mackie, I.M. & Ritchie, A.H. (1974) In: *Proc. IV Int. Congr. Fd Sci. and Technol., Madrid, Spain,* **1,** 29.
Pariser, E.R., Wallerstein, M.B., Corkery, C.J. & Brown, N.L. (1978) *Fish Protein Concentrate: Panacea for Protein Malnutrition?* MIT Press, Cambridge, Mass., U.S.A.
Schmandke, H., Paul, D., Friebe, R., Anger, H., Bartsch, D., Purz, H.J., Luther, H., Maune, R. & Webers, V. (1976) *Nahrung,* **20** (2), 195.
Spinelli, J., Groninger, H., Koury, B. & Miller, R. (1975) *Process Biochem.* **10** (1), 31.
Swingler, G.R. & Lawrie, R.A. (1977) *Meat Sci.* **1,** 161.
Thomson, B.W. & Mackie, I.M. (1982) *J. Fd Technol.* (in press).
Young, R.H. & Lawrie, R.A. (1975) *J Fd Technol.* **10,** 453.

(Received 22 September 1981)

J. Fd Technol. (1982) **17**, 499–506

Parboiling of paddy by simple soaking in hot water

K. R. UNNIKRISHNAN, C. S. VIRAKTAMATH, H. KRISHNAMURTHY AND K. R. BHATTACHARYA*

Summary

Parboiling of paddy by mere hot water soaking, without steaming, has the advantage of not requiring a boiler. Soaking at a temperature 10–15°C above the gelatinization temperature (i.e. generally at about 80–85°C) is necessary to achieve fair to moderate gelatinization (i.e. parboiling). However, soaking at such temperatures for a time sufficient for the water to penetrate and gelatinize the grain core invariably leads to over-imbibition of moisture, husk splitting and leaching; soaking for less time, on the other hand, leaves many grains with 'white belly'. The best method therefore is to soak the grain for such time as to imbibe about 30% moisture (wet basis) (i.e. for about 1½–2 hr), drain out the water, and temper the hot paddy for another 1–2 hr. This process yields optimally parboiled paddy without 'white belly' or grain splitting and with reasonably good milling quality and colour.

Introduction

Parboiling of paddy consists, in essence, of pre-cooking of rice within the husk (Bhattacharya, 1979). The usual method is to soak paddy in water until it is saturated (about 30% moisture, wet basis), steam it (after draining the water) to gelatinize the starch, and then dry it.

The possibility of parboiling paddy merely by soaking in hot water (without further steaming) has been considered. The main advantage is that it can be used in small-scale industry, where a boiler may not be available. In fact, a process along this line has been proposed from the Rice Process Engineering Centre, Indian Institute of Technology, Kharagpur (Ali, 1974; Ali & Ojha, 1976). The process consists in soaking at, or slightly above the gelatinization temperature

Authors' address: Discipline of Rice and Pulse Technology, Central Food Technological Research Institute, Mysore 570013, India.

* To whom correspondence should be addressed.

0022–1163/82/0800–0499 $02.00

(GT) until the grain (after drying and milling) becomes fully translucent up to the centre; no other test of parboiling, if any, was carried out.

However, these proposals need further consideration. A very comprehensive and systematic laboratory study of the various processing factors during parboiling was carried out in this laboratory in the early sixties (Bhattacharya & Subba Rao, 1966). It was clearly demonstrated in this study that optimal parboiling by hot water soaking alone was not possible by continuous soaking at any given temperature. The reason was related to three basic phenomena:

(1) the soaking process of paddy is always accompanied by development of a moisture gradient from the outer to the inner layers, the gradient being steeper as the temperature of soaking increases;
(2) as the moisture content of paddy exceeds a value of a little over 30% (wet basis, w.b.), the husk begins to split, leading to leaching of grain contents and finally to more or less deformity of the kernel;† and
(3) when soaking near or below the GT of the variety, the moisture content finally reaches more or less an equilibrium value of about 30%. However, if soaked above the GT, the moisture goes on increasing with time even far beyond 30%.

The combined effect of these three phenomena are as follows:

(a) when soaked below or just about the GT, the grain can be fully hydrated without splitting, but no significant gelatinization (i.e. parboiling) occurs;
(b) when soaked fairly above the GT, on the other hand, if the soaking is limited to about 30% moisture to avoid splitting, a distinct ungelatinized opaque core ('white belly') is left due to the high moisture gradient mentioned above; while
(c) if the soaking is prolonged sufficiently to gelatinize the core, there is invariably overimbibition and splitting, leading to leaching and possible grain deformity.

In other words, continuous soaking at such a temperature as would cause gelatinization (i.e. parboiling) must yield either 'white belly' or leaching and possible grain deformity and never an optimally parboiled product. The hydration studies of Bandyopadhyay & Roy (1977) are in general conformity with these conclusions.

The same logic, however, also provides an answer to the problem. If the paddy is soaked (above the GT) for such time as to imbibe about 30% moisture and no more, but then drained and tempered at the same temperature for an optimal time, then we should have an optimally parboiled rice without either

† Later studies in this laboratory showed that the excessive grain deformity following husk splitting observed in the above-mentioned work was due to the use of a stirrer with rather sharp blades during the soaking step. With a blunt stirrer (or none), deformity is either negligible or occurs only upon wide splitting of husk (after prolonged or very high temperature soaking). However, any husk splitting, with or without grain deformity, leads to considerable leaching which is itself most undesirable. In the present studies, perceptible kernel deformity after drying and milling was observed only in a few cases of extreme treatment; hence this aspect is not reported upon further. However, husk splitting is duly reported.

'white belly' or leaching. Testing this hypothesis was the object of the present studies.

Materials and methods

Laboratory studies

For the laboratory studies, two varieties of paddy, GEB24 and Taichung Native 1 (TN1), from laboratory stock, about 6 months after harvest, were used. As measured by the alkali test with 1.4% KOH (Bhattacharya & Sowbhagya, 1972), the former had a GT of about 70°C and the latter about 60°C. Soaking was carried out in test tubes immersed in a water bath (maintained at 0.5°C above the desired temperature). Five grams of paddy each was put into three pairs of test tubes containing 10 ml distilled water each (which were pre-heated in the water bath) and stirred well with a fine glass rod to dislodge air bubbles. The three pairs were soaked for three different periods – representing under, optimal (to give about 30% moisture) and over soaking for the respective temperature, as determined from preliminary experiments. The under and over soaked samples were dried in shade without any further treatment. For the optimally soaked sample, the water was drained, the paddy of the two tubes were combined into one tube so as to be full, this tube was corked and then put back into the water bath for tempering for the desired period. Another portion of the same optimally soaked paddy, without tempering, was steamed at atmospheric pressure for 10 min to represent fully parboiled rice control. Finally, all paddy samples were dried in shade, shelled in a McGill sheller and milled with a hand polisher to roughly 3–4% degree of milling (by weight).

Any husk splitting during soaking, and 'white belly' (and deformity) in the milled grain, were noted visually. The following tests (Bhattacharya, 1979) were carried out to determine the degree of parboiling, if any, of the milled rice:

(1) equilibrium moisture content attained by the rice when soaked in water at room temperature (EMC-S) was determined as described (Indudhara Swamy, Ali & Bhattacharya, 1971);
(2) the apparent water uptakes of the rices were determined using 1.0 g rice for each test as described earlier (Ali & Bhattacharya, 1972a); 60 and 96°C were employed for GEB24 and 55 and 96°C for TN1 (in view of its lower GT), and the respective ratios were calculated; and
(3) alkali test was done as per Ali & Bhattacharya (1972b), using 1.0% KOH for GEB24 and 0.6% KOH for TN1. A score card was prepared attributing a score of 1 to raw rice (undegraded) and a score of 10 to TN1 highest parboiled rice (soaked at 80°C for 2 hr and steamed at atmospheric pressure for 10 min) which showed maximum degradation.

Pilot-scale studies

Pilot-scale studies were carried out in the laboratory using an overhead tank essentially of the same design and following the same procedure as in the

large-scale trials mentioned below. The capacity was about 20 kg paddy per batch. Several varieties of paddy procured from the market were used for these experiments. The paddy was soaked at the desired temperature for the desired period, the water was then drained, and the paddy was left in the tank, covered all around with jute bags, for tempering as desired. Grain splitting was noted. It was then dried in shade and a representative sample was milled using a McGill sheller and a McGill miller No. 1 to about 6–8% degree of milling. The grain breakage (g broken rice/100 g total milled rice) and 'white belly' were noted.

Large-scale studies

These were conducted in the rice mills of M/s. K.N. Oil Industries, Mahasamund, Madhya Pradesh. A special mild-steel overhead tank was designed and installed as shown in Fig. 1. It was lagged all around by tying jute bags. One variety, Gurmatia, routinely parboiled in the mill, was used. Sufficient water was run into the tank (as determined from preliminary trials to finally remain at least 15 cm above the paddy level) and it was heated by passing steam to a temperature of about 10°C above the desired soaking temperature. Paddy (about 200 kg) was then charged in and the circulating pump was started.

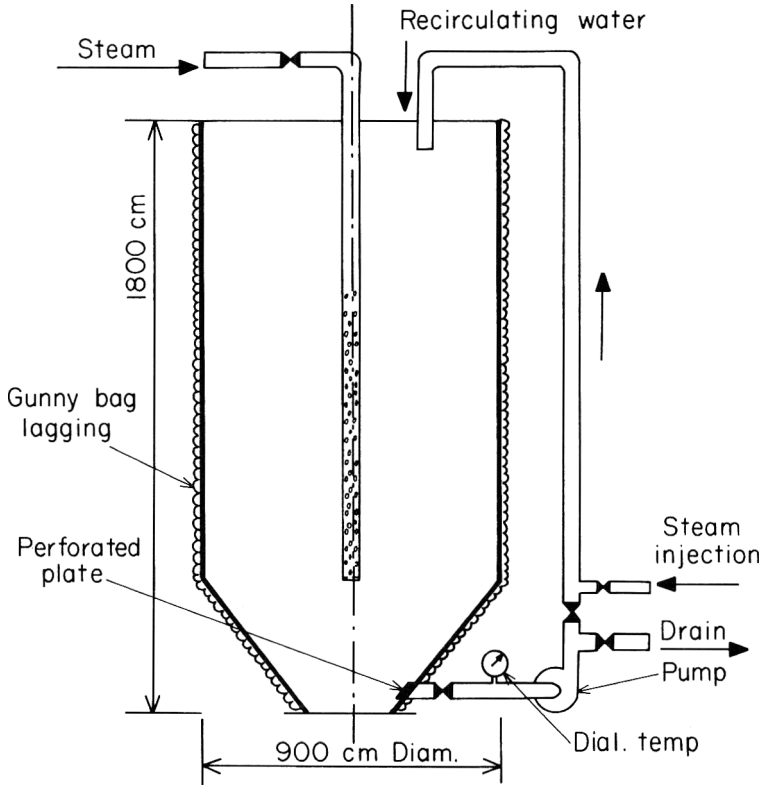


Figure 1. Schematic diagram of parboiling tank.

The appropriate soaking temperature was maintained by intermittent injection of steam, as needed, in the water circulating line. After the desired period of soaking, the water was drained out and the paddy was tempered in the same tank covered with jute bags on top. It was then discharged. Representative samples were collected during discharging, dried in shade and portions were milled in a McGill miller No. 1 (The mill authorities were sceptical of the process and would not agree to dry and mill the product as such. They put the treated paddy back in their cement soaking tanks for their normal cold-soaking–parboiling process. Hence, the above procedure of drying and milling only a representative sample.) The breakage, etc., were noted.

Results and discussions

Laboratory-scale studies

The various results are shown in Table 1. The results of EMC-S, ratio of apparent water uptake at 55 (TN1) or 60°C (GEB24) to that at 96°C (W'_{55}/W'_{96} or W'_{60}/W'_{96}), and alkali score, all bring out the extent of parboiling, if any. The raw rice control is shown in top under each variety, while the optimally soaked and steamed (i.e. properly parboiled) control is shown at the end of each temperature of soaking.

It can be seen from the results that soaking at up to 75°C in the case of GEB24 had very little parboiling effect (results at 70°C were still lower, hence not shown). Continuous over-soaking at this temperature no doubt gave a fully translucent, and hence apparently parboiled product, with no 'white belly' and little husk splitting; but the degree of parboiling, as judged by the three indices, was only minimal or negligible. This shows the hazard of facile conclusion about parboiling merely from grain translucence (Ali, 1974; Ali & Ojha, 1976; Bandyopadhyay & Roy, 1977). The situation was somewhat better at 80°C, but there was distinctly moderate parboiling effect at 85°C. However, it is interesting to note that best results in terms of absence of 'white belly' and grain splitting were obtained by optimal soaking followed by draining and tempering for 2 hr. Less or no tempering yielded 'white belly', while more prolonged soaking resulted in husk splitting.

Results with TN1 were more or less similar except that identical results were obtained at about 5°C lower soaking temperatures.

On this basis, it can be said that soaking paddy at a temperature of 10–15°C above its GT for an optimal period (i.e. to give a grain moisture of about 30%) followed by draining and tempering for 1–2 hr would yield a good, moderately parboiled rice.

Pilot-scale trials

Pilot-scale trials (20 kg paddy in each batch) were carried out on the above basis with several varieties. The approximate GT of the samples were deter-

Table 1. Properties of variously hot water soaked rice

Variety	Soaking: Temp. (°C)	Soaking: Time (hr)	Tempering time (hr)	Steamed	Husk splitting*	'White belly'†	EMC-S (%, w.b)	Water uptake ratio‡ (%)	Alkali score
GEB24									
Raw rice control							27.1	9.3	1
	75	2.0	0	No	−	R	28.8	—	2
		3.0	0	No	−	R	29.7	10.6	2
			1	No	−	++	30.9	12.7	3
			2	No	±	+	30.9	13.3	3
		4.0	0	No	±	−	30.6	—	3
		3.0	0	Yes	±	−	38.5	25.2	8
	80	1.0	0	No	−	R	28.4	—	2
		2.0	0	No	−	++	—	—	—
			1	No	±	+	32.9	16.2	3
			2	No	+	±	33.7	18.8	3
		3.0	0	No	±	±	32.8	—	3
		2.0	0	Yes	+	−	39.6	34.8	9
	85	0.75	0	No	−	++	28.4	—	2
		1.5	0	No	−	++	—	—	—
			1	No	±	±	35.4	20.1	4
			2	No	+	−	38.2	24.2	4
		2.25	0	No	++	−	35.2	—	3
		1.5	0	Yes	+	−	40.1	34.8	9
Taichung Native 1									
Raw rice control							29.8	11.9	1
	70	2.5	0	No	−	R	30.6	—	2
		3.75	0	No	−	—	32.1	12.9	3
			1	No	−	+	33.4	13.6	3
			2	No	−	±	34.0	14.6	3
		5.0	0	No	−	±	33.3	—	3
		3.75	0	Yes	±	−	43.5	29.0	8
	75	2.0	0	No	−	R	32.9	—	2
		3.0	0	No	−	++	34.0	14.3	3
			1	No	±	++	37.7	15.8	4
			2	No	±	+	38.2	17.0	4
		4.0	0	No	±	+	37.8	—	4
		3.0	0	Yes	+	−	46.8	31.6	9
	80	1.0	0	No	−	++	31.6	—	2
		2.0	0	No	−	++	—	—	—
			1	No	±	+	40.8	21.6	5
			2	No	+	−	43.2	24.8	6
		3.0	0	No	+	−	42.0	—	5
		2.0	0	Yes	+	−	46.4	34.6	10

* −, Not split; ±, slightly split; +, fair. There is always some splitting of husk upon steaming, as also often after tempering seen here. This is not a drawback. But splitting *during soaking* leads to leaching and possible deformity.

† R, somewhat like raw rice, 'white belly' indeterminate; −, no white belly; ±, trace; +, fair; ++, high.

‡ Represents $W'_{60}/W_{\%}$ in the case of GEB24 and $W'_{55}/W_{\%}$ in the case of TN1.

Table 2. Pilot-scale trials of parboiling by soaking

Variety	Approximate GT (°C)	Soaking		Tempering time* (hr)	'White belly'†	Milling breakage (%)
		Temp. (°C)	Time (hr)			
Halubbulu	68	80	2	2	±	0
				3	−	0
Madhu	75	80	2	2	++	5
		85	2	2	±	2
SR26B	70	80	2	2	±	7
				3	−	7
Gourisanna	70	80	2	2	+	10
		85	2	2	±	5
				3	−	2

*The small batch size, which caused quick loss of heat, often necessitated longer than usual tempering time.

†Key as in Table 1.

mined by the alkali test in 1.4% KOH (Bhattacharya & Sowbhagya, 1972) and the soaking temperature was adjusted accordingly.

The results are shown in Table 2. It is again evident that soaking at a temperature of 10–15°C above GT for an optimal period, followed by draining and tempering for about 2 hr yielded reasonably good parboiled rice without 'white belly' or husk splitting and having reasonably good milling quality.

Large-scale trials

These trials were conducted with only one variety, Gurmatia. The results are shown in Table 3, which generally confirmed the earlier results. The colour of the rice was good.

Table 3. Large-scale trials of parboiling by soaking

Soaking		Tempering time (hr)	Husk splitting*	'White belly'*	Milling breakage (%)
Temp. (°C)	Time (hr)				
80	2	2	±	+	8
85	1.5	2	±	+	13
	2	2	+	±	8
Raw rice control					50
Conventional parboiled rice of the mill					6

*Key as in Table 1.

Conclusions

It can therefore be concluded that soaking paddy at a temperature of 10–15°C above the gelatinization point for 1½–2 hr followed by draining out and hot tempering for another 1–2 hr can give reasonably good parboiled rice with acceptable degree of parboiling, milling breakage and colour. Continuous soaking at the same temperature for a longer period to avoid 'white belly' would lead to leaching and possible grain deformity; there would also be over-imbibition of moisture, so that the time and cost of drying would be higher. If, however, the temperature is lowered to avoid these drawbacks, the degree of parboiling achieved would be negligible and insufficient.

Acknowledgments

We are grateful to Mr M. K. Bhashyam and Mr C. V. Raghavan for help in the large-scale trials. We are very thankful to Mr Y. M. Indudhara Swamy and Dr H. S. R. Desikachar for creating interest in the subject. Our thanks are due to M/s. K.N. Oil Industries, Mahasamund, Madhya Pradesh, for providing all facilities for the large-scale trials.

References

Ali, N. (1974) Studies on some engineering aspects of parboiling of paddy. PhD. Thesis, Indian Inst. Technol., Kharagpur, India.

Ali, S.Z. & Bhattacharya, K.R. (1972a) *Lebensm.-Wiss. u. Technol.* **5,** 207.

Ali, S.Z. & Bhattacharya, K.R. (1972b) *Lebensm.-Wiss. u. Technol.* **5,** 216.

Ali, N. & Ojha, T.P. (1975) *J. Agric. Eng. Res.* (in press).

Ali, N. & Ojha, T.P. (1976) In: *Rice: Postharvest Technology* (Eds E. V. Araullo, D. B. de Padua and M. Graham) p. 163. Int. Devel. Res. Centre, Ottawa, Canada.

Bandyopadhyay, S. & Roy, N.C. (1977) *J. Fd Sci. Technol.* **14,** 95.

Bhattacharya, K. R. (1979) In: *Chemical Aspects of Rice Grain Quality* p. 363. Int. Rice Res. Institute, Los Baños, Laguna, Philippines.

Bhattacharya, K.R. & Sowbhagya, C.M. (1972) *J. Fd Technol.* **7,** 323.

Bhattacharya, K.R. & Subba Rao, P.V. (1966) *J. Agric. Fd Chem.* **14,** 473.

Indudhara Swamy, Y.M., Ali, S.Z. & Bhattacharya, K.R. (1971) *J. Fd Sci. Technol.* **8,** 20.

(Received 15 July 1981)

J. Fd Technol. (1982) **17**, 507–511

Technical note: Cooking losses in rice—a preliminary study of the effect of grain breakage

P. A. CLARKE

Introduction

In rural rice processing, the use of traditional and other outdated technology for processing and milling of paddy can result in a very high proportion of broken rice which frequently is not separated. Studies of post-harvest processing of rice have tended to concentrate on milling out-turn and even when reporting head yield (Head rice = grains which are considered to be whole after milling) the impression conveyed is one of depression of quality.

In the more affluent markets of the world, milled rice often contains 5% or perhaps 10% of large brokens (grain pieces with half to three-quarters of the length of whole, milled grains – FAO, 1972). In poorer markets, attempts are rarely made to size-grade, and consequently rice enters the market chain with a high proportion of broken grain, much of which may be small brokens or chips. From personal observation, the proportion of brokens may be up to 50%. When such a mixture of sizes is cooked it is highly probable that the cooking time chosen will closely approximate to the time necessary to ensure soft, whole grains and therefore the broken grain could be over-cooked, with possible dispersion into the cooking water. The effect of such high concentrations of broken grain on the loss of gross food value in cooked rice, as presented for consumption at table, was investigated initially as loss of solids (Dendy, 1978).

Batcher, Staley & Deary (1963) surveyed domestic cooking procedures and reported that rice may be boiled up to eight times its weight of water. Swaminathan (1942) and Chattopadhyay (1977) report that the cooking water (called 'conjee' in India) may be discarded or used for non-food uses, alternatively it may be consumed as a gruel by the poor or fed to animals.

The literature revealed numerous papers on the vitamin losses incurred by washing and cooking rice, and several papers reporting solids loss on cooking as a parameter for quality assessment. However, only one paper (Cheigh *et al.*, 1978) reported solids losses on combined washing and cooking, but not in excess water and not the effect of broken grain. In the present study, new data are

Author's address: Tropical Products Institute, Culham, Abingdon, Oxon OX14 3DA.

0022–1163/82/0800–0507 $02.00

presented which show that the proportion of broken grain has a major influence on overall cooking loss and the consequent reduction in food availability may influence policy makers in the decision whether or not to implement schemes to upgrade the quality of milled rice.

Materials and methods

Grain fractions

Two types of milled rice were obtained from a local commercial source: a long grain raw rice and a medium grain parboiled. To help ensure purity of grain, the rice was thickness-graded through slot-perforated sheet and length-graded through an indented cylinder. Throughout, only unblemished whole grain was selected for use. Broken grain was produced by passing whole grain through a hammer-mill from which the screen was removed. From the product, woven wire screens and indented cylinders were used to select two fractions of broken rice: large brokens approximating to half whole grain weight and small brokens approximating to one-tenth whole grain weight. Examples of average weights are shown in Table 1. After grading, the fractions were sealed in polythene bags for several days to allow moisture to equilibrate and were then tested for moisture by the hot-air oven method of the AACC (1969).

Cooking procedure

Initially, it was planned to measure the cooking losses directly by analysing the drained liquor. However, in practice, particularly when using higher contents of brokens, it proved very difficult to ensure replication of results owing to inconsistent drainage. A method was therefore devised to measure losses indirectly, by difference.

Preliminary tests showed that to ensure (1) a manageable level of frothing during cooking, and (2) a level of fluidity, after cooking, necessary for drainage, the ratio of water to rice should be approximately 10:1 by weight. To guarantee that drainage of suspended material was complete, and to give the degree of cooked grain separation demanded by most consumers of long-grain rice, it was

Table 1. 1000 piece weight of milled raw rice and brokens fractions

Pieces	1000 piece wt (g, dry wt basis)
Whole	13.8
Large broken	6.6
Small broken	1.2

decided to rinse the draining grain with boiling water. The grain was then placed on a tared Petri dish and oven dried to constant weight for analysis.

Prior to each series of tests, the optimum cooking time for whole grain was determined by organoleptic assessment.

Subsequent work has shown that when cooking rice in an excess of water (i.e. ratios ranging from 1:6 to 1:10) the gross losses experienced are broadly similar.

Results and discussion

After preliminary trials, the loss of solids proved reasonably easy to determine and, as may be expected, increased with proportion of brokens. However, the baseline for loss varied inversely with age of milled rice in agreement with the solubility studies described by Barber (1972). Table 2 shows the results from three experiments on one sample of raw milled rice at approximately 3 month intervals. The proportionate loss calculated from the results in Table 2 for large and small brokens was 22 and 47% respectively.

In certain parts of the world, notably the Indian subcontinent and West Africa, a large proportion of rice is consumed in the parboiled form. To investigate the effect of parboiling, a similar sequence of tests was performed on parboiled rice and the results are shown in Table 3.

Raghavendra Rao & Juliano (1970) reported that parboiled rice is less dispersible than raw and in the present test the parboiled whole grain lost less than the raw; however, the parboiled broken grain losses exceeded those of raw brokens. The average proportionate loss calculated for large and small brokens of parboiled rice was 32 and 72% respectively. The loss from small brokens is

Table 2. Solids loss on cooking of raw rice with varying content of broken grain

Grain composition (% by weight)	Losses (% by weight)					
	Experiment 1*		Experiment 2*		Experiment 3*	
	Total solids loss	Broken grain loss†	Total solids loss	Broken grain loss†	Total solids loss	Broken grain loss†
100 whole	13.0	—	12.7	—	11.6	—
90 whole, 10 large broken	13.9	22	12.5	11	12.6	22
70 whole, 30 large broken	15.4	21	15.0	20	13.9	19
50 whole, 50 large broken	17.9	23	15.5	18		
90 whole, 10 small broken	16.9	52	16.0	46	15.8	54
70 whole, 30 small broken	23.1	47	20.7	39		
50 whole, 50 small broken	27.2	41	27.3	42		

* Experiments conducted approximately at 3 month intervals.

† Proportionate loss by calculation.

Table 3. Solids loss on cooking of parboiled rice with varying content of broken grain

Grain composition (% by weight)	Losses (% by weight)	
	Total solids loss	Broken grain loss*
100 whole	7.9	
90 whole, 10 large broken	10.7	36
70 whole, 30 large broken	13.7	27
90 whole, 10 small broken	17.0	99
70 whole, 30 small broken	19.2	46

* Proportionate loss by calculation.

higher than perhaps may have been expected owing to their almost complete loss at the 10% rate of addition. Kurien *et al.* (1964) have shown that parboiled rice absorbs more water but at a slower rate than raw rice, leading to an increased cooked volume, which suggests that its solids are less soluble, at least at the grain surface, where gelatinization would be expected to be greatest. However, at the centre of the grain, gelatinization may be incomplete owing to poor process control and when the grain is broken open the central solids may be lost at an increased rate during cooking. The parboiled rice used in the experiment showed evidence of 'white core' (incomplete gelatinization) and the defect may account for the relatively higher losses from broken grain shown by the study.

The origin of the broken piece, raw or parboiled, is likely to effect its resistance to dispersion on cooking. It is reasonable to suppose that grain pieces which have broken during processing, perhaps as a result of structural weakness, may be at a greater risk than those used in this study which were purposely broken.

Conclusion

In attempting to quantify the losses associated with the breakage of rice, the present study has shown that losses of solids of up to one-fifth or more of the material can readily occur during cooking and preparation of a mixture of grain sizes. However, the losses sustained by the individual fractions, in particular the small brokens (the fraction most at risk) can be much higher.

When rice is prepared by cooking and rejecting the excess water, the loss of gross food value on such a scale could well be classified as a major post-harvest loss.

Acknowledgments

The author extends his thanks to Suzanne Brown, Ruth Kasasian and Dr D. A. V. Dendy of TPI for their assistance and interest, and to Fazlul Huq, Ministry of Food, Bangladesh, who, as part of a training programme at TPI, undertook some of the benchwork.

References

AACC (1969) *Approved Methods,* 44–19 Am. Assoc. Cereal Chem. Inc., St Paul, Minn.

Barber, S. (1972) *Rice: Chemistry and Technology*, (Ed. by D. F. Houston) p. 215. Am. Assoc. Cereal Chem. Inc., St Paul, Minn.

Batcher, O.M., Staley, M.C. & Deary, P.A. (1963) *Rice J.* **66** (9), 19 and (10) 13.

Chattopadhyay, P.K. (1977) Pers. commun.

Cheigh, H.S., Ryu, J.H., Jo, J.S. & Kwon, T.W. (1978) *Food and Fertiliser Technology Centre Ext. Bull. 103,* Taipei City, Taiwan.

Dendy, D.A.V. (1978) *Proc. 11th Nutr. Congr.* Rio de Janeiro.

FAO (1972) *Recommended Model Grading System for Rice in International Trade* (revised).

Kurien, P.P., Radhakrishna Murthy, R., Desikachar, H.S.R. & Subrahmanyan, V. (1964) *Cereal Chem.* **41,** 16.

Raghavendra Rao, S.N. Juliano, B.O. (1970) *J. Agric. Fd Chem.* **18,** 289.

Swaminathan, M. (1942) *Ind. J. Med. Res.* **30,** 409.

(Received 4 September 1981)

J. Fd Technol. (1982) **17**, 513–515

Technical note: Resazurin test for microbiological control of deep-frozen shrimps

R. KÜMMERLIN

Introduction

The microbiological examination of exuded tissue juice of deep frozen shrimps when thawed quickly at 35–40°C yields results similar to those of homogenates obtained by mechanical blending of the food (Mitchell, 1970; Kümmerlin, Vega & Lillo, 1975; Kümmerlin, 1976). Thus, these exudates can be considered undiluted aliquots of the solid sample. As such, they have proven suitable for bacteriological analysis by miniaturized and/or simplified techniques (Kümmerlin *et al.*, 1975).

In search of methods for industrial quality control, a resazurin reduction test (Otsuka & Nakae, 1969) was modified and assayed on exudates of IQF shrimps.

Materials and methods

Samples of pre-cooked, pasteurized and glazed IQF shrimps from local sources were thawed for 30 min in a water bath at 35°C. Exuded tissue juices were aseptically collected and kept in cold storage until required. Exudates within a wide range of microbial loads were obtained by incubation of some of these juices at 35°C for variable periods.

Sterile antibiotic assay filter paper discs (Whatman AA, 13.0 mm) were aseptically dipped into an aqueous 0.005% (w/v) resazurin solution, dried for 2 hr at 55°C and placed in sterile 20 × 40 mm polythene pouches, which were then heat-sealed and stored at 5°C in the dark until used.

Up to ten replicate dye-impregnated discs were dipped into each test exudate and then returned to their plastic envelopes. After removing excess liquid and air from the pouches, they were re-sealed and incubated in a light tight water bath at 35°C. Parallel viable plate counts of the same exudates were performed on standard plate count agar (Difco) and incubated at 35°C for 48 hr.

The colours of the impregnated discs were visually compared at half-hour intervals with the Methuen Colour charts (Kornerup & Wanscher, 1978).

Author's address: Departamento de Microbiología, Facultad de Ciencias Biológicas y de Recursos Naturales, Universidad de Concepción, Casilla 2407, Concepción, Chile.

0022–1163/82/0800–0513 $02.00

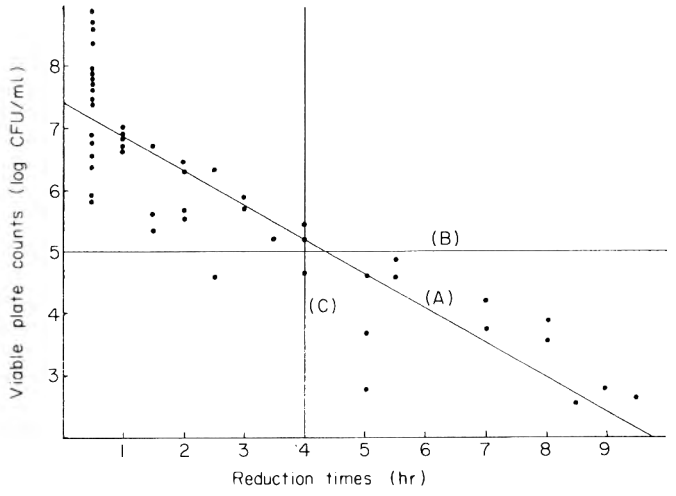


Figure 1. Relationship between resazurin reduction times and viable plate counts of exudates from IQF shrimps. (A) Least squares line of best fit ($y = 7.372 - 0.545x$); (B) bacteriological acceptance standard; (C) limit reduction time.

Previous trials had shown that very slight colour changes could be distinguished due to the opacity of the filter paper. The time elapsed to reach the first discernible difference (from Methuen 21A5 to 20A5) was set as end point – i.e. reduction time – of the test.

Results and discussion

Figure 1 shows the relationship between reduction times and viable plate counts of fifty test exudates. Both parameters correlated well ($r = -0.88$), although the colour readings at half-hour intervals caused a misleading spreading of the points on the scatter diagram. The reduction times of replicate tests were, without exception, remarkably uniform.

A reduction time of 4 hr discriminated well between samples that complied or not comply with the normally used bacteriological limit of 10^5 CFU/g, and did not differ significantly from the corresponding plate counts (chi-square $[\chi^2] = 0.857$; d.f. = 1). The dye reduction test rejected three samples with acceptable plate counts, but no false negatives were recorded after the cited test time.

With regard to the possible industrial application, the assay should be useful for final product inspection, where simultaneous testing of several samples and short report times are advantageous. Its simplicity and low cost makes it apt for situations of restricted laboratory facilities.

Acknowledgments

The author is indebted to Dr R. Davies, National College of Food Technology, University of Reading, for helpful suggestions during part of this work.

References

Kornerup, A. & Wanscher, J.H. (1978) *Methuen Handbook of Colour,* 3rd ed. Eyre Methuen, London.
Kümmerlin, R. (1976) M.Sc. thesis, University of Reading, U.K.
Kümmerlin, R., Vega, Y. & Lillo, S. (1975) *Bol. Inst. Bact. Chile,* **17,** 31.
Mitchell, N.J. (1970) *J. appl. Bact.* **33,** 523.
Otsuka, G. & Nakae, T. (1969) *J. Diary Sci.* **52,** 2041.

(*Received 28 March 1980*)

J. Fd Technol. (1982) **17**, 517–520

Technical note: Trinitrobenzenesulphonic acid and ninhydrin reagents for the assessment of protein degradation in cheese samples

K. M. CLEGG, Y. K. LEE AND J. F. McGILLIGAN

Introduction

The enzymic hydrolysis of proteins for dietary purposes in the treatment of clinical conditions and to alter their functional properties has been receiving attention in recent years. The degree of hydrolysis, i.e. the percentage of peptide bonds cleaved, is generally determined by measuring the increase in free NH_2 groups by reaction with ninhydrin. However, Satake *et al.* (1960) introduced trinitrobenzenesulphonic acid (TNBS) as an alternative reagent for the determination of NH_2 groups and this method has been recommended and modified by Adler-Nissen (1979).

Cheese manufacture is based on the controlled enzymic hydrolysis of casein by the activity of the mild proteolytic micro-organisms of the starter culture during the subsequent ripening. Protein degradation in some varieties of cheese is enhanced by the addition of moulds, which are relatively highly proteolytic, prior to ripening. In addition to cleavage of peptide bonds, ammonia is also released during the maturation of cheese.

The present study was undertaken to compare the merits of TNBS and ninhydrin for the determination of the extent of degradation of protein in different varieties of cheese. Ninhydrin is known to give approximately the same colour intensity for ammonia as amino acids on a molar basis, whereas the situation as to whether TNBS reacts with ammonia has not been resolved; Kossman (1971) stated that ammonia does not form a complex with TNBS and Adams *et al.* (1976) stressed that contamination with ammonia must be avoided in the analysis of amino acids.

Materials and methods

Varieties of cheese were purchased locally: Double Gloucester (hard), Edam (semi-hard), Stilton (internally mould-ripened), Brie externally mould-ripened).

Authors' address: Department of Food Science and Nutrition, University of Strathclyde, 131 Albion Street, Glasgow G1 1SD.

0022–1163/82/0800–0517 $02.00

Before undertaking the colour reactions, the macerated cheese samples required to be defatted to prevent the formation of a turbid extract. In the case of the soft cheeses, the water content was removed by freeze drying before Soxhlet extraction with diethyl ether; for the hard cheeses, the moisture was reduced during the oven drying (< 60°C/24 hr) of the defatted material. The total nitrogen content of the cheese protein samples was determined by the micro-Kjeldahl method.

The TNBS reaction was carried out according to the procedure of Adler-Nissen (1979) after dispersing 100 mg samples of defatted cheese in 100 ml 1.0% sodium dodecyl sulphate (SDS) solution; a further 1:10 dilution with SDS was made and 0.25 ml was taken for analysis. A leucine standard (0.25 ml containing 1.25 μg amino nitrogen) and an SDS blank were run with every analysis. The absorbance of the TNBS complex was measured at 340 nm and the reading for the blank was subtracted.

The ninhydrin procedure was a modified version of that described by Yemm & Cocking (1955). The original SDS extracts of cheese protein prepared for the TNBS procedure were diluted 1:100 and 1.0 ml samples were taken for reaction with ninhydrin; the concurrent 1.0 ml standard contained 2.0 μg leucine amino nitrogen and the reading for the blank was substracted from the experimental absorbance values obtained at 570 nm.

Ammonia determinations were carried out with 1.0 g samples of whole cheese or defatted cheese protein by steam distillation in a Tecator unit in the presence of 10 ml 0.05 N NaOH. The liberated ammonia was trapped in 4% boric acid solution and titrated with 0.02 N HCl. Under these mild conditions, it was found that less than 8% of the total amide nitrogen of pure glutamine was converted to ammonia; therefore, any contribution to the ammonia content due to degradation of protein during analysis would be negligible.

Results and discussion

A leucine standard was used with both spectrophotometric analyses; ninhydrin showed greater absorbance than TNBS for the same concentration of amino nitrogen when read at the respective optimal wavelength. Equal molar concentrations of leucine nitrogen and ammoniacal nitrogen, when reacted separately with TNBS, showed that ammonia yielded only 20% of the absorbance of amino nitrogen; when combined for reaction with TNBS, the absorbance was additive.

The mean of four to six replicates for the amino nitrogen contents of the defatted cheese samples, based on leucine equivalents and determined with ninhydrin and TNBS, are given in Table 1; data for the ammoniacal nitrogen contents determined by steam distillation are also included. The values obtained with ninhydrin were consistently higher than with TNBS, the discrepancy being greater for the varieties with higher ammonia contents. When the TNBS values were adjusted for the ammonia contribution and then subtracted from the

Table 1. Amino nitrogen (leu NH_2 eqv.) and ammoniacal nitrogen contents of defatted dried cheese samples

Cheese	Amino nitrogen (mg/100 mg N)			Δ (a–c)	Ammoniacal nitrogen (mg/100 mg N)
	Ninhydrin	TNBS			
	(a)	(b)* (b – 20% of the NH_3 nitrogen)	(c)		Steam distillation
Double Gloucester (hard)	6.3	5.1	4.8	1.5	1.4
Edam (semi-hard)	7.4	6.0	5.7	1.7	1.6
Stilton	14.0	11.5	10.7	3.3	4.2
Brie – cream	12.4	8.0	6.9	5.5	5.3
Brie – rind	13.0	8.4	7.1	5.9	6.3

*s.d. = ±0.09, except for Stilton where s.d. = ±0.21.

ninhydrin values, the difference between the two methods is in agreement with the ammonia content determined directly. The errant result for Stilton cheese arose from problems in obtaining homogeneous test material despite thorough maceration.

It is emphasized that the above findings are for defatted cheese protein and not for whole cheese. Some ammonia would inevitably be lost during the drying stages associated with the pre-treatment of the samples in preparation for spectrophotometric analysis. However, the steam distillation method for the

Table 2. Ammonia content of cheese samples before and after defatting and drying

Cheese	Method of drying	pH	Ammoniacal nitrogen (mg/100 mg N)		
			Whole cheese (a)	Defatted dried cheese (b)	Loss during sample preparation (a–b)
Double Gloucester	Air*	5.45	2.0	1.4	0.6
Edam	Air*	5.70	1.7	1.6	0.1
Stilton	Freeze dried	6.38	6.7	4.2	2.5
Brie – cream	Freeze dried	7.10	7.2	5.3	1.9
Brie – rind	Freeze dried	7.38	10.2	6.3	3.9

*Post-Soxhlet extraction in air oven, <60°C/24 hr.

determination of ammonia is equally applicable to whole cheese or defatted cheese and the results in Table 2 show, as expected, that the varieties with the higher pH values lost more ammonia during dehydration and fat extraction.

Using defatted cheese containing free NH_2 groups and ammonia as the test material, it has been established that analysis with ninhydrin will yield a comprehensive value for these two components; on the other hand, the TNBS reagent reacted primarily with NH_2 groups but the presence of ammonia made a small contribution to the overall value. Although TNBS is not as sensitive as ninhydrin it is recommended as the preferred reagent for the measurement of NH_2 groups because of the simpler analytical procedure; for increased precision of the TNBS method, an adjustment can be made for the ammonia content determined in a separate analysis and application of a 0.2 correction factor.

References

Adams, C.A., Roberts, T.C. & Butler, K.C. (1976) *Anal. Biochem.* **70,** 181.
Adler-Nissen, J. (1979) *J. Agric. Fd Chem.* **27,** 1256.
Kossman, K.T. (1971) *Ärztl. Lab.* **17,** 375.
Satake, K., Okuyama, T., Ohashi, M. & Shinoda, T. (1960) *J. Biochem.* **47,** 654.
Yemm, E.W. & Cocking, E.C. (1955) *The Analyst,* **80,** 209.

(*Received 18 July 1981*)

J. Fd Technol. (1982) **17**, 521–529

Book reviews

Nutritional Improvement of Food and Feed Proteins. Ed. by M. Friedman. New York: Plenum Press, 1978. Pp. xiii + 882. ISBN 0 306 40026 X. $69.50.

During the 1950s and 1960s malnutrition in the developing world was thought to be caused by a lack of protein in the diet. In the 1970s protein and energy requirements of humans were reassessed and this led to lower levels of protein being recommended for all human groups by FAO. Using these lower recommended daily allowances, data collected from surveys in different parts of the world were reassessed. This re-analysis suggested that the course of malnutrition was not a lack of protein *per se*, as much as a lack of total energy (or food). Of course certain communities which eat staples particularly low in protein such as cassava, plantain or sago, may have protein intakes lower than recommended levels, but for most staples (e.g. rice, wheat), as long as energy requirements are fulfilled, then enough protein will be supplied, even allowing for protein quality.

Against this background *Nutritional Improvement of Food and Feed Proteins* was published in 1978. The aim as stated early in the Preface was to help solve the shortage of protein as 'five hundred million people are presently estimated to suffer protein malnutrition, with about fifteen thousand daily deaths'.

This book, although primarily aimed at solving the world 'protein' problem, has other subsidiary aims. These were to assess critically (1) methods of measuring protein malnutrition and (2) the improvement of quality and quantity of protein for humans and animals.

To achieve these aims, subject matter ranges over a wide area of topics, such as:

(a) improvement of protein quality by genetic methods;
(b) fortification of foods and feeds with essential amino acids;
(c) enrichment of foods and feeds with protein supplements;
(d) use of special processes such as the pastein reaction to maximize protein quality and utilization;
(e) use of protected amino acids and protein in ruminant feeds to increase meat, milk and wool production;
(f) chemical and microbiological syntheses of essential amino acids;
(g) interactions of supplemental amino acids or proteins with other food ingredients during storage and processing and ways to minimize such undesirable interactions;

(h) economic aspects, including calculation of least cost–optimum quality supplemented foods and feeds;
(i) animal and human feeding tests of nutritional availability of amino acids or proteins in supplements; and
(j) safety of supplemented or specially processed foods and feeds.

For these purposes outstanding scientists from ten different countries were chosen to contribute to the book 'to bring together ideas and experiences needed for synergistic interaction among different yet related disciplines'.

The aims of the book are therefore very broad and this is reflected in the diverse nature of the forty chapters. However, the chapters do not make a cohesive whole, rather they remain as excellent reviews in their own right with little to link them together. Additionally, reasons for the presence of a chapter (No. 10) by G. A. Spiller *et al.* on 'Defining dietary plant fibres in human nutrition' is not at all clear and generally adds to the lack of continuity.

These reviews would interest a wide range of people, including those in protein chemistry, agriculture, food science, animal and human nutrition and biochemistry.

Ann F. Walker

Living Nutrition, 3rd edn. By F. J. Stare and Margaret McWilliams. New York: John Wiley & Sons, 1981. Pp. vi + 580. ISBN 0 471 04940 9. £10.50.

As the preface of this book states, nutrition is a unique and fascinating field of study which embraces the biological, physical and behavioural sciences. The subject is made even more interesting, however, by the good presentation and layout of the text. Throughout the book, tables and illustrations are used effectively to present information, and to stimulate the reader as the chapters unfold. The refreshing style of this book is certainly likely to generate enthusiasm.

This 580 page hardback book consists of nineteen chapters and eight very useful appendices. The introductory chapter, 'Nutrition – a Science of Living', summarizes the physiological roles of the various nutrients, human nutritional requirements and provides a practical guide to the four food groups. Chapters 2, 3 and 4 form a section which looks at the psychological aspects of nutrition. Chapter 2 discusses the worldwide concern over population growth, agricultural production and food distribution, and presents an excellent overview of various ethnic and cultural food patterns. Chapter 3 reviews the important factors which have influenced nutritional studies in the U.S.A. and describes the various surveys of food consumption and Federal programmes which have been implemented to improve nutrition. Chapter 4 explores 'Nutrition – Myth and Reality' and the sources of misinformation, unresolved controversies and even magic that appear to influence what consumers buy. Health foods, natural foods, food additives, megavitamin therapy, vegetarianism and fad diets are all considered.

The following eight chapters study nutrition from the physiological viewpoint. Carbohydrates, lipids, proteins, energy needs and weight control, water, minerals, vitamins and 'chemical highlights' are taken in turn. The latter chapter perhaps reminds us that nutrition is, or should be, a science with a strong foundation in chemistry.

Chapters 13–17 discuss 'Nutrition Throughout Life' – namely 'Pregnancy and Lactation', 'Feeding the Infant', the 'Preschool Child', 'Nutrition in the School Years' and 'Nutrition for Adults' – the latter chapter highlighting the growing awareness of the problems of the elderly. The last two chapters consider the consumer in the market place, shopping practices, food preparation, food planning for the individual and family meals, as well as foods eaten away from home.

Each chapter is interesting and readable; each has very succinct summaries, a number of selected references – not perhaps as comprehensive as they could have been – suggestions for further reading, and a series of interesting questions to aid the reader to master the material presented.

The Appendices are a valuable addition to this book and include: 'Nutritive values of the edible part of foods' from the *Home and Garden Bulletin No. 72*, USDA, Washington, DC, 1971; the 'Zinc content of foods'; 'Foods that are important sources of nutrients' (this section is most useful); 'Vitamin structures'; 'Amino acid structures'; 'Recommended daily allowances from selected countries'; a general description of 'Common ethnic foods'; a 'Glossary of terms'.

Overall, this American book serves as a useful introductory text and, as a teaching aid, its major value would be to stimulate the student and create an enthusiastic approach to this exciting area of science.

D. P. Richardson

The Analysis of Dietary Fiber in Food. Ed. by W. P. T. James and O. Theander.
Basel, Switzerland: Marcel Dekker AG, 1981. Pp. viii + 276. ISBN 0 8247 1192 0. Sw Fr. 78.

The hypothesis that there is a link between the lack of fibre in the diet and the high incidence of certain degenerative diseases in industrialized countries, has led to considerable research effort in this field over the last decade. As research proceeded it became clear that not only was the total dietary fibre in the diet of interest, but also its composition. Dietary fibre is composed of celluose, hemicelluloses (water soluble and insoluble) and lignin and each of these has unique properties. Their proportions in dietary fibre from different food materials varies greatly.

Several methods have now been described for dietary fibre analysis, but that of Southgate (1969) yields most information. Unfortunately, in its original form

this method is time consuming and requires considerable skill. Various modifications to the Southgate (1969) method have now been published. These aim, in particular, to reduce time of analysis. In this book detailed descriptions of dietary fibre methods are given, with comparative data. The fifteen chapters are written by eminent workers in the field from different parts of the world. These authors analysed a series of the same food samples which showed a wide variety of dietary fibre contents and composition. Results of these analyses are presented, and reproducibility and comparability of methods is critically assessed. Aspects of analysis which receive particular attention are the necessity for the pre-treatment of samples prior to analysis and the analysis of starch.

As this book gives detailed analytical data not published elsewhere, these data are sure to be of value for future research in this field. In summary, this book will be a necessary addition to the bookshelves of all research workers in the field of dietary fibre. For those seeking methods of routine analysis, some of the details may prove confusing. However, dietary fibre analytical methodology has not yet been developed to the stage at which a single standard method can be recommended.

Southgate, D.A.T. (1969) *J. Sci. Fd Agric.* **20,** 326.

Ann F. Walker

The Biochemistry of the Carotenoids (Volume 1, Plants), 2nd edn. By T. W. Goodwin.
London: Chapman & Hall, 1981. Pp. xviii + 377. ISBN 0 412 21690 6. £27.50.

The carotenoids are uniquely important among natural colouring matters because they represent the ultimate source of vitamin A, an essential dietary requirement in all animals. As pigments, they are extremely widespread: in plants, they provide most yellow to red colours of flower and fruit; in animals, they are found in many different groups from the lobster to the flamingo. Besides colouring reproductive tissues in higher plants, they occur hidden by the chlorophylls in all green leaves and through their ability to absorb visible light, have a variety of other functions. In particular, they are accessory light receptors in photosynthesis and play a role in both photoprotection and phototropism.

Because of their central importance in both plant and animal metabolism, the biochemistry of carotenoids has received a great deal of attention and an enormous literature has developed, particularly during the last two decades. It was, therefore, a formidable if not Herculean task that Professor Goodwin set himself when he agreed to prepare a second edition of his classic monograph on carotenoid biochemistry published in 1952. Indeed, so much has happened in this field that the work has spread to two volumes. The present review is concerned only with the first volume dealing with plant carotenoids; a second volume covering animal carotenoids will appear subsequently.

Professor Goodwin has generally followed the same outline as in the first edition, except for the introduction of two new chapters near the beginning on

biosynthesis and function. The book begins logically with an introductory account of carotenoid chemistry, covering particularly nomenclature, stereochemistry and spectral properties. Since some 400 carotenoids have now been characterized, this chapter might have been extremely extensive, but fortunately the author is able to provide a cross-reference to the book on carotenoid chemistry edited by O. Isler and published in 1971, where nearly all these structures are illustrated and enumerated.

Carotenoid biosynthesis had hardly been studied at all in 1952, when the first edition appeared. By contrast, in 1980 the main pathways are now very well established and in the second chapter, the author is able to provide a succinct and authoritative treatment of a topic, to which he and his co-workers have made the major research contribution. Chapter 3, which is also new, discusses briefly the various functions of carotenoids, many aspects of which are not fully understood; this is an area where further advances can be expected in the future. The remaining chapters follow the outline of the first edition, but of course are much expanded in view of the many more recent discoveries. They cover in turn the occurrence of carotenoids in seed-bearing plants (separately in reproductive and in photosynthetic tissues), in mosses and liverworts, in algae, in fungi and in different kinds of bacteria. A final brief new chapter discusses the biogeochemistry of these ubiquitous substances, derivatives of which have been able to survive in sediments and deposits for many thousands of years.

Needless to say in view of the author's outstanding scientific distinction, this book is beautifully written, excellently illustrated with many tables, diagrams and figures and is a joy to read. It will undoubtedly remain the major source of information on the natural distribution of carotenoids for many years to come. It will be of value and significance to all those working in the plant sciences. Food technologists, concerned with the preservation of carotenoids during fruit or vegetable processing, will need to consult this book regularly and all those research workers studying carotenoids themselves will need to have this book on their own laboratory shelves.

J. B. Harborne

Algae Biomass: Production and Use. (Proceedings of an International Symposium on Production and Use of Micro-algae Biomass held at Akko, Israel, in 1978; updated to 1980.) Ed. by G. Shelef and C. J. Soeder.
Amsterdam: Elsevier, 1980. Pp. xvii + 852. ISBN 0 444 80242 8. U.S.$149.00, D fl. 305.00.

The purposes of this book are well introduced by two quotations from the editors of this book:

> 'Assembling together most of the researchers, scientists, engineers and biotechnologists involved in micro-algae mass cultures and algal biomass production, processing and use, while bringing together in a published book the treasure of scientific findings and technological knowhow, has long been

sought by us during our long association in the German–Israeli algae research and development endeavour.'

'The last such event took place in the 1952 Algae Mass-culture Symposium held in California at Stanford University, assembling together the "pioneer giants" of this field such as H. Tamiya, B. Kok, J. Myers and others. This 1952 Symposium which provided the far-reaching foundations to present work yielded the book edited by J. S. Burlew, "Algae Culture – from Laboratory to Pilot Plant" (Carnegie Institution of Washington, Publication No. 600), which despite being based on relatively short-term outdoor experiments with limited outdoor facilities while reflecting somewhat idealistic and occasionally naive approaches, has still been, until today, the most frequently quoted and the best reference book in existence on algae culture biomass production.'

The editors' intentions were realized to a large degree and a comprehensive account of the field up to 1978 is presented.

The main objectives in algal culture have been either food protein production or effluent purification exploiting the oxygen evolved in photosynthesis; additional new objectives are production of organic chemicals and biomass as a renewable energy source. The intrinsic merits of algal culture systems, in contrast to conventional agriculture, are the generally higher yield of biomass per hectare and the possibility of developing it on areas of barren ground or over water, where conventional agriculture is impossible.

The main species of micro-algae cultivated belong to *Chlorella, Scenedesmus* or to the cyanobacteria, *Spirulina*. The photosynthetic culture process is strictly analogous to fermentation technology with light as the energy source and carbon dioxide as the source of carbon. However, the great development of fermentation process kinetics and the understanding of the controlling factors achieved over the past two to three decades has strangely barely penetrated into the algal culture field. This is borne out by the papers in the section of the book devoted to physiological and environmental factors affecting algal systems. The discussion of the kinetics makes vague references to 'self shading' and the 'flashing light effect' which have remained ill-defined and a cover for lack of understanding of the control mechanisms involved.

The means proposed for realizing algal culture are open ponds or channels with recycle of culture. These are simple primitive means or 'low technology', which reflect the lack of analysis of the controlling factors. For instance, it seems to be accepted that the channel system should have a culture depth of 20 cm or more which give maximum algal concentrations usually under 1.0 g dry weight per litre. Such a low concentration poses problems in economic means of harvesting the culture. But since the algal concentration varies inversely as the depth with a given light input there is obvious scope for increasing the algal concentration substantially. Essentially, with regard to the algal concentration in the open channel system no progress has been made in the last 30 years, although there is enormous scope to do so.

There are reports on pilot plant experiments on algal protein production in nine different countries, all in the tropics or warm temperate lands. The algal protein sources are proposed as food for humans, farm animals, fish and molluscs; however, the schemes have got little further than design studies. The toxicology and nutritional aspects of algal protein are extensively treated. Unexplained toxic effects were encountered if the product exceeded more than about 10% of the food intake of poultry. The algae were a potent source of carotenoids which coloured the flesh of poultry, but appeared not to contribute to vitamin A synthesis. Algal protein sells as a health food in Japan at consumer prices up to \$100 kg^{-1}. Considerable information on the costs of production are presented. A major factor in the cost is the burning of fuel to provide the carbon dioxide required. Again this illustrates lack of process development since algal culture could be regarded as a means of recycling carbon dioxide from the numerous industrial sources in which it is an effluent.

This book is timely and could mark the end of one era and the beginning of the new one, in which, as the editors predict, 'micro-algae biomass production will definitely play a major role in the effective conversion of solar energy through intensive photosynthetic systems'.

The editors are to be complimented on the care they have taken in their considerable task of ensuring a good finish to the papers. There is a valuable index. The book constitutes essential reference material to all workers in the field.

S. J. Pirt

Books received

The Science of Food: An Introduction to Food Science, Nutrition and Microbiology, 2nd edn. By P. M. Gaman and K. B. Sherrington.
Oxford: Pergamon Press, 1981. Pp. xii + 245. ISBN 0 08 025895 6. £5.50.

A textbook intended primarily for students following either TEC diploma courses in Catering or other related diploma courses in Food Science, Food Technology, etc. Suitable also for relevant secondary school courses.

The Aerobic Endospore-forming Bacteria: Classification and Identification. Ed. by R. C. W. Berkeley and M. Goodfellow.
London: Academic Press, 1981. Pp. xv + 373. ISBN 0 12 091250 3. £15.00.

The fourth of the Special Publications of the Society for General Microbiology, derived from a symposium organized by the Systematics Group of the Society held at Cambridge in April 1979. It deals primarily with the genus *Bacillus*, including discussions of identification using rapid diagnostic aids; study using pyrolysis GLC; marine bacilli; insect pathogens; thermophiles; *B. cereus* and other food poisoning bacilli.

Thornton's Meat Hygiene, 7th edn. By J. F. Gracey.
London: Baillière Tindall, 1981. Pp. viii + 436. ISBN 0 7020 0831 1. £18.00.

The latest edition of a well-known text first published in 1949. The implications of advances and changes in food technology, in food legislation (both U.K. and EEC) and food hygiene aspects have been incorporated.

Kaffee (in German). By H. G. Maier.
Berlin: Paul Parey, 1981. Pp. ix + 199. ISBN 3 489 61414 3. DM 78.

A text which provides details of the analysis and composition of coffee and its products – raw, roast, extracts, decaffeinated products, etc.

Chemical Technicians' Ready Reference Hand Book, 2nd edn. By G. J. Shuger, R. A. Shugar, L. Bauman and R. Shugar Bauman.
New York: McGraw-Hill, 1981. Pp. xxii + 867. ISBN 0 07 057176 7. £27.95.

This handbook covers basic laboratory techniques and procedures such as how to handle reagents and solutions, procedures including heating and cooling, evaporation, agitation, filtration, recrystallization, sublimation, centrifugation, distillation, chromatography. Measuring techniques – e.g. weighing, measurement of moisture, temperature, pressure, vacuum, pH – are included. Although elementary glassblowing is included, fabrication of custom-built laboratory equipment (e.g. methods of working polymethyl methacrylate) is not covered. Throughout the book attention is paid to identifying hazardous substances and operations, and to recommending safe handling procedures.

Report of the Government Chemist, 1980.
London: HMSO, 1981. Pp. 201. ISBN 0 11 513474 3. £7.50.

The latest report of the work of the Laboratory of the Government Chemist includes topics such as screening of milk in Scotland for radioactive isotopes of iodine and caesium, monitoring of foods for pesticide and fungicide residues.

NIR '80. Ed. by C. R. H. Parsons.
Chorleywood, Rickmansworth: Flour Milling and Baking Research Association, 1981. Pp. 52. ISBN 0 907503 00 4. £4.00 (members) £5.00 (non-members).

The Proceedings of a Symposium on Near Infra-red Analysis held at Chorleywood in October 1980. Included is a discussion of NIR equipment and calibration and applications in the cereals industry, particularly grain trading and malting barley.

Nationwide Food Consumption Survey 1977–78.
Washington, DC: USDA, 1981.

Preliminary Reports:

3. Nutrient levels in food used by households in the United States, Spring 1977. Pp. ii + 16 (15 figs).
4. Food consumption and dietary levels of households in Hawaii, Winter 1978. Pp. ii + 29 (9 tables and 8 figs).
5. Food and nutrient intakes of individuals in 1 day in Hawaii, Winter 1978. Pp. vii + 66. (29 tables and 12 figs).

These are available free from the Consumer Nutrition Center, Federal Building, Hyattsville, Md 20782, U.S.A.

Tables of Food Composition: an Australian Perspective. Ed. by H. Greenfield and R. B. H. Wills.
Published in *Food Technology in Australia, Vol. 33, No. 3*, March 1981. Also available separately from School of Food Technology, University of New South Wales, PO Box 1, Kensington, NSW 2033, Australia (cost Aust. $3.00).

This thirty page report consists of the Proceedings of a Workshop held at the University of New South Wales in June 1980. The problems of compilation and the requirements of users are discussed; tables of food composition are not presented.

Directory of Association of Consulting Scientists, 1981–82.
Buntingford, Herts: Association of Consulting Scientists, 1981. Pp. 87. £2.00.

Provides details of the practices and expertise of the members of the Association of Consulting Scientists, with subject index.

J. Fd Technol. (1982) **17,** 530

Corrigendum

J. Fd Technol. (1982) **17,** 281–283

Cholesterol content of poultry meat and cheese determined by enzymic and gas–liquid chromatography methods

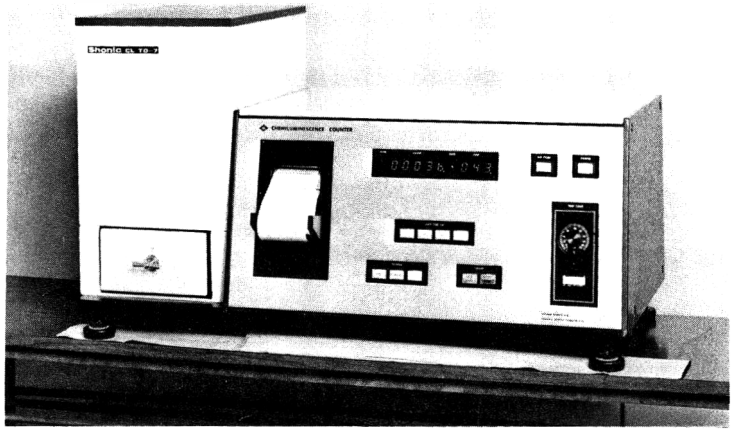
J. Karkalas, A. E. Donald and K. M. Clegg

On p. 283 the first two references should read

Kovac, M.I.P., Andersen, W.E. & Ackman, R.G. (1979) *J. Fd Sci.* **44,** 1299.

Paul, A.A. & Southgate, D.A.T. (1978) *The Composition of Foods,* 4th ed., p. 302. HMSO, London.

SHONIC CL
Chemiluminescence Counter



Shonic CL Chemiluminescence Counter is a new device to detect ultraweak chemiluminescence, using a single photoelectron system. (Sensitivity: 10^{-15} W). The ultraweak Chemiluminescence, which cannot be detected by human eyes, radiates from almost every material.

The application of this device is finding its way in many sectors, such as:

- Food industry (deterioration of fats, cooking oils, chocolate, potato chips, cakes, breakfast cereals, soda crackers, ground coffee and foods containing oils)
- Oil industry (deterioration analysis of kerosene, fuels, grease and oils)
- Polymer industry (deterioration analysis of plastics, rubbers, paints, resins, polypropylene, polystyrol, polyurethane, paints and other polymers)
- Technical products (autoxidation of solvents, epoxy coatings)
- Medical and biochemical research
- Discrimination of enzyme immunoassay
- Analysis of various chemical reactions which accompany CL.

For further details contact: **Dr KUBELIK (Co.), A.d. Guggelensberg 14, D-7500 Karlsruhe 41, F. R. Germany. Telex: 7825303 (KUB D).**

Journal of Chemical Technology and Biotechnology

The *Journal of Chemical Technology and Biotechnology* is concerned with the publication of new work carried out in the U.K. and overseas in the fields of applied chemistry and biotechnology and is an important source of information for industrial and academic chemists and related technologists. Topics include building and road materials, ceramics, metallurgy, dyes, resins, solvents, extraction ion exchange, fermentation and enzyme technology, industrial production of microbial protein, antibiotics, enzymes, vaccines and other biological products, biodegradation and effluent disposal.

Published on behalf of the Society of Chemical Industry, the journal is designed to combine quality and flexibility of presentation with rapidity of publication. Its critical but constructive editorial policy ensures that only work of the highest scientific standards is reported in its columns.

Subscription Information
Journal of Chemical Technology and Biotechnology is published monthly. Subscription rates for 1982 are: £57.50 (U.K.), £69.00 (overseas), $157.50 (U.S.A. and Canada) post free, including delivery by air freight to subscribers in the U.S.A. and Canada, and delivery by air freight to Singapore for forwarding by surface post to subscribers in Australasia, India and the Far East.

Order Form — send to **Blackwell Scientific Publications Ltd, P.O. Box 88, Oxford, England**
Please tick the appropriate box

☐ I would like to subscribe to *Journal of Chemical Technology and Biotechnology* and I enclose my remittance for the current volume

☐ I would like a free specimen copy of *Journal of Chemical Technology and Biotechnology*

Name .....

Address .....

.....

Blackwell Scientific Publications

Journal of the Science of Food and Agriculture

Published on behalf of the Society of Chemical Industry, the *Journal of the Science of Food and Agriculture* is an international journal concerned with the publication of research papers, notes and review articles in the fields of agriculture, the food sciences and other associated sciences. It is of interest to scientists and technologists in agriculture, the food industry and horticulture and deals with vegetable, fruit, meat, egg and milk technology together with all branches of agriculture.

Subscription Information
Journal of the Science of Food and Agriculture is published monthly. Subscription rates for 1982 are: £77.50 (U.K.), £93.00 (overseas), $215.00 (U.S.A. and Canada) post free, including delivery by air freight to subscribers in the U.S.A. and Canada.

Order Form — send to **Blackwell Scientific Publications Ltd, P.O. Box 88, Oxford, England**
Please tick the appropriate box

☐ I would like to subscribe to *Journal of the Science of Food and Agriculture* and I enclose my remittance for the current volume

☐ I would like a free specimen copy of *Journal of the Science of Food and Agriculture*

Name .....

Address .....

.....

Blackwell Scientific Publications

JOURNAL OF FOOD TECHNOLOGY: NOTICE TO CONTRIBUTORS

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

Typescripts (two complete copies) should be sent to the Editor, Dr H. Liebmann, c/o Institute of Food Science and Technology (U.K.), 105–111 Euston Street, London NW1 2ED. Papers should be typewritten on one side of the paper only, with a $1\frac{1}{2}$ inch margin, and the lines should be doubled-spaced. In addition to the title of the paper there should be a 'running title' (for page headings) of not more than 45 letters (including spaces). The paper should bear the name of the author(s) and of the laboratory or research institute where the work has been carried out. The full postal address of the principal author should be given as a footnote. (The proofs will be sent to this author and address unless otherwise indicated.) The Editor reserves the right to make literary corrections.

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

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

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

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

SI UNITS

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

NON SI UNITS

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

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

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

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

Journal of Food Technology
Volume 17 Number 4 August 1982

Contents

Printed by Adlard and Son Ltd, Bartholomew Press, Dorking