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Freeze-Chilling and Gas Flushing of Raw Fish Fillets

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Contents

FREEZE-CHILLING AND GAS FLUSHING OF RAW FISH FILLETS

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SUMMARY

Freeze-chilling involves freezing and frozen storage followed by thawing and chilled storage. It offers logistic benefits for fish packers as it enables packaged fillets to be held frozen and then released into the chill chain as required. Trials with whiting and mackerel fillets/portions (Part 1) indicated no difference in odour scores (raw samples) between freeze-chilled and chilled samples; however, freeze-chilled salmon portions were inferior to chilled in terms of odour. Fresh fillets received the highest acceptability scores as cooked samples followed by frozen, chilled and freeze-chilled fillets. The pattern in the data was the same for each species and there was no statistically significant difference between the freeze-chilled and chilled fillets. Freshness indicators (total volatile base nitrogen, trimethylamine and total viable count) were the same for the three species in that the chilled and freeze-chilled fillets had the highest values and the fresh and frozen the lowest. Freeze-chilled fillets had the highest free fatty acid and peroxide values but the levels were low and did not influence sensory response. Gravity drip was significant in the frozen and freeze-chilled fillets but presented no major visual problems and could readily be absorbed by drip pads. The effects of the four treatments on the colour and texture of the raw fillets were small in practical terms and typical shelf-lives in the chill phase of the freeze-chill process were 3 to 5 days. This is similar to that found for chilled fillets that had not been frozen before chilling.

In Part 2, modified atmosphere packaging (MAP) was combined with freeze-chilling to further extend the shelf-life of raw whiting, mackerel and salmon fillets/portions. The MAP packs for mackerel and salmon (60% N_2 / 40% CO_2) and for whiting (30% N_2 / 40% CO_2 / 30% O_2) maintained their shape during freeze-chilling whereas packs with 100% CO_2 were slightly imploded with concave sides. Typical shelf-lives in the chill phase for the freeze-chilled fillets were 5 (whiting and mackerel) and 7 (salmon) days. This compares with shelf-lives of 3 and 5 days respectively for freeze-chilled fillets in air. However, the use of fillets frozen at sea, as distinct from iced fillets, would further extend the shelf-life of the fillets in the chill phase of the freeze-chill process. Samples in MAP had lower total viable counts than samples in air for raw fillets/portions of each of the three species. However, MAP did not

influence odour or acceptability scores but had a variable effect (generally small) on fillet colour, springiness, drip loss and freshness indicators (total volatile base nitrogen / trimethylamine content, peroxide values and free fatty acid contents). Good manufacturing practice coupled with HACCP and careful tempering (thawing) are essential for the successful freeze-chilling of raw fish fillets. Packs should be labelled 'previously frozen' for consumer information. It is concluded that freeze-chilling with MAP is a suitable technology for extending the shelf-life of raw fish fillets.

Iced counter for seafood

PART 1: FREEZE-CHILLING OF RAW FISH FILLETS

INTRODUCTION

The market for seafood products in Europe has grown strongly in recent years, fuelled by increases in the average unit value of seafood products. Multiples are pushing suppliers to innovate to allow them to grow their share of the lucrative fresh/chilled seafood market and eliminate the requirement for low-yield fresh seafood counters (*Price Waterhouse Coopers, 2001*). Two methods which individually have considerable potential to extend the shelf-life of raw fish fillets are freeze-chilling and modified atmosphere packaging (MAP); a combination of the two could have a synergistic effect.

Freeze-chilling offers logistic and other advantages. For example: (i) foods can be prepared in bulk, frozen and stored at deep freeze temperatures until required. Some, or all of the batch can then be thawed as necessary; (ii) freeze-chilling enables chilled foods to reach distant markets in that product can be shipped deep frozen and then thawed when it reaches its destination prior to retail display; (iii) freeze-chilling can reduce the number of product recalls as it enables routine microbiological tests to be completed before the product is released from the factory. The objective in Part 1 was to assess the suitability of freeze-chilling as a technology for raw whiting and mackerel fillets and for salmon portions. Samples were compared as fresh, chilled, frozen and freeze-

chilled and were then subjected to physical, chemical and sensory tests to determine portion quality.

MATERIALS AND METHODS

For each species, four process treatments were compared: (i) fresh (tested on day of purchase with no storage); (ii) chilled (4°C for 3d); (iii) frozen (air-blast at -35°C for 3h, stored at –30°C for 3d and thawed overnight at 4°C) and (iv) freeze-chilled (blast frozen at -35°C for 3h, stored at -30°C for 3d followed by 3d of chilled storage at 4°C for whiting and mackerel, and 5d for salmon). Whiting and mackerel were purchased from a local fish company as skin-on fillets and were transported to the laboratory on ice (internal temperature 0-1°C). The whiting was tested between January and March and consisted mainly of 30-36cm specimens. Mackerel were tested between April and June and comprised mainly 29-38cm specimens. Farmed salmon were obtained as skinned boneless fillets and were transported to the laboratory on ice. They were tested between July and September and consisted of 2.5 year old fish (6-7 kg). The salmon fillets were cut into portions of about 250g. Twenty-five fillets were tested within 6h to provide data for the fresh samples. Ten randomly chosen fillets were used for sensory analysis (acceptability) and 15 for physical and chemical tests. The chill, freeze and freeze-chill process treatments also used 25 fillets per replicate. Fillets were weighed, placed individually into plastic (HDPE) trays (Dynopak, Ireland), and sealed with a film (340 mm Antifog high barrier film; Dynopak, Ireland). Six pinholes were punched in the film to maintain aerobic conditions. The trial was replicated five times over a 10-week period using new batches of fish each time. The experimental design was 4 process treatments x 5 replicates and the results were tested by analysis of variance (ANOVA).

Odour evaluation

The odour status of the prepacked fish from the chilled, frozen and freeze-chilled treatments was assessed by a sniff test using a panel of three trained assessors. The packs (one fillet or portion per pack) were opened and sniffed immediately (time 0) and again after 10 min (time 10) at room temperature.

The samples were scored on a scale from 1 to 6 (1 - fresh seaweed-like smell; 2 - odourless; 3 - slight fishy odour; 4 - significant fishy odour; 5 - strong fishy odour; 6 - totally-off, i.e. putrid smell). The data were tested by ANOVA as 3 process treatments x 2 times of testing (0 and 10 min) x 5 replicates. Three packs were assessed per process treatment per replicate.

Acceptability scores

An untrained sensory panel of 25 tasters was used for each of the five replicates. Panelists were asked to score the acceptability of a sample of fish (circa 50g) [steamed for 6 min (whiting), grilled for 6 min (mackerel) or steamed for 30 min (salmon)] on a 6 cm line from 0 (unacceptable) to 6 (very acceptable). The marked point on the line was measured with a ruler and the data were tested by ANOVA as 4 process treatments x 25 tasters for each individual replicate and also collectively as 4 process treatments x 25 tasters x 5 replicates. Only one sample was tasted each time as the samples from the four treatments were due for testing on different days.

Physico-chemical tests

The samples were tested for colour (Hunter Lab), texture (springiness and shear), centrifugal drip, gravity drip, free fatty acids (FFAs), peroxide values (PVs), total viable count (TVC), total volatile base nitrogen (TVBN), trimethylamine (TMA) and moisture content as described by Fagan *et al.*, (2003) and as outlined in Tables 3-5.

RESULTS AND DISCUSSION

Freeze-chill technology has logistic and other advantages in the preparation, distribution and retailing of chilled foods. It has particular application for prepacked fish fillets which are increasingly replacing the traditional iced counter in some supermarkets. The 3-day chilled storage times chosen for whiting and mackerel and the 5-days for salmon were based on pre-tests and on discussions with supermarkets on the likely shelf-life of prepacked chilled fish portions from fish currently being landed at Irish fishing ports or obtained

from Irish fish farms (in the case of salmon). This 3-5 day timespan in chilled storage agrees with the recommendations of Church (1998) but is considerably shorter than the extended chilled storage times for thawed cod in modified atmosphere packs reported by Bøknæs *et al.* (2001).

Odour tests

Odour testing was utilised to determine if there was an off-odour at time of pack opening and, if so, if it remained 10 min after pack opening. Such odours are off-putting for the consumer. The results showed no difference between the odour scores at times 0 and 10 min. With the exception of the chilled and freeze-chilled salmon portions, all odour scores were below the mid-point (i.e. 3.5) of the odour scale (1 - fresh seaweed-like smell; 6 - putrid) and could be classed as odourless or having a slight fishy odour i.e. they were acceptable (Table 1). The freeze-chilled samples received more favourable odour scores than the chilled but the data were not statistically significant except in the case of salmon. The freeze-chilled salmon portions also received middle ratings (3.60) but the chilled sample score of 5.61 indicated an off-odour and that 5 days at 4°C was the upper limit of its shelf-life (Table 1); these two samples also had the highest TVC values (Table 5).

Table 1: Odour (sniff) scores[a,b] for chilled, frozen and freeze-chilled portions of raw whiting, mackerel and farmed salmon

Species	Chilled	Frozen	Freeze-chilled	LSD[c]	F-test
Whiting	3.15	2.67	3.13	0.36	P< 0.05
Mackerel	2.86	3.00	2.83	0.28	NS[d]
Salmon	5.61	1.94	3.60	0.62	P< 0.001

[a] On a 6-point scale from 1 (fresh seaweed-like smell) to 6 (totally off i.e. putrid smell)

[b] Mean values for tests at pack opening and 10 minutes later

[c] Least significant difference

[d] Not significant

Taste panel acceptability

The scores indicated that the freeze-chilled portions received the lowest ratings but the results were not statistically different from the chilled samples indicating that freeze-chilling, in comparison with chilling, was not having a deleterious effect on acceptability (Table 2). The freeze-chilled samples were the oldest and could be expected to have the lowest ratings, whereas fresh should have the highest acceptability rating which was the case. However, the acceptability scores for all the samples were > 3.0 on a scale from 0 (unacceptable) to 6 (very acceptable) indicating that all were acceptable. The single stimulus taste panel procedure used in these tests proved satisfactory and 25 or more tasters were used each time.

Chemical indicators of freshness

The odour and acceptability data related well to the TVBN, TMA and TVC values for the different species from the four process treatments (Tables 3-5). The slight fishy odours encountered in the whiting, mackerel and salmon were most likely due, in part, to the production of TVBN and TMA. The measured TVBN and TMA values after 3-5 days at 4°C were relatively high. These were

Table 2: Taste panel acceptability scores[a,b,c] for fresh, chilled, frozen and freeze-chilled portions of whiting (steamed), mackerel (grilled) and salmon (steamed)

Species	Fresh	Chilled	Frozen	Freeze-chilled	LSD[d]	*F*-test
Whiting	4.52	3.70	4.16	3.60	0.32	P< 0.001
Mackerel	4.07	3.61	3.85	3.30	0.35	P< 0.001
Salmon	4.22	3.48	4.20	3.24	0.40	P< 0.001

[a] On a 6 cm line with endpoints of 0 (unacceptable) and 6 (very acceptable)
[b] Single sample hedonic panels; 25 tasters
[c] Collective data for 5 replicates
[d] Least significant difference

typical of values for commercially-landed fish in Ireland. There was no difference in the TVBN values between chilled and freeze-chilled samples of the three species and TMA values followed a similar pattern. As expected, the fresh and frozen samples had lower TVBN and TMA values than the chilled or freeze-chilled. In whiting, the fishy odour can be attributed to the production of TVBN and TMA predominately by spoilage organisms belonging to the genus *Pseudomonas*. The TVBN values were positively correlated with TVC [r = +0.62 (whiting); +0.77 (mackerel); +0.81 (salmon)] and negatively correlated with acceptability scores [r = -0.61 (whiting); -0.63 (mackerel); -0.65 (salmon)]. The TVC values were, in turn, negatively correlated with acceptability scores [r = -0.65 (whiting); -0.65 (mackerel); -0.81 (salmon)]. These data show that fillet acceptability decreased as TVBN, TMA and TVC values increased. The role of TVBN and TMA as indicators of fish quality has been discussed by Dalgaard (2000). The TVBN values in the current study were all below the EC guidelines (Council Regulation No. 95/149/EEC of March 1995) for raw fish (maximum of 35 mg N/100g flesh). The TMA values for mackerel were much higher than those of 1 mg/100g reported by Jhaveri *et al.* (1982) for mackerel stored at 0°C on ice for 4 days. In salmon, the lowest TVBN value was in the frozen sample and this could be due to a reduction in the TMA-producing bacteria caused by blast-freezing followed by storage at –30°C for 3 days.

Total viable count

The TVC values for the three species from the four process treatments were acceptable for raw fish (Department of Health and Children Guidelines, Ireland, 1992) with the exception of chilled and freeze-chilled salmon portions which had values above $\log_{10}$ 7 cfu/g (i.e. above 10 million colony forming units per gram) after 5 days in chilled storage. Fresh salmon portions had a TVC value of $\log_{10}$ 5.08 cfu/g which is indicative of slack in-factory hygiene practices. Specific spoilage organisms were not identified and the TVC data should be interpreted as indicators of hygiene (Tables 3-5). It was anticipated that freeze-chilling could be conducive to microbial growth as freezing opens up product structure and results in more drip than chilling alone. However, the data showed this was not the case as the freeze-chilled fish portions had lower TVC values than the chilled samples with the exception of salmon in which

the freeze-chilled sample was the same as the chilled. The TVC values for the chilled and freeze-chilled mackerel samples at $\log_{10}$ 5.34 and $\log_{10}$ 5.14 cfu/g were higher than the value of less than log 5 cfu/g reported by Jhaveri *et al.* (1982) for mackerel on ice for 4 days. Overall, the TVC values for the three species were generally slightly lower than those of Guldager *et al.* (1998) for modified atmosphere packed cod held on ice for 3-4 days.

Rancidity

Development of off-flavour is one of the major effects of lipid oxidation. The freeze-chilled mackerel samples had the highest FFA values (Table 4) but the concentrations were low and unlikely to affect sensory responses; this was borne out in the odour and taste acceptability panel results as rancid off-notes were never cited by the panelists. The FFAs were similar and the PVs lower than those reported by Gormley *et al.* (2002) for deep-frozen salmon and smoked mackerel.

Table 3: Effect of process treatments on a number of quality parameters for raw whiting fillets prepacked in air

Parameter	Fresh	Chilled	Frozen	Freeze-chilled	LSD[a]	F-test
Yellowness (b)	-0.79	+0.23	+0.90	+2.53	1.39	P< 0.01
Springiness (%)[b]	79.6	85.6	84.0	85.4	6.90	P< 0.05
Gravity drip (%)	-	1.0	9.0	6.0	1.13	P< 0.001
TVBN[c] (mg N/100g)	13.7	25.0	17.5	25.5	6.36	P< 0.01
TMA[d] (mg N/100g)	2.4	12.0	6.9	12.1	4.08	P< 0.001
TVC[e] ($\log_{10}$ cfu/g)	4.14	5.54	4.04	5.24	0.50	P< 0.001

[a] Least significant difference
[b] Recompression / compression x 100
[c] Total volatile base nitrogen
[d] Trimethylamine
[e] Total viable count

Table 4: Effect of process treatments on a number of quality parameters for raw mackerel fillets prepacked in air

Parameter	Fresh	Chilled	Frozen	Freeze-chilled	LSD[a]	F-test
Yellowness (b value)	4.4	6.9	7.9	7.9	1.43	P< 0.001
White/yellow ratio (L/b ratio)	9.4	5.9	5.0	5.7	1.45	P< 0.001
Colour difference (ΔE^b)	-	3.44	4.98	5.36	1.15	P< 0.05
Centrifugal drip (%)	5.1	5.7	10.1	9.6	3.18	P< 0.05
Gravity drip (%)	-	2.0	10.0	4.0	4.0	P< 0.05
FFA[c] (% oleic acid)	0.58	0.51	0.51	2.02	0.39	P< 0.001
TVBN[a] (mg N/100g)	15.9	23.1	17.9	21.1	3.70	P< 0.05
TMA[a] (mg N/100g)	3.9	8.7	3.5	5.3	2.98	P< 0.05
TVC[a] ($\log_{10}$ cfu/g)	4.52	5.34	4.26	5.14	0.56	P< 0.01

[a] See footnotes in Table 3

[b] $\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2}$

[c] Free fatty acids

Measuring salmon fill colour

Table 5: Effect of process treatments on a number of quality parameters for raw salmon portions prepacked in air

Parameter	Fresh	Chilled	Frozen	Freeze-chilled	LSD[a]	F-test
Whiteness (L value)	40.3	42.7	40.8	39.8	1.82	P< 0.05
Redness (a value)	21.2	17.7	19.0	17.4	1.08	P< 0.001
Yellowness (b value)	14.6	12.2	12.6	12.7	0.74	P< 0.001
White/yellow (L/b ratio)	2.78	3.52	3.24	3.14	0.24	P< 0.001
Centrifugal drip (%)	2.80	1.02	5.08	1.06	0.79	P< 0.001
Gravity drip (%)	-	1.7	3.3	3.2	0.35	P< 0.001
Moisture (%)	68.3	66.8	67.9	66.7	0.82	P< 0.001
FFA[b] (% oleic acid)	0.31	0.74	0.33	1.60	0.26	P< 0.001
Peroxide value (meq. perox /kg)	1.62	2.22	2.18	4.40	0.23	P< 0.001
TVBN[a] (mg N/100g)	17.0	22.7	14.8	20.2	2.33	P< 0.001
TMA[a] (mg N/100g)	2.1	6.0	1.7	4.1	0.91	P< 0.001
TVC[a] (log_{10} cfu/g)	5.08	7.36	4.78	7.56	0.20	P< 0.001

[a] See footnotes in Table 3

[b] See footnotes in Table 4

Fish colour

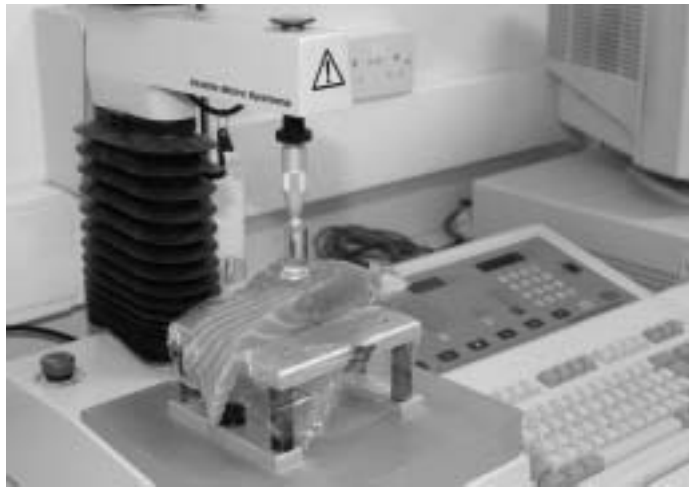
Raw fillet colour is of major importance as it is highly visible to the consumer both at time of purchase and during cooking in the home. Freeze-chilling promoted some yellowing in whiting fillets (Table 3) while, in mackerel and salmon portions, the chill, freeze and freeze-chill treatments gave an inferior colour to the flesh (Tables 4 and 5). In addition, the biggest colour difference (delta E) from fresh was in the freeze-chilled sample. However, all the colour effects were small in practical terms.

Fish texture

The freezing treatments (i.e. freeze or freeze-chill) did not influence the shear values of the raw fillets of any of the species and the only textural difference was the lower springiness value in fresh whiting fillets (Table 3). The frozen storage time of 3 days was short but some toughening could be anticipated if the frozen storage component of the freeze-chill treatment was extended to several months (Howell, 1995).

In-pack drip

Visible drip in the pack is off-putting for the consumer. The gravity drip was significant for the freeze and freeze-chill treatments (Tables 3-5) but



Springiness measurements on a salmon portion

presented no major visual problems; any potential negative effects could be overcome by the use of drip pads. Gravity drip for the frozen portions was higher than for the freeze-chilled and a similar effect was found for centrifugal drip in mackerel. An explanation may be that the fillets reabsorbed some of the drip generated by the freezing step during the chilled storage period. However, this was not reflected in the moisture contents of the fish samples.

GMP, HACCP and tempering

The use of good manufacturing practice (GMP) and hazard analysis and critical control point (HACCP) is imperative in the production, storage, distribution and retailing of freeze-chilled foods. National and EU guidelines for frozen and chilled foods should also be adhered to. Particular attention should be focused on the thawing step and careful temperature control exercised. In the case of freeze-chilled fish fillets, tempering can be achieved by transferring prepacks from the supermarket deep freeze room to the chilled retail display cabinets in the evening time, thus resulting in a tempered (thawed) product the following morning. The normal safety rules for frozen foods prevail in the frozen component of the process and those for chilled foods in the chill phase. The labelling requirements are those of conventionally chilled foods. However, it is desirable for reasons of consumer information and product liability to label the product as "previously frozen". A use-by-date must also be employed and this label should be attached at the start of the thawing process.

CONCLUSIONS

- Freeze-chilling is a suitable technology for prepacked whiting, mackerel and salmon portions and confers logistic and shelf-life benefits during distribution and retailing.
- Extended times of frozen storage (i.e. much longer than the 3 days used in the current tests) in the freeze-chill process should be tested to study potential deleterious changes in product quality including increased drip, toughening, oxidation and changes in colour.

PART 2: MODIFIED ATMOSPHERE PACKAGING (MAP) AND FREEZE-CHILLING OF RAW FISH FILLETS

INTRODUCTION

Modified atmosphere packaging (MAP) has been used successfully to extend the raw fillet shelf-life of many fish species (Cann, 1984; Church, 1998). Relatively little work has been done on the combination of freeze-chilling with MAP for extending the shelf-life of raw fish portions. The objective in Part 2 was to assess the suitability of freeze-chilling in combination with MAP for extending the shelf-life of raw whiting, mackerel and salmon portions. This combination will allow fish fillets to reach distant markets in a frozen condition followed by thawing for sale at retail outlets. The inclusion of a modified atmosphere should be beneficial in the chill storage phase (post-thawing) by inhibiting the natural spoilage organisms on the surface of the fillet thus extending the shelf-life.

MATERIALS AND METHODS

Whiting, mackerel and salmon were procured as outlined in Part 1. Twenty-five fillets/portions (ca 250 g) of each species were tested within 6 hours of purchase to provide data for the fresh samples. Ten were used for sensory analysis and 15 for physical and chemical tests. One hundred and fifty fillets/portions of each species were placed individually in plastic (HDPE) trays (Dynopak, Ireland) per replicate. Fifty fillets/portions (per species) were packed in air and these conditions were maintained by piercing 6 small holes in the film. Fifty packs were filled with premixed 60% N_2 / 40% CO_2 (mackerel and salmon) or 30% N_2 / 40% CO_2 / 30% O_2 (whiting) gas and the remaining 50 packs with 100% CO_2. All packs were sealed with a 340 mm Antifog high barrier film (Dynopak, Ireland) and were blast-frozen at –35°C for 2.5 hours. Packs were stored at –30°C for 3 days after which time they were placed in a chill room at 2 to 4°C for 5 (whiting and mackerel) or 7 (salmon) days. In the case of whiting and mackerel, 25 packs were tested on days 3 and 5 while for salmon, 25 packs were tested on days 5 and 7. A range of physical, chemical and sensory tests were carried out for each species as outlined in Part

1. Taste panel acceptability (25 tasters) was measured using a hedonic scale with a 6 cm line (0 unacceptable to 6 very acceptable) as cited by Fagan *et al.* (2003). On days 3 and 5, taste panelists were given three samples of fish (circa 50g) i.e. one sample from each gaseous atmosphere and were asked to mark a 6 cm line. The gaseous atmospheres in the packs were measured using a MAP-test 3050-gas analyser (Hitech Instruments, England). Four packs from each treatment per replicate were sampled before opening and the results were averaged. The experimental design was 3 gaseous atmospheres x 3 test days x 4 replicates and the results were tested by ANOVA.

RESULTS AND DISCUSSION

The MAP packs stood up well to the freeze-chill process and the 60% N_2 / 40% CO_2 (mackerel and salmon) or 30% N_2 / 40% CO_2 / 30% O_2 (whiting) packs maintained their shape (no implosion) throughout the storage period. However, all the 100% CO_2 packs imploded slightly, with the sides of the packs being concave. In the presentation of the results, the term MAP refers to packs with air or the various gas mixtures while day 0, day 3, day 5 and day 7 refers to MAP packs held at 2 to 4°C, i.e. in the chilled phase of the freeze-chill process.

The chosen shelf-life of 5-7 days in the MAP trials (2 to 3 days longer than freeze-chilling alone - see Part 1) was vindicated by the results as the products were near the end of their shelf-life (in acceptability terms) after 5 (whiting and mackerel) and 7 days (salmon). This is much shorter than the shelf-life of 15 days reported by Church (1998) for whiting stored in 100% CO_2 (4°C), and for salmon steaks (13 days) packed in 60% CO_2 / 40% N_2 as reported by Cann (1984). This difference in shelf-life may be due to the freshness of the fish at time of packaging and also to the different experimental conditions prevailing in the various studies.

Odour tests

Odour is an attribute immediately sensed by the consumer on opening a pre-packaged fish portion. However, if the fillets are in good condition, odours are minimal and will dissipate soon after pack opening as indicated by the results

for the MAP whiting (odour score decreased from 2.99 on pack opening to 2.65, 10 min later), mackerel (3.44 to 3.04), and salmon (3.04 to 2.71) (Table 6). Five or 7 day old portions had stronger odours than day 3 samples. However, all samples were below the midpoint of the odour scale (i.e. 3.5) which put them in the slight-fishy-odour category (Table 6). This indicated that the MAP samples had an acceptable odour after 5 (whiting and mackerel) and 7 (salmon) days. MAP treatment itself had no effect on the odour scores of the three species. However, an interaction between storage time (days) and MAP treatment ($P< 0.05$) showed that the 60% N_2 /40 % CO_2 and the 100% CO_2-stored salmon portions scored better than samples packed in air.

Table 6: Odour scores[a] for raw whiting, mackerel and salmon portions subjected to modified atmosphere packaging with freeze-chilling

	Whiting	Mackerel	Salmon
Time of sniffing[b] (min)			
0	2.99	3.44	3.04
10	2.65	3.04	2.71
F-test	P< 0.05	NS[e]	NS[e]
LSD[c]	0.28	0.51	0.36
Day of sniffing[d]			
3	2.57	2.96	-
5	3.01	3.52	2.63
7	-	-	3.12
F-test	P< 0.001	P< 0.05	P< 0.01
LSD[c]	0.28	0.51	0.36

[a] Scale from 1 to 6 (1 - fresh seaweed-like smell; 2 - odourless; 3 - slight fishy odour; 4 - significant fishy odour; 5 - strong fishy odour; 6 - putrid smell)

[b] Data averaged over days of sniffing and gaseous atmospheres

[c] Least significant difference

[d] Data averaged over times of sniffing and gaseous atmospheres

[e] No significant difference

Taste panel acceptability

Taste panel acceptability scores showed that there was no difference between cooked fillets from the three gaseous atmospheres for each species. However, the fresh fillets (no freeze-chilling or MAP) scored higher ($P<0.05$ to $P<.001$; Table 7) than some of the MAP samples. All fillets scored above the mid-point of the line which suggested that all were acceptable after 5 (whiting and mackerel) or 7 days (salmon) (Table 7). In the case of fatty fish (lipid content ca 10-30%) such as mackerel and salmon, off-tastes can be attributed to oxidative rancidity of the fat and also to microbiological spoilage. Mackerel fillets packed in 100% CO_2 had the highest FFAs while salmon in this atmosphere had the lowest values (Table 8). Storage time (days) influenced PVs for mackerel and salmon with the day 5 fillets having higher values than the day 3 samples (Table 9). The highest recorded FFAs and PVs for mackerel and salmon were well below those reported by Zotos *et al.* (1995) and were unlikely to adversely affect the sensory scores of the cooked products.

Table 7: Taste panel acceptability scores[a,b] for whiting (steamed), mackerel (grilled) and salmon (steamed) previously packaged in modified atmospheres and freeze-chilled

		Gaseous atmosphere					
Species	Fresh[c]	Air	30/40/30[d]	60/40[e]	CO_2[f]	*F*-test	LSD[g]
Whiting	3.93	3.46	3.50	-	3.18	P< 0.001	0.34
Mackerel	3.66	3.23	-	3.37	3.23	P< 0.05	0.32
Salmon	4.40	3.44	-	3.75	3.56	P< 0.001	0.61

[a] Scale from 0 (unacceptable) to 6 (very acceptable)
[b] For 5 replicates
[c] Day 0 (no freeze chilling or MAP)
[d] 30% O_2/40% CO_2/30% N_2
[e] 60% N_2/40% CO_2
[f] 100% CO_2
[g] Least significant difference

Table 8: Effect of modified atmosphere packaging with freeze-chilling on raw fish quality[a]

Quality	Gaseous atmosphere					
	Air	30/40/30[b]	60/40[c]	CO_2[d]	*F*-test	LSD[e]
Whiting						
Hunter b	2.59	2.91	-	3.07	P< 0.05	0.36
Gravity drip (g/100g)	4.71	9.40	-	16.4	P< 0.05	1.47
TVC[f]	4.81	4.48	-	4.34	P< 0.001	0.20
Mackerel						
Shear (kN)	1.95	-	1.94	1.99	P< 0.001	0.02
Springiness (%)	92.3	-	91.3	92.8	P< 0.01	0.87
TVBN[g]	18.7	-	17.6	18.0	P< 0.05	0.87
TMA[h]	3.88	-	2.84	2.91	P< 0.001	0.46
CD(g/100g)[i]	9.36	-	7.79	9.86	P< 0.01	1.21
Gravity drip(g/100g)	4.63	-	5.00	6.63	P< 0.001	0.65
FFA[k]	1.02	-	1.07	1.35	P< 0.05	0.23
TVC[f]	4.88	-	4.18	3.99	P< 0.001	0.16
Salmon						
Hunter b	14.2	-	14.5	15.7	P< 0.01	0.74
Hunter L/b	2.96	-	2.90	2.70	P< 0.05	0.18
CD (g/100g)[i]	2.08	-	2.78	3.58	P< 0.001	0.69
Gravity drip (g/100g)	2.45	-	4.68	5.84	P< 0.001	0.46
PV[j]	0.92	-	0.95	0.84	P< 0.01	0.06
FFA[k]	0.75	-	0.76	0.59	P< 0.001	0.08
TVC[f]	6.23	-	5.04	4.53	P< 0.001	0.32

[a] Data for five replicates averaged over three test dates

[b] 30% O_2 / 40% CO_2 / 30% N_2

[c] 60% N_2 / 40% CO_2

[d] 100% CO_2.

[e] Least significant difference.

[f] Total viable count (log_{10} cfu/g)

[g] Total volatile base nitrogen (mg N/100g flesh)

[h] Trimethylamine (mg N/100g flesh).

[i] Centrifugal drip (g/100g)

[j] Peroxide value (meq/kg fat).

[k] Free fatty acids (expressed as % oleic acid).

Table 9: Effect of storage time[a,b] (days) on the quality parameters of raw fish portions subjected to modified atmosphere packaging with freeze-chilling

	Time (days) after thawing					
	0	3	5	7	F-test	LSD
Whiting						
Hunter L	44.9	52.3	56.4	-	P< 0.05	7.20
Hunter a	1.69	-1.83	-2.07		P< 0.05	2.86
Hunter b	0.39	3.26	4.91	-	P< 0.01	2.19
Shear (kN)	1.61	1.54	1.54	-	P< 0.001	0.02
TVBN	13.5	17.7	24.2	-	P< 0.001	3.12
TMA	3.60	4.64	8.52	-	P< 0.001	1.70
CD (g/100g)	4.37	14.1	18.8	-	P< 0.001	5.11
TVC	2.97	4.88	5.76	-	P< 0.05	1.95
Mackerel				-		
Hunter b	4.42	8.73	9.07	-	P< 0.001	1.27
Shear (kN)	1.93	2.01	1.93	-	P< 0.05	0.05
TVBN	14.1	19.2	21.0	-	P< 0.001	2.47
CD (g/100g)	5.53	10.0	11.5	-	P< 0.001	2.13
PV	0.40	2.27	2.59	-	P< 0.001	0.64
FFA	0.25	1.42	1.78	-	P< 0.001	0.45
TVC	4.03	4.32	4.70		NS	1.17
Salmon						
TVBN	12.9	-	17.5	18.0	P< 0.01	2.03
CD	1.00	-	3.66	3.78	P< 0.05	1.82
PV	0.31	-	1.06	1.34	P< 0.01	0.46
FFA	0.31	-	0.81	1.01	P< 0.001	0.17
TVC	4.50	-	5.58	5.71	P< 0.01	0.62

[a] Data for five replicates averaged over gaseous atmospheres

[b] See footnotes, Table 8

Chemical indicators of freshness

The TVBN and TMA content of the MAP fillets were moderately high for whiting and mackerel but were much lower in the farmed salmon. This is attributed to the initial freshness of the salmon prior to freezing and confirms the need for high quality raw fish for MAP (Bøknæs *et al.*, 2000). In the case of whiting, MAP did not affect TVBN or TMA development but values were higher on day 3 and day 5 than on day 0 indicating that enzymatic activity (degradation of protein and TMA oxidase) continued at 4°C (Table 9). Mackerel fillets packed in 60% N_2 / 40% CO_2 had the lowest TVBN values while TMA values were lowest in the 100% CO_2 and 60% N_2 / 40% CO_2 gas mixture (Table 8). The TVBN and TMA levels for the three species were generally lower than those recorded for freeze-chilled fish fillets packaged in air. This suggests that gas flushing was better than packaging in air for freeze-chilled fillets. The TVBN content influenced acceptability scores for the three species (Table 10) (negative correlation) but was positively correlated with TVC values, particularly for whiting. TVCs were also negatively correlated with acceptability scores with whiting again having the highest coefficient.

Total viable count

The TVC values were in the order air (highest), $O_2/CO_2/N_2$ mixtures (intermediate) and 100% CO_2 (lowest) for all three species (Table 8) and were acceptable (i.e. below $\log_{10}$ 6 cfu/g) for raw fish fillets (Department of Health and Children, 1992 Guidelines) The values in the MAP packs were circa 1 log cycle lower than the TVCs for the freeze-chilled fillets packaged in air (see Table 3-5) and contributed to the additional shelf-life found with MAP. As in air packed products (Part 1), the TVCs for salmon were higher than those for whiting or mackerel. As expected, TVCs increased with time for the three species (Table 9). The microbiology of fish products has been reviewed by Shewan (1961) and low counts are a key indicator of fish freshness and good hygiene practices. Fish portions in the current trial were packed in MAP, blast-frozen at – 35°C and stored at – 30°C for 3 days prior to thawing. It is likely, therefore, that the freezing component of the freeze-

chill process had little impact on the TVC status of the fish portions since the frozen storage time was well below the 5 weeks suggested by Bøknaes *et al.* (2000) as the minimum frozen storage period required to reduce bacterial numbers in cod.

Rancidity

Mackerel fillets packed in 100% CO_2 had the highest free fatty (FFA) acid values while salmon fillets in this atmosphere had the lowest (Table 8). Storage time increased the FFA values for both mackerel and salmon (Table 9). The pattern for the peroxide values (PVs) was largely similar to that for FFAs in that they increased with time (Table 9) for both mackerel and salmon. The levels of FFAs and PVs found in these tests were low and did not influence taste panel response.

Fish colour, texture

Whiting and salmon portions in 100% CO_2 were yellower than those from the other treatments and whiting and mackerel fillets became progressively more yellow over time (Hunter b values; Tables 8 and 9). Springiness and Kramer shear values for whiting fillets were not affected by MAP. However, day 3 and day 5 whiting fillets had lower shear values than day 0 (fresh). This could be due in part to the soft muscle structure of this species and in part to immersion of the fillets in the gravity drip (up to 16% in 100% CO_2 pack) over the 3 to 5 day chilled storage period. Mackerel portions were toughest in 100% CO_2 while 60% N_2 / 40% CO_2 and 100% CO_2 gave salmon portions with the highest shear values. The toughening of fish during freezing and cold storage is well documented (IIR, 1986; Gormley *et al.*, 2002) and the freezing component of the freeze-chill process also leads to increased drip (O'Leary *et al.*, 2000).

In-pack drip

Portions packed in 100% CO_2 had higher gravity drip (GD) than those in 30% O_2 / 40% CO_2 / 30% N_2 (whiting) and 60% N_2 / 40% CO_2 (mackerel; salmon) while portions in air had lower values still (Table 8). Storage time at

2 to 4^{0}C had no effect on the GD values for the three species. However, whiting, mackerel and salmon showed an increase in centrifugal drip (CD) with time of storage at 2-4^{0}C (Table 9) while atmosphere had a variable effect on CD values (Table 8). The drip values were low to intermediate overall but were high in the CO_2 atmosphere for both whiting and mackerel and would require controlling via drip pads.

GMP, HACCP, tempering

The application of GMP, HACCP and controlled tempering is essential for freeze-chilled MAP packs and was discussed in Part 1 of this report together with labelling requirements.

Table 10: Correlation coefficients[a] for selected quality parameters of whiting, mackerel and salmon portions after modified atmosphere packaging with freeze-chilling

	Species		
	Whiting	Mackerel	Salmon
TVBN[b] x ACC[c]	- 0.93	-0.71	-0.91
TVBN[b] x TVC[d]	+0.85	+0.45	+0.48
TVC[b] x ACC[c]	-0.86	-0.44	-0.63

[a] Based on 20 data points

[b] Total volatile base nitrogen (mg N per 100g flesh)

[c] Sensory acceptability: 0 (unacceptable); 6 (very acceptable)

[d] Total viable count ($\log_{10}$ cfu/g)

CONCLUSIONS

- The MAP packs performed well during freeze-chilling but the packs with 100% CO_2 showed slight implosion after 5 days storage at 3-4^{0}C.
- The combination of MAP with freeze-chilling gave shelf-lives of 5 days (whiting and mackerel) and 7 days (salmon) which is 1-2 days longer than freeze-chilled portions packaged in air.
- Drip losses in freeze-chilled packs with 100% CO_2 were high for whiting and mackerel and would require drip pads.
- The test packs conformed with EC and National Guidelines in that TVBN (total volatile base nitrogen) values were below 35 mg N/100g (Council Regulation No. 95/149/EC) and TVCs (total viable counts) less than log 6 cfu/g (one million viable cells per gram).

RECOMMENDATIONS TO INDUSTRY

Freeze-chilling is a suitable technology for raw fish fillets and confers logistic advantages during storage, distribution and retailing. It provides a viable alternative to the wet fish counter.

Raw material freshness is of paramount importance and fillet freezing at sea or on-farm would be highly beneficial in delivering an extended shelf-life in the chilled phase of the freeze-chill process. It is probable that a shelf-life of at least 8-10 days at 4^{0}C could be obtained with fish frozen at sea compared with 2-3 days (or 5-7 days with MAP) found in the current trials for whiting, mackerel, and salmon portions.

Modified atmosphere packaging increased the chilled shelf-life of freeze-chilled fish fillets by about 50%. However, some packs with 100% CO_2 imploded slightly during the freezing phase of the process and gave an increased drip in the chilled phase, especially for whiting fillets.

Good manufacturing practice and HACCP are essential when freeze-chilling fish. Careful attention must be focused on the tempering (thawing) step.

Transferring from the supermarket deep freeze storage room to the chill cabinet in the evening time is recommended resulting in a well-tempered chilled product for retailing the following morning.

The freeze-chilled fillets/portions should be labelled 'previously frozen' for the information of the consumer. Best-before-date should be affixed to the packs at the start of tempering.

Industry facts sheets on freeze-chilling are available from the authors at The National Food Centre.

OTHER R&D ON FREEZE-CHILLING AT THE NATIONAL FOOD CENTRE

Freeze-chilling tests have been conducted at The National Food Centre on a range of ready-meal components including mashed potato, steamed green beans, broccoli and carrots, steamed salmon, lasagne, tagliatelle and cereal-based products. The results indicated that chilling and freeze-chilling gave similar sensory acceptability scores in the product range and the process is being used by a number of food companies. Special attention was focused on tempering/thawing in a state-of-the-art controlled tempering unit capable of thawing 5000 ready-meals (6 per outer) in 12 hours. An overview of freeze-chilling R&D has been published (Gormley *et al.*, 2003).

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