

Atlantic salmon (*Salmo salar*, L) as raw material for the smoking industry. II: Effect of different smoking methods on losses of nutrients and on the oxidation of lipids

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Abstract

The changes in total fat content, fatty acid composition, tocopherol, ascorbic acid, pH and oxidation were analysed in Atlantic salmon (*Salmo salar*, L.) in response to either cold smoking (20 or 30 °C) or electrostatic smoking. Both fresh and frozen fillets were dry-salted before smoking. The fish smoked were the lean ocean-ranched salmon caught off Iceland in June 1998 and farmed Norwegian salmon, slaughtered in either November 1998 or April 1999, differing in fresh fillet fat content from 84 to 169 g·kg⁻¹ wet weight. The fresh material used in smoking significantly affected the smoking loss of nutritive components in the fillets. The leaner the fish the higher percentile loss in fillet fat. Ascorbic acid decreased about 80 percent from the fresh value, independent of smoking temperature (20 or 30 °C). The fish that were dry-salted and electrostatically smoked only lost about 10 percent of the fresh ascorbic acid content, independent of the type of raw material used, indicating a conserving effect on ascorbic acid by the electrostatic process. Also, the electrostatically smoked fish showed a smaller drop in fillet pH than cold-smoked fillets, while tocopherol was little affected by the smoking methods tested. © 2002 Elsevier Science Ltd. All rights reserved.

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1. Introduction

Traditionally, the frozen, wild coho salmon was used in the European smoking industry but, within recent decades, farmed Atlantic salmon (*Salmo salar*, L.) has replaced most of the wild salmon (Hafsteinsson, Vallett, Torrissen, Lie, Thomassen, & Boderias, 1998). Wild salmon grow more slowly and are leaner than farmed salmon, as both the feed composition and the feeding regimes in aquaculture have been improved with the aim of maximal growth. Intensive fish production may affect the composition of the fish as well as its suitability for processing. The chemical composition of salmon is affected by both nutritional stage (Bell, McEvoy, Webster, McGhee, Millar, & Sargent, 1998; Hemre & Sandnes, 1999; Lie, Sandvin, & Waagbø, 1993; Shee-

han, O'Connor, Sheehy, Buckley, & FitzGerald, 1996; Sigurgisladottir, Parrish, Ackman, & Lall, 1994; Sigurgisladottir, Torrissen, Lie, Thomassen, & Hafsteinsson, 1997) and body weight of the fish (Fauconneau, Andre, Chmaitilly, LeBail, Krieg, & Kaushik, 1997; Storbakken, Hung, Calvert, & Plisetskaya, 1991). Moreover, farmed salmon has become fatter during the last decade due to the use of high energy feeds (Einen & Roem, 1997; Hemre & Sandnes, 1999; Lie, Waagbø, & Sandnes, 1988). Complaints about higher losses of fat during processing, high frequency of gaping, low and uneven colour distribution have increased concomitantly with the increased fillet fat content. Therefore, it is important to gain more knowledge about interactions between the fresh fat content and other chemical changes during the processing step, both in farmed salmon and in the slower-growing wild salmon.

The present study examines the effects of freezing, dry-salting and different cold-smoking methods upon changes in fat content, fatty acid composition, pH and oxidation, as well as protection against oxidation, by

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consumption of the natural anti-oxidative vitamins, tocopherol and ascorbic acid, during the processing step. The traditional cold-smoking was compared to an electrostatic smoking method. The fish tested were lean ocean-ranched salmon, representing the wild fish and farmed salmon, slaughtered during the spring or during the autumn, thus differing in fresh fat content because of seasonal variation.

2. Materials and methods

2.1. Fish material

Ocean-ranched Atlantic salmon (2.6 ± 0.3 kg mean round weight) were caught off Iceland in June 1998. Farmed salmon were supplied from commercial farms in the northern part of Norway and slaughtered at the beginning of November 1998 (4.0 ± 0.2 kg mean round weight) or from the south-west coast of Norway, slaughtered in late April 1999 (3.7 ± 0.5 kg mean round weight).

The ocean-ranched salmon were slaughtered in a commercial slaughter plant in Iceland. The Norwegian farmed salmon were fed commercial feeds (Biomar: Bio optimal™). Salmon slaughtered in November were starved for 14 days at a seawater temperature between 9.6 and 11.0 °C, while those slaughtered in April were starved for 30 days at a seawater temperature of 6 °C prior to slaughter. The temperatures of fillets after slaughtering were 2.5 and 6 °C in fish slaughtered in April and November, respectively. After slaughtering, the fish were filleted, individually tagged and the left fillet was analysed when fresh, while the right fillet was processed and analysed after smoking. The following processing methods were used: Cold smoking at 20 or 30 °C, or electrostatic smoking at 20 °C. Before smoking, the fresh fillets were either dry salted directly or frozen for one month before dry-salting. Freezing of the fillets was done with cryogenic equipment for 1 h (CO₂ at -60 °C), and stored at -20 °C for 30 days. Prior to salting, the frozen fillets were thawed overnight in air at 4 °C. Fillets were dry-salted by hand, using refined salt, and left at 12 °C for 6 h, then rapidly rinsed in fresh water (15 °C) and stored at 2 °C until smoking, which was done in a tunnel with an air velocity of 2 m·s⁻¹. The smoking time was four hours and chips of beech were used. The electrostatically smoked fillets were smoked for 20 min by use of 8×8 cm oak beams (Cardinal et al., 2001).

Following processing, the Norwegian quality cut (NQC) of 15 trimmed, fresh and smoked samples from each treatment group were analysed and the changes in tocopherol and ascorbic acid, in total fat content and in fatty acid composition, as well as the pH and the oxidation status during processing, were studied.

2.2. Chemical methods

Ascorbic acid was analysed in fresh and smoked salmon by reverse phase HPLC, using electrochemical detection. 0.5 g fillet was homogenised in 5% metaphosphoric acid. Ascorbic acid released from the homogenates was stabilised by addition of 0.54% EDTA. Ascorbic acid was separated by use of an ODS Hypersil (C18, 5 µm, 100×4.6 mm) column (Hewlett Packard), equipped with a similar quality 20×4 mm guard column. Ascorbic acid was detected at 0.6 V by an electrochemical detector (Hewlett Packard). A standard curve of ascorbic acid was used for quantification (Sandnes, Hansen, Killie, & Waagbø, 1990).

Tocopherol was analysed in both fresh and smoked fillets by normal phase HPLC with fluorescence detection (excitation: 289 nm, emission: 331 nm). 0.5 g sample was homogenised and saponified in 4 ml ethanol, 0.5 ml saturated EDTA and 0.5 ml 20% KOH and extracted in 2×2 ml hexane. Ascorbic acid and pyrogallol were added before saponification to prevent oxidation during the process (Lie, Sandvin, & Waagbø, 1994).

Total fat content in fresh and smoked fillets was determined gravimetrically, after extraction with ethyl acetate (Losnegård, Bøe, & Larsen, 1979).

In both fresh and smoked fillets, lipids were extracted in chloroform:methanol (2:1, v/v) and fatty acid composition of total lipids was obtained by saponification of the extracted lipids, esterification by 12% BF₃ in methanol, and the methyl esters separated by capillary gas chromatography (50 m, 0.3 mm id., CP-sil 88 fused silica capillary column; Chromopack). Peaks were identified by reference to a standard mixture of methyl esters (Rønnestad, Finn, Lein, & Lie, 1995).

The value for thiobarbituric reactive substances (TBARS) was determined in both fresh and smoked salmon to evaluate the oxidation stability, during processing, of the salmon fillets. Samples were homogenised under nitrogen with chloroform:methanol (2:1, v/v). Two parts of water were then added to make a two-phase system, and an aliquot of the methanol-water phase, containing the short chain aldehydes, was heated in the presence of excess thiobarbituric acid in trichloroacetic acid. The complex created was measured spectrophotometrically at 532 nm using malondialdehyde as standard (Schmedes & Hølmer, 1989).

2.3. Statistical methods

Fresh fat content was correlated with fresh fatty acids, tocopherol, ascorbic acid, pH and TBARS, while mean smoked values in fillet chemical composition, through the processing steps, were compared by ANOVA. If significant differences ($P < 0.05$) between means were obtained, Tukey's honest significant test was used to differentiate between means. Statistics were

performed using CSS Statistica (Ver. 5.5: 99, Statsoft Inc, 1991, Tulsa, USA). The mean chemical values, as well as the respective percentual changes through processing, were also explored by principal component analysis, PCA (Martens & Naes, 1989). This was done to visualise and thus improve the interpretation of the relative changes in chemical composition observed in the different fillets. PCA was carried out by the Sirius (Ver. 6.5, Pattern Recognition Systems Ltd., Bergen, Norway) software (Kvalheim & Karstang, 1987).

3. Results and discussion

The ocean-ranched fish were leaner than the farmed salmon, and the fish slaughtered in November contained more fat than those slaughtered in April. Generally, the farmed salmon contained 1.5 to 2 times as much total fat as the ocean-ranched, depending on season, as previously reported (Bell et al., 1998; Espe, Nortvedt, Lie, & Hafsteinsson, in press). Freezing the fish for one month at -60°C prior to dry-salting and smoking, had no effect on fat loss or any of the other parameters analysed within the frozen, thawed fillets. The mean fresh fat values in the fillets were correlated with the highly valuable fatty acids EPA (eicosapentaenoic acid 20:5n-3) and DHA (docosahexaenoic acid, 22:6n-3), the n-3/n-6 ratio, the tocopherol and the ascorbic acid content and the pH (Table 1). No correlations between fresh fat content and the sum of either, saturated, monounsaturated or polyunsaturated fatty acids, in the fresh fillets were found. Neither was there any correlation between fresh fat content and oxidation measured as TBARS. The leaner the fish the higher the ratio of n-3 to n-6 fatty acids ($r = -0.40$). EPA and DHA, showed opposite trends to one another in that the leaner the fillet, the less EPA ($r = 0.62$) and the more DHA

($r = -0.64$). These differences in fresh fatty acid composition between “wild” and farmed salmon accord with results reported previously (Cronin, Powell, & Gormley, 1991; Farmer, McConell, & Graham, 1997). The vitamins, having anti-oxidative properties, also seemed to depend on the fresh fat contents, as tocopherols were higher ($r = 0.73$) and ascorbic acid lower ($r = -0.39$) in the fattier fillets. Also pH was highly dependent on fresh fat content, due to the higher muscle pH found in the leaner fish ($r = -0.76$). The differences observed are probably due to different feed compositions as well as deposition of lipids within the fillets. Probably this is also partly due to the fact that the ocean-ranched fish are forced to swim and catch their own feed and never feed as intensively as do farmed fish on the high energy diets (Jónsson, Pálmadóttir, & Kristbergsson, 1997).

The chemical composition (wet weight) of the smoked fillets generally reflected the values of the fresh fillets (Table 2), but fat content decreased during processing, and generally the fillets smoked at the higher temperature contained more fat, although the differences were insignificant. Espe et al. (in press) previously reported that smoked lipid content reflected the fresh lipid content. The smoking method used, however, showed a significant effect on the smoked fat content, as electrostatically smoked fillets lost more fat than the traditionally cold-smoked fillets. The nutritionally valuable fatty acids, EPA and DHA, also reflected the different raw materials used, but EPA showed interaction between the smoking methods and raw material used, as EPA was higher in the fattiest and leanest fish when frozen prior to processing (Table 2). This also resulted in a higher ratio of n-3 to n-6 fatty acids within those being frozen. These differences are due partly to rupture of cells and differences in dry material among those fillets being frozen.

Oxidation status (TBARS) and the levels of tocopherol and ascorbic acid were affected by both the raw

Table 1

Total fat content (g kg^{-1} wet weight) values in fresh salmon are means \pm S.E.M. Correlations of fatty acid composition (sum of saturated, SFA, mono unsaturated, MUFA, and poly unsaturated, PUFA), n-3/n-6 fatty acid ratio, EPA, DHA, tocopherol, ascorbic acid, pH and oxidation (TBARS) to the fresh fat content in the raw materials are represented by the respective Pearson r correlation coefficients and the P -values

Raw materials	Ocean-ranched June	Farmed November	Farmed April
Fresh fat content	84.0 \pm 2.9	169.3 \pm 2.9	123.2 \pm 5.3
Correlation with fresh fat content			
	Regression, (r)		P -value
Sum of SFA	-0.36		0.051
Sum of MUFA	-0.14		0.50
Sum of PUFA	0.27		0.14
Ratio of n-3/n-6	-0.40		0.03
EPA	0.62		0.0005
DHA	-0.64		0.0002
α -Tocopherol	0.73		0.000001
Ascorbic acid	-0.39		0.04
PH	-0.76		0.000001
TBARS	-0.03		0.84

material used and by the processing method chosen. Oxidation was lowest in those fillets being smoked by the electrostatic method, and highest in the frozen fillets at the higher smoking temperature (Table 2). Tocopherol was best conserved in those fillets smoked at the higher temperature when fresh fat was low (ocean-ranched), but more so in those fillets being frozen when fresh fat content was high (farmed and slaughtered in November). Regarding the method of smoking, no clear effect on tocopherol content was observed between the cold-smoking and the electrostatic method. Ascorbic acid, on the other hand decreased by about 80% of the fresh value during the processing steps, except during the electrostatic smoking, where only a 10–30% decrease occurred. The ocean-ranched fish had higher contents of ascorbic acid in the fresh fillets and also showed the highest loss (22.5–27.4 mgkg⁻¹ or 69–84%). Furthermore, a higher smoking temperature caused a higher loss of ascorbic acid in ocean-ranched salmon and in farmed salmon slaughtered in November. In the farmed salmon, slaughtered in April, the smoking temperature did not affect the content of ascorbic acid (Table 2). The pH in the fillets generally decreased through the processing steps and the highest temperature caused the highest drop in pH, except in the fish slaughtered in April. The fish being electrostatically smoked showed the least decrease in pH, independent of which raw material was used (Table 2).

A three-component PCA model of the mean fresh values of total fat, TBARS, pH, tocopherol and ascorbic acid and their respective changes under processing

explained 84.1% of the variance in the data matrix (Fig. 1). A high loading value in the plot for variables coding for relative changes reflects a large change in the actual parameter during the processing steps, and vice versa. The ocean-ranched fish were separated from the farmed fish along PC1, in showing higher relative loss (13–48%) of total fat during processing, while both the farmed fish groups were observed close to the fresh fat variable, confirming their higher fat contents (Fig. 1), as reported by Espe et al. (in press). This also accords with the higher total loss in fat from round weight to smoked fillet in ocean-ranched fish than farmed ones, as reported by Cardinal et al. (2001). There was, however, a marked positive effect of electrostatic smoking, which resulted in low loss of ascorbic acid through the process, visualised along PC2 by the negative correlation between the variable ‘change in vitamin C’ and all the electrostatic-smoked groups (Fig. 1). This difference is probably due to the much shorter period of smoking in the electrostatically smoked fish than the traditionally cold-smoked fish. The decrease in pH and loss of vitamin C were closely correlated along PC 2, and demonstrated a higher loss of ascorbic acid in the frozen than in the unfrozen, dry-salted groups. The ‘change in vitamin E’ variable was more closely correlated with the low temperature (20 °C) groups than with the respective high temperature (30 °C) groups, showing that the tocopherol was best retained in the fish smoked at the higher temperature (30 °C).

The raw material thus seems to have a stronger effect upon losses during processing than does the processing

Table 2

Mean smoked values of total fat content, tocopherol, ascorbic acid, TBARS, pH, n-3/n-6 fatty acid ratio, EPA and DHA in ocean-ranched and farmed Atlantic salmon slaughtered in either November or April after being frozen (Frz) and dry salted (Ds) or only dry-salted, followed by cold-smoking at 20 or 30 °C, or electrostatically smoked at 20 °C (Elect.), respectively^a

Raw material/ Smoking groups	Total fat (gkg ⁻¹)	Tocopherol (mgkg ⁻¹)	Ascorbic Acid (mgkg ⁻¹)	TBARS (μmolkg ⁻¹)	pH	Ratio of n-3/n-6 fatty acids	EPA (g100 g ⁻¹ total FA)	DHA (g100 g ⁻¹ total FA)
<i>Ocean ranched/June</i>								
FrzDs20	48±5.7Ca	10.8±1.07d	15.2±1.53b	7.5±1.07ab	6.2±0.01c	11.9±0.75Aab	5.6±0.34cd	13.9±1.22A
FrzDs30	74±6.0Ca	12.8±1.08cd	7.8±0.74de	8.6±0.68a	5.9±0.02g	13.0±0.76Aa	6.4±0.11bc	14.3±1.06A
Ds20	52±6.1Ca	11.3±0.71d	10.3±1.86bcd	7.6±0.76ab	6.3±0.02b	12.0±0.37Ab	5.1±0.27d	13.4±0.58A
Ds30	61±5.2Ca	13.0±0.92cd	8.2±0.86de	6.0±0.58bc	6.1±0.02d	12.1±0.41Ab	5.7±0.35cd	13.8±0.38A
Elect.	44±3.2Cb	12.6±0.90d	29.2±2.13a	4.6±0.71c	6.3±0.01b	11.3±0.36Ab	4.9±0.22d	12.9±0.46A
<i>Farmed/November</i>								
FrzDs20	143±8.2Aa	27.7±1.07a	7.0±0.89def	7.7±0.90ab	6.0±0.01de	7.8±0.06Bab	7.6±0.11a	10.1±0.12C
FrzDs30	148±7.3Aa	28.0±1.72a	3.6±0.40ef	11.8±0.47a	5.8±0.01h	7.8±0.13Ba	7.4±0.18a	10.0±0.16C
Ds20	143±7.1Aa	24.5±0.98ab	9.0±0.78cd	6.0±0.76ab	6.1±0.01d	6.5±0.24Bb	6.9±0.08ab	9.7±0.34C
Ds30	162±7.3Aa	24.6±1.36ab	5.4±0.51def	7.0±0.45bc	6.0±0.01fg	6.4±0.27Bb	7.0±0.16ab	9.4±0.27C
Elect.	124±8.6Ab	22.4±0.67ab	16.0±0.84b	5.1±0.49c	6.2±0.01bc	6.6±0.22Bb	7.1±0.21ab	9.6±0.23C
<i>April</i>								
FrzDs20	128±12.3Ba	23.0±1.16ab	2.2±0.20f	8.5±1.43ab	6.0±0.01ef	4.2±0.20Cab	7.5±0.17a	11.0±0.25B
FrzDs30	113±11.3Ba	19.2±1.00bc	3.6±0.68ef	9.1±0.90a	6.2±0.01c	4.7±0.16Ca	7.8±0.15a	11.7±0.20B
Ds20	137±5.0Ba	22.3±0.81ab	3.4±0.25ef	9.3±1.19ab	6.0±0.01ef	3.9±0.21Cb	7.6±0.06a	10.8±0.11B
Ds30	127±8.1Ba	19.9±0.67b	3.6±0.25ef	7.6±1.10bc	6.2±0.01c	3.8±0.24Cb	7.7±0.10a	11.0±0.07B
Elect.	101±9.3Bb	19.7±0.84bc	13.8±1.39bc	6.4±2.33c	6.4±0.02a	3.9±0.28Cb	7.6±0.08a	11.2±0.19B

^a Values (means±S.E.M.) followed by different uppercase letters differ in raw materials, whereas different lowercase letters indicate significant effects from variable processing methods and bold letters indicate interactions between smoking method and raw materials ($P < 0.05$).

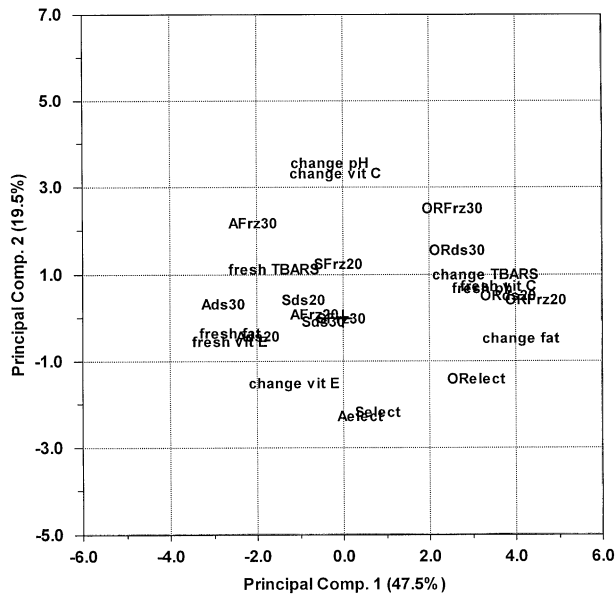


Fig. 1. Biplot from PCA analysis of the ocean-ranched (OR) and farmed salmon (slaughtered in either A, the autumn, or S, the spring), and their respective chemical fresh values and relative changes during freezing and dry-salting (Frz) or dry-salting only (ds), followed by cold-smoking at either 20 or 30 °C, or electrostatic smoking (elect). Explained variance along each principal component is given in parentheses.

method, with the exception of the low loss of ascorbic acid and low pH drop in those fillets being electrostatically smoked. The amount of naturally occurring anti-oxidative vitamins in the raw materials tested was enough to prevent oxidation in any of the smoked salmon in the present experiment.

4. Conclusion

The fresh material used in smoking significantly affected the smoking loss of nutritive components in the fillets. The leaner the fish the higher percentile loss in fillet fat. Ascorbic acid decreased by about 80% from the fresh value, independent of the smoking temperature of 20 or 30 °C. The fish that were dry-salted and smoked electrostatically lost only about 10% of the fresh ascorbic acid value, independent of the type of raw material used. Electrostatically smoked fillets, however, lost more lipids and were less oxidised than the traditional cold-smoked salmon.

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