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Effect of freeze storage on the volatiles of butter

By **Magda A. Abdel-Mageed** and **Hoda H.M. Fadel**
Dept. of Flavour and Aroma. National Research Centre, Cairo Egypt.

RESUMEN

Efecto del almacenamiento en estado congelado sobre los volátiles de mantequilla.

Se han estudiado los cambios en los componentes volátiles de tres muestras de mantequilla durante su almacenamiento en estado congelado por siete meses y medio.
El almacenamiento tuvo un efecto significativo en los componentes carbonilos y lactonas analizadas. El aumento en el rendimiento de los carbonilos causado por la peroxidación de ácidos grasos insaturados se tomó como indicador en la deterioración de las muestras de mantequilla durante el almacenamiento.

PALABRAS-CLAVE: Almacenamiento en estado congelado — Componentes volátiles — Mantequilla.

SUMMARY

Effect of freeze storage on the volatiles of butter

The changes in the volatile components of three butter samples during freeze storage for 7.5 months were studied.
The storage had a significant effect on the carbonyl and lactone components analyzed. The increase in the yield of the carbonyls caused by peroxidation of unsaturated fatty acids was taken as indicator for the deterioration of the butter samples during storage.

KEY-WORDS: Butter — Freeze storage — Volatile components.

1. INTRODUCTION

Butter is undoubtedly one of the most important foods of man. In addition to its dietary value, it has a pleasant aroma.

The identification of a total of 233 compounds in the volatile fractions of butter and butter oil has been reported (1). Stark and his group (2-5) detected the compounds contributed to the flavour of butter oil. They concluded that decanoic acid, lauric acid, δ-octalactone, δ-decalactone, indol and skatole played an important role in the flavour of butter oil.

On the basis of the actual concentration to threshold ratios, butyric and hexanoic acid make no flavour contribution to butter investigated by Urbach *et al* (6). On the other hand Mick *et al* (7) suggested that, diacetyl, hexanoic acid and δ-decalactone determined the aroma, as their concentrations exceeded their sensory thresholds.

The effect of storage on the butter flavour were studied (8). Badings (9) found a complex mixtures of carbonyl compounds in butter after the cold storage. More recently Widder *et al.* (10) studied the change in the flavour of butter oil during storage at room temperature for 43 days. They found that among the volatiles formed by lipid peroxidation 1-octene-3-one, 2-nonenal and 1,5-octadiene-3-one showed the greatest increase during storage.

The object of the present work was to detect the effect of freeze storage for 7.5 months on the volatiles of three different butter samples.

2 . MATERIALS AND METHODS

2.1. Samples

The samples used were local fresh cows butter (C) and local fresh buffaloe's butter (B). Kindly supplied by the Animal Production Research Statiroduct of the spring season 1993. The imported butter (I) purchased from local market product of E.E.C.

The three butter samples were stored in deep freeze at -18°C for 7.5 months. Samples of each butter were taken at various time periods (1.5,3,4.5,6 and 7.5 months). A sample of each butter before storage (0.0 month) was taken as control.

2.2. Isolation of volatiles

The volatiles were separated from each butter sample (200 g) by using the techniques of vacuum steam distillation according to Sike and Lindsay (11). Volatiles were recovered by diethyl ether, dried over anhydrous Na_2SO_4 and concentrated on a Vigreux column (60x1 cm).

2.3. Fatty acid composition

Butter fat was separated from each butter sample by melting, decanting and filtering. The fatty acid methyl ester of each sample was prepared (12).

2.4. Gas Liquid Chromatographic (GLC) analysis

Quantitative analysis of the volatiles and the fatty acid methyl esters of each sample were performed by GLC. The separated components were identified by comparing their retention times with those of authentic samples which were purchased from Aldrich chemical Co.,.

A varian 3700 gas chromatograph equipped with flame ionization detector and computing integrator was used under the following conditions:

9mx3mm ID Stainless-steel column, packed with 15% polyethylene glycol adipate (PEGA) on 60-80 mesh chromosorb W; carrier gas (N_2) flow rate 30 ml/min; injector temperature, 220°C; detector temperature 250°C, oven temperature, (programming) 70-190°C (4°C/ min).

3. RESULTS AND DISCUSSION

The principal volatile components of the three investigated samples, cow's butter (C), buffaloe's butter (B) and imported butter (I) stored in the deep freeze for 0.0 (control), 1.5,3,4.5,6 and 7.5 months are reported in Table I.

Table I

Changes in the volatile components of three butter samples during storage in deep-freeze for 7.5 months

Peak No.	t_R	Local Cow's butter (C)						Local Buffaloe's butter (B)						Imported butter (I)						Components
		0.0 control	1.5 month	3.0 month	4.5 month	6.0 month	7.5 month	0.0 control	1.5 month	3.0 month	4.5 month	6.0 month	7.5 month	0.0 control	1.5 month	3.0 month	4.5 month	6.0 month	7.5 month	
1	2.67	7.43*	3.55	3.43	0.93	1.61	0.90	3.33	1.91	1.10	0.91	0.06	5.61	6.27	3.05	1.00	0.50	0.46	0.21	Diacetyl
2	3.14	0.04	0.20	3.86	0.30	5.00	T	T	0.03	0.69	5.08	0.80	1.07	0.09	0.16	0.05	1.18	1.50	2.00	1-Hexene-3-one
3	3.30	0.09	0.11	2.01	6.00	10.32	14.19	T	0.04	0.62	2.25	4.60	5.09	0.53	1.00	0.88	1.09	10.21	14.51	Pentanal
4	3.49	0.27	35.41	34.66	28.79	23.40	6.01	-	3.55	5.07	6.05	10.35	5.06	1.11	2.18	1.98	2.08	26.50	10.99	2-Pentenal
5	4.56	T	0.08	0.12	3.01	18.09	20.25	0.08	2.05	0.10	5.09	15.89	17.04	1.02	0.65	1.77	5.21	40.00	47.41	Hexanal
6	5.43	T	8.00	12.39	6.99	2.15	1.41	0.02	25.17	3.03	6.01	15.01	0.27	2.38	3.09	1.25	1.62	0.71	0.12	2-Hexenal
7	5.48	T	7.40	4.11	4.67	3.60	4.62	-	6.46	0.28	0.51	8.00	0.13	1.11	1.06	0.15	0.20	1.91	0.88	Hexenol
8	5.50	T	T	2.16	2.72	2.68	2.56	0.14	0.03	0.15	2.00	1.11	0.05	2.08	0.19	0.18	0.90	1.09	0.95	Heptanone
9	5.52	T	T	2.87	2.15	2.68	1.09	-	3.11	5.55	0.19	8.55	0.16	0.18	11.05	8.00	9.04	2.09	T	Heptanal
10	5.55	3.52	2.18	7.72	1.55	3.38	4.11	2.00	2.02	9.12	2.14	4.50	0.74	2.06	2.26	1.92	2.23	1.80	1.02	Butyric acid
11	6.68	-	T	0.17	1.50	1.90	2.20	2.22	0.47	0.20	1.02	2.11	1.32	0.20	5.53	10.01	9.80	1.23	1.05	Heptenal
12	7.18	-	-	-	-	-	-	T	-	-	-	-	-	-	-	-	-	-	-	———
13	8.53	-	T	0.16	0.12	T	0.11	0.10	0.05	0.28	0.02	0.02	-	0.11	10.89	0.05	0.08	0.07	0.03	———
14	9.45	-	T	0.79	0.43	0.02	0.08	0.05	0.07	0.51	0.60	0.48	0.32	0.32	0.02	0.04	1.73	0.01	0.10	2-Octenal
15	10.88	3.42	3.91	2.66	1.00	2.05	1.08	T	3.46	1.45	3.46	3.00	8.62	3.04	4.37	2.29	1.00	2.01	1.07	Hexanoic acid
16	11.97	-	0.03	2.05	1.55	0.01	-	2.54	1.04	1.95	1.02	T	0.01	0.13	3.16	2.01	0.08	0.17	0.04	2,4-Heptadienal
17	12.02	0.11	-	0.68	T	0.01	-	-	0.42	1.23	-	-	0.31	0.15	1.09	0.04	0.09	-	-	———
18	12.05	1.66	-	0.40	T	-	-	1.00	0.95	1.81	-	-	0.22	0.45	-	-	T	-	-	———
19	12.13	0.48	0.51	1.01	1.76	2.11	2.99	0.02	0.04	0.37	0.31	0.39	1.16	1.83	0.72	0.64	1.08	1.50	1.04	1-Octene-3- one
20	12.21	0.51	0.50	1.88	5.00	0.49	1.01	0.32	0.12	0.34	-	0.04	0.22	2.48	0.21	0.32	0.23	-	-	———
21	13.97	0.07	0.01	0.01	0.09	T	T	-	0.79	0.27	0.20	0.04	0.16	0.16	0.19	0.24	0.07	0.03	0.17	Nonanal
22	15.35	0.02	0.03	0.37	0.09	0.13	0.16	0.05	0.13	0.35	0.60	0.53	0.24	3.19	0.05	0.16	T	0.21	0.27	2-Nonenal
23	18.15	-	-	0.21	2.60	0.11	0.19	-	1.45	0.85	1.06	0.03	0.74	0.61	0.56	0.81	0.34	0.02	0.18	2,4-Nonadienal
24	20.07	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	———
25	20.53	0.90	-	0.09	0.03	0.20	0.45	0.45	0.26	0.93	0.58	1.15	0.78	37.55	0.48	0.16	0.39	0.17	0.43	———
26	21.42	0.65	0.01	0.45	0.07	0.22	0.67	0.28	0.29	2.18	1.16	0.75	1.28	0.26	0.01	0.21	0.01	0.46	0.42	2,4-Decadienal
27	23.60	-	-	0.11	-	-	0.86	-	0.45	0.49	0.32	0.71	1.28	0.24	1.17	0.04	0.01	0.26	0.27	———
28	24.05	1.17	0.07	0.16	0.01	-	0.67	2.85	0.16	0.63	1.09	0.25	0.28	0.40	0.16	0.06	0.52	0.16	0.06	Undecanone
29	25.37	0.31	-	0.02	1.02	0.67	-	1.22	1.35	9.26	0.78	0.03	0.58	2.38	0.34	0.39	0.16	0.28	0.17	———
30	26.36	0.03	0.05	0.16	5.47	0.95	0.90	0.26	0.23	12.84	1.52	0.22	0.51	0.17	0.22	0.07	0.16	0.60	0.27	Undecadienal
31	28.20	0.44	0.03	0.13	-	-	0.09	0.40	0.18	1.67	0.04	0.59	0.38	0.15	0.26		0.33	0.16	0.03	———
32	29.96	1.03	0.03	0.22	0.11	0.89	0.24	0.35	0.17	0.64	1.98	-	0.22	1.41	0.06	0.03	0.47	0.12	0.01	Dodecanone
33	31.15	6.14	0.08	0.39	0.50	1.62	0.24	14.60	0.53	0.83	0.94	0.49	0.20	1.52	0.44	0.17	38.18	0.07	0.09	δ-Octalactone
34	33.60	1.09	0.07	0.27	0.30	1.23	0.14	4.00	0.28	2.95	6.19	0.23	0.02	3.27	0.52	43.04	1.59	0.61	0.06	Octanoic acid
35	35.46	2.00	-	0.09	-	0.04	0.16	-	5.29	5.26	4.01	-	0.01	-	0.13	0.26	1.52	0.01	0.02	———
36	36.30	14.22	2.00	0.03	0.30	0.10	0.86	1.48	0.84	2.25	8.29	0.18	0.65	3.97	1.17	0.20	1.91	0.13	0.49	δ-Decalactone
37	38.20	2.40	3.64	0.46	0.10	0.32	T	2.00	0.86	0.25	6.75	0.92	0.03	1.84	0.28	1.85	0.18	0.06	0.08	———
38	40.81	0.79	T	0.10	3.26	2.59	1.89	1.00	0.33	0.25	1.42	0.03	0.71	T	0.90	0.63	2.38	0.09	T	Dodecadienal
39	41.50	38.30	8.26	1.29	5.17	4.17	24.44	30.30	2.00	7.78	5.38	9.27	2.99	10.20	1.77	3.24	1.91	1.83	11.69	δ-Dodecalactone
40	44.30	0.11	-	0.64	0.17	1.65	3.11	10.06	1.87	0.02	0.52	1.97	28.21	2.33	0.10	3.44	1.69	0.12	0.46	γ-Dodecalactone
41	46.60	1.11	3.39	0.52	4.55	3.48	1.07	2.07	2.85	2.16	3.40	3.82	2.09	1.43	3.43	2.60	2.73	0.23	0.84	Decanoic acid

Table I. (Contd.)

Peak No.	t_R	Local Cow's butter (C) 0.0 control	1.5 month	3.0 month	4.5 month	6.0 month	7.5 month	Local Buffaloe's butter (B) 0.0 control	1.5 month	3.0 month	4.5 month	6.0 month	7.5 month	Imported butter (I) 0.0 control	1.5 month	3.0 month	4.5 month	6.0 month	7.5 month	Components
42	47.17	0.31	3.00	4.16	1.47	0.14	-	0.70	2.32	2.21	1.40	0.28	2.19	-	0.96	2.37	1.99	0.26	0.53	———
43	48.50	-	-	0.20	1.22	0.48	0.24	1.71	6.25	3.18	2.62	1.57	0.17	-	3.55	-	2.96	0.65	T	———
44	49.60	3.55	5.49	0.26	0.39	0.39	0.58	7.39	3.51	3.65	2.51	0.53	0.80	0.90	1.69	1.62	1.38	0.53	0.42	δ-Tetradecalactone
45	50.70	0.64	1.90	0.32	0.23	0.24	0.05	2.53	1.88	2.85	1.76	0.46	2.14	0.48	1.13	1.88	0.04	0.46	0.36	Ethyl tetradecanoic acid
46	51.90	0.11	2.00	0.32	0.19	0.73	0.38	-	5.29	1.05	2.59	0.19	1.60	-	0.51	1.76	0.08	0.15	0.61	
47	53.40	3.52	3.37	0.81	1.01	0.75	-	0.06	6.37	6.30	0.22	0.71	1.23	-	0.02	0.95	0.75	0.43	0.45	
48	56.60	0.78	1.14	T	0.02	T	-	0.32	2.24	2.00	6.01	0.14	3.99	-	0.11	1.24	0.11	0.42	0.13	
49	57.70	2.88	3.12	5.08	T	T	T	0.10	0.84	1.05	-	-	0.50	-	0.07	-	0.04	0.21	0.07	
50	59.00	-	0.44	T	0.15	-	-	-	-	-	-	-	-	-	0.04	-	-	0.01	-	
51	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
52	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	

* Values expressed as area percentages

T= trace.

The carbonyl components comprised the major qualitatively contribution in the Table I. Diacetyl had considerable concentration in the three butter samples, this component plays an important role in the aroma of butter (13). 2-Hexenal, 2-pentenal and 2,4-heptadienal increased during storage in the three investigated samples, however their concentration decreased at the end of storage (7.5 months). The formation of these components consistent with the peroxidation of Linolenate (14,15). These results agreed with the observed decrease in linolenic acid, Table II.

Table II
The change in the fatty acid composition of the freeze stored butter fat

Fatty acids	Local Buffaloe's butter (B) 1	2	3	4	5	6	Local Cow's butter (C) 1	2	3	4	5	6	Imported butter (I) 1	2	3	4	5	6
C_4	4.81	4.70	4.19	3.71	2.23	2.30	4.14	4.35	3.69	2.02	2.66	3.50	3.43	3.43	3.24	3.68	3.02	3.43
C_6	3.60	3.52	2.46	3.26	1.17	1.50	2.66	3.02	2.31	1.72	2.14	2.10	2.72	2.78	2.84	3.04	2.44	2.41
C_8	1.40	1.71	1.30	1.50	1.65	1.55	1.44	2.90	2.38	0.95	1.27	1.00	1.63	1.91	1.86	1.73	1.78	1.86
C_{10}	3.15	3.33	2.66	3.11	1.57	1.63	3.27	3.24	3.11	1.30	2.97	1.50	3.85	3.95	4.42	4.34	4.69	4.63
C_{12}	3.21	2.60	2.45	4.02	2.08	2.03	3.75	3.56	1.09	4.29	3.64	3.15	4.31	4.61	4.89	4.22	4.58	4.28
C_{14}	11.71	12.75	12.55	12.90	15.97	14.06	11.64	11.40	12.33	9.68	11.14	11.51	11.78	11.65	12.25	12.00	12.93	13.01
$C_{14:1}$	1.78	1.71	2.38	2.28	1.84	1.71	2.17	2.51	1.96	2.89	1.64	1.85	1.86	1.81	2.23	2.37	1.29	1.25
C_{15}	2.02	2.11	1.97	2.75	0.99	0.81	2.76	2.61	3.59	3.65	2.35	2.13	2.15	2.07	3.33	2.63	2.34	1.77
$C_{16\,iso}$	3.09	2.91	2.94	2.43	1.34	1.53	3.71	3.92	3.42	3.29	3.25	3.25	2.62	3.16	4.16	3.36	2.18	1.51
C_{16}	25.59	26.54	26.95	27.21	32.98	34.82	26.72	27.19	39.76	30.23	30.56	30.63	26.01	26.05	27.71	27.43	29.02	30.81
$C_{16:1}$	0.50	0.50	2.66	2.02	2.42	2.11	0.64	2.35	2.79	3.11	2.12	2.31	2.05	2.11	2.85	1.21	2.16	2.52
C_{17}	2.17	0.78	1.69	1.36	1.34	1.33	2.76	1.87	2.95	3.44	1.16	1.80	3.03	2.87	2.82	1.49	1.09	0.91
$C_{17:1}$	1.38	1.11	-	-	-	-	1.72	-	-	2.5	-	-	1.95	1.85	-	T	-	-
C_{18}	13.61	13.91	13.91	12.69	13.45	14.0	10.65	10.80	11.34	10.77	11.73	11.96	11.12	11.33	11.58	12.28	12.24	12.11
$C_{18:1}$	20.40	20.51	21.16	20.14	20.79	20.62	19.94	19.17	21.19	19.04	23.22	22.80	19.02	19.31	20.84	20.22	20.24	19.50
$C_{18:2}$	1.11	1.01	0.50	0.44	0.18	-	1.36	0.60	0.59	0.45	0.05	-	1.32	1.10	0.45	-	-	-
$C_{18:3}$	0.47	0.30	0.23	0.18	-	-	0.67	0.51	0.45	0.37	T	T	1.15	0.51	0.33	-	-	-
C_{19}	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
C_{20}	T	T	-	-	-	-	T	T	-	-	-	-	-	T	-	-	-	-

T= trace.

Table III
Changes in the total concentration of volatile carbonyls and lactones of butter samples during freeze storage (values expressed as area percentages)

Chemical classes	Local Buffaloe's butter (B)						Local Cow's butter (C)						Imported butter (I)					
	0.0 control	1.5 month	3.0 month	4.5 month	6.0 month	7.5 month	0.0 control	1.5 month	3.0 month	4.5 month	6.0 month	7.5 month	0.0 control	1.5 month	3.0 month	4.5 month	6.0 month	7.5 month
Carbonyls	16.09	41.06	37.35	39.56	55.2	43.73	12.07	48.08	67.2	67.83	74.86	56.41	22.34	32.95	32.77	38.26	87.16	79.8
Lactones	48.10	7.01	14.53	31.56	12.44	32.49	58.11	15.83	1.61	6.53	7.93	30.23	18.92	4.58	52.81	46.89	2.68	13.15

Heptanal,2-octenal,2-nonenal,2,4-nonadienal and 2,4-decadienal showed the same trend, whereas pentanal and hexanal had their highest concentrations by storage for 7.5 months. All these aldehydes are peroxidation products of linoleic acid (16) which showed gradual decrease during storage Table II. Pentanal was reported to be arise from stepwise oxidation of nonanal through C_8-C_4 Alkanals (17).

The second important class of volatiles consistes of lactones. Table I shows that five lactones were isolated among them δ-dodecalactone was the predominant component in the control of samples C and B (38.30% and 30.30%, respectively) whereas it was identified with less concentration (10.20%) in control of sample I.

δ-Octalactone showed high concentration (14.60%) in control of sample B while δ-decalactone had nearly equal concentration (14.22%) in sample C. The usual description of lactones flavour has been coconut (6).

Four free fatty acids were identified in the investigated butter samples Table I. δ-Lactones and free fatty acids have been considered as important flavour components in dairy product (4).

However, on the basis of the actual concentration to threshold ratios. Urbach (6) and Sark *et al* (4) reported that butyric and hexanoic acids make no flavour contribution to butter.

In view of the results reported in Table I, it is clear that freeze storage of the three butter samples led to a significant effect on the separated carbonyls and lactones. In order to elucidate this effect, the total area percentages of these two chemical classes were determined and cited in Table III .The data in the table show that, the freeze storage gave rise to a gradual increase in the total yield of the carbonyls reaching their maximum value after 4.5 months followed by a noticeable decrease at the end of storage, this decrease may be attributed to the loss of some volatile carbonyl components during storage. These results are agreed with those obtained by Badings (9).

From the data shown in Table III, it is clear that, there is no regular behaviour for the yield of lactones components during storage. Samples C and B had the maximum value in their control samples, whereas sample I showed the highest yield after storage for 4-5 months. δ-Lactones are the conversion products of δ-hydroxy acids present in butter (18).

From the above mentioned results, it is clear that, the yield of the carbonyls which caused by peroxidation of fatty acids can be use as indication for the deterioration of the butter samples during storage.

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