## INVESTIGACIÓN

# Volatile compounds of Domiati cheese made from buffaloe's milk with different fat content

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#### RESUMEN

### Compuestos volátiles de queso Domiati fabricado de leche de búfalo con diferentes contenidos grasos.

La leche de búfalo fue procesada para obtener queso Domiati con diferente contenido graso en la cuajada (1%, 3.5% y 7%). Los resultados obtenidos durante el período de maduración revelaron que el queso con bajo contenido en grasa (Karish) no permite un largo período de almacenamiento, mientras que el queso con un contenido medio en grasa tuvo una buena calidad y flavor durante el período de maduración. El queso con alto contenido graso no duró más que el primer mes de maduración, deteriorándose posteriormente.

Los principales componentes encontrados fueron acroleína (propenal), heptanal, acetona, butan-2-ona, etanol, butan-2-ol, 2metil-propan-1-ol, 3 metil butan-1-ol, propionato de etilo, propionato de propilo, pentano y octano. Metil mercaptol, tiopropionato de metilo y trisulfuro de dimetilo junto con butirato de propilo se encontraron en muestras que fueron caracterizadas como muestras de quesos malos.

La mayoría de los compuestos anteriores se produjeron después de un mes de período de maduración.

PALABRAS-CLAVE: Compuestos volátiles – Leche de búfalo – Queso Domiati.

#### SUMMARY

Volatile compounds of Domiati cheese made from buffaloe's milk with different fat content.

Buffaloe's milk was manufactured to Domiati cheese with different fat content in the cheese milk (1%, 3.5% and 7%). Results obtained during the ripening period revealed that the low fat cheese (Karish) is not able to long period storage, while half cream cheese had a good quality and flavour along the ripening period. The full cream cheese did not exceed the first month of ripening, then it deteriorated.

The main components found were acrolein (propenal), heptanal, acetone, butan-2 one, ethanol, butan-2 ol, 2-methylpropan-1-ol, 3-methyl butan-1-ol, ethyl propionate, propyl propionate, pentane and octane. Methyl mercaptan, methyl thiopropionate, and dimethyl trisulfide together with propyl butyrate, were existed in the samples which are characterized as bad cheese samples. Most of the previous compounds were developed after 1 month of ripening period.

KEY-WORDS: Buffaloe's milk – Domiati cheese – Volatile compounds.

#### **1. INTRODUCTION**

Domiati cheese is the most popular soft pickled cheese, not only in Egypt, but it is also widely consumed allover Arab World. Domiati cheese was first investigated in 1947 (1) followed by further studies in 1950 (2). The volatile compounds of Domiati cheese and of other types of cheese form (soft or hard) depends on numerous factors. Cow's milk showed faster rate of protein breakdown than that made from buffaloe's milk (3), fresh milk released more free fatty acids than those released from dried milk (4). The additives in the milk prior to manufacture and their effect on ripening acceleration were also studied, e.g. whey protein (5), trypsin hydrolysate (6) or free fatty acids (7). The addition of hydrogen peroxide gave a firm - bodied and typical flavoured cheese, but usually with a slight chemical aftertaste (8, 9). An increase in the fat content improved the cheese flavour but had an adverse effect on the body and texture (10).

Cotton seed oil, when added to cheese milk, led to a slight oily flavour (11, 12). Using highly autolyzed cheese slurry (Mish) to enhance the flavour of Domiati cheese made from recombined dried milk, showed acceleration in ripening, besides reducing its time and resulting cheese is characterized by good flavour within one month (13). The hydrolysis of fat and released fatty acids were more evident in Domiati cheese where milk was treated with either homogenization or lipase addition. The most remarkable variation in the cheese flavour was observed at the end of storage period (14). The addition of lipase enhanced the development of pickled soft cheese flavour after 30 days of storage (15). Domiati cheese made from skim milk or whole dried had the same pattern of free fatty acids when lipase preparation added (16). The volatile compounds isolated from fresh Feta cheese by the head space method contained relatively large quantities of ethanol, propan-1-ol, butan-2-ol and butan-2-one as well as smaller amounts of pentane, propan-2-ol, ethyl acetate, 2-methyl propan-1-ol, toluene and ethyl butyrate (17). In Domiati cheese it was investigated that within three months of ripening the volatile compounds were acrolein, propane-1-ol, butan-2-one, butan-2-ol in addition to large number of esters which appear during maturation. Moreover, various sulfur compounds contribute significantly to the overall cheese aroma. Most of the volatile compounds seemed to develop after 2-months of maturation (18), However the aim of this study was to investigate the role of fat in flavour development of pickled soft cheese.

#### 2. MATERIALS AND METHODS

#### Samples

Buffaloe's milk (20 kg) was divided into three parts and standardized to contain fat as follows:

The first 1% fat (Karish, low fat),

The second 3.5% fat (half cream),

The third 7% fat (full cream).

The three parts of milk were manufactured separately to Domiati cheese samples by the traditional method (1).

Each of the produced samples was canned with its whey (6% salt) and stored at room temperature. The samples were analysed for the volatile compounds and sensory evaluation after one week (fresh), 1 month, 2 months, and three months.

The analysed samples composition were as follow: – Karish, Low fat: 10% fat / dry matter, 70% moisture and pH 6.03.

- Half cream cheese: 20% fat / dry matter, 65% moisture and pH 6.4.

- Full cream cheese: 45% fat / dry matter, 60% moisture and pH 6.51.

#### Organoleptic scoring of cheese

The cheese was scored for 1-Flavour 50 points

30 points
10 points
10 points
40 points
10 points

by a regular score pannels from the scientific staff of Department of Food Technology & Dairing, National Research Centre (19).

#### Isolation of volatile components

The volatile components of the cheese samples were extracted by simultaneous distillation method (20). About 200g of each sample were suspended in 500 ml distilled water, then continuously extracted with 200 ml pentane-diethyl ether (1:1). The pentane/ether extracts were dried over anhydrous sodium sulfate and the solvents were removed on a  $25 \times 1$  cm VIGREUX distilation column. The concentrates were stored under nitrogen.

### Gas chromatography - mass spectrometric analysis

A Varian 3400 GC equipped with DB - wax capillary column (O. 32 m m i.d. x 30 m) and coupled to a Finnigan - Mat 55 Q 7000 was used.

Analyses were carried out by using helium as carrier gas, flow rate 1.1 ml/min. Column temperature was maintained initially at 50°C for 6 min and then programmed from 50 to 260°C at a rate of 6°C/min. The injection port temperature was 250°C. The ionization voltage applied was 70 ev. Peaks were identified by comparison with data from the library of mass spectra, literature data and mass spectra retention times of authentic samples. Quantitative determination was carried out based on peak area integration. Authentic components were purchased from Aldrich and Sigma Co, s.

#### 3. RESULTS AND DISCUSSION

Table I shows the volatile components obtained from the three different cheese samples (Karish, half and full cream). About 44 compounds belonging to six chemical groups, namely, aldehydes, ketones, alcohols, esters, sulfur compounds, and hydrocarbons were identified.

Table II shows the component groups of the investigated cheese samples. It is observed that aldehydes content in the karish cheese fluctuated throughout the ripening period. It started with 5.79% in fresh cheese and decreased to 1.63% after one month, while the second month revealed the maximum value of aldehydes reaching 12.56%. The increment of aldehydes did not occur in the third month but, in contrary, decreased sharply to reach 3.70%. However, the major aldehyde for the fresh sample was heptanal compared to acetaldehyde and propenal (acrolein) which reported significant values during the second and third month of ripening. Ketones on the other hand started with a value of 5.15%, then increased to 11.51% and 14.76% in the first and the second month of ripening, respectively. Thereafter, the value decreased to 8.29% during the third month of ripening. However, the predominant ketone for the fresh karish cheese sample was heptan-2-one while the major ketone during the first and the second month of ripening was butan-2-one. In the third month of

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		Low fat		Half cream			Full cream							
Peak t <sub>R</sub> No								Ripen	ing period	d / month	1			Components
		Fresh	I			Fresh	I	]]		Fresh	1			
1	4.6	1.23*	0.25	4.24	0.92	4.17	0.60	4.09	0.94	3.56	4.16	21.39	0.07	Acetaldehyde
2	5.1												0.15	Methyl mercaptan
3	6.3	2.42	0.07	1.16	0.03	18.57	0.94	1.27	1.14	5.21	0.94	3.14	0.28	Ethanol
4	6.7		0.06	4.05	2.32		11.55	1.16	0.99		5.72	6.11	1.81	Acrolein (propenal)
5	7.1	0.63	0.09	1.05	7.50	8.88	0.35	1.96	1.91	4.63	0.27	1.59	0.62	Acetone
6	7.9	6.05	0.15	0.61	10.06	2.12	0.31	1.01	0.74	0.97	0.22	1.52	0.23	Pentan
7	8.2		0.09	2.92	14.42	0.64	2.94	2.26	1.15	2.60	0.79	1.74	0.17	Ethyl formate
8	8.4													Dimethyl sulfide
9	10.3	0.17				0.94				1.06				lso butyraldehyde
10	11.2	1.43	4.34	12.51	2.20	13.1	5.82	2.56	2.96	8.76	0.69	0.97	1.06	Propan-1-ol
11	12.3		0.40	1.51										Diacetyl
12	12.7		0.40											Butyraldehyde
13	13.5	0.87	11.02	8.35	0.20		0.54	0.97	1.07	5.97	0.66	0.42	0.84	Butan-2-one
14	15.1	0.20	2.19	1.63	2.18	2.87	3.67	1.93	1.39	3.55	11.13	0.77	1.108	Butan-2-ol
15	16.0	0.35	7.26	11.02	2.18	1.57	1.04	1.34	1.90	2.24	0.46	0.46	0.22	Ethyl acetate
16	16.2		5.73											Unknown
17	17.9	0.81	6.09	2.59	0.11	6.16	12.19	1.97	1.79	6.98	11.44	0.86	2.44	2-Methyl-propan-1-ol
18	19.7	0.19	0.92	0.77				·Τ	2.90			0.44	Т	3-Methyl butyraldehyde
19	21.1				0.20	1.77	0.94	0.61	0.63	5.34		0.62	5.20	2-Methyl butyraldehyde
20	24.2	1.26			0.03	2.16	0.36	0.49	0.67	6.98		0.65	1.11	Pentan-2-one
21	24.6								0.79					Pentan-2,3-dione
22	24.8													Valeraldehyde
23	25.1		1.05				0.27	0.61			0.61	0.59	1.03	Methyl thioacetate
24	27.1		1.34		0.01		0.26	1.01	0.94		0.75	0.65	1.43	Pentan-2-ol
25	28.2				0.06			0.63	0.41		0.20			Ethyl propionate
26	28.6	0.84	1.48	1.81	0.10	30.64	0.27	0.77	3.26	5.68	0.91	0.48	0.68	Propyl acetate
27	30.7				0.07		0.17	0.71						Dimethyl disulfide
28	31.0	1.24	0.90	0.36	0.04	2.46	0.30	1.04	3.46	13.73	1.81	0.96	0.61	3-methyl butan-1-ol
29	31.8				Т				Т	Т			Т	2-Methyl butan-1-ol
30	34.1	3.81	0.40	0.36		2.10	0.12	0.36	1.64	4.22	0.54	0.96	23.05	Un known
31	34.3				Т		Т	Т	3.56			Т	Т	Toluene
32	36.2													Hexan-2-one
33	37.3			Т	Т				Т				2.53	Methyl thiopropionate
34	37.5													Capraldehyde
35	38.6	9.54	40.68	15.20	0.64	0.67	1.40	6.86	5.70	5.74	27.42	2.79	11.34	Ethyl butyrate
36	39.7	55.20		4.10	0.45		54.43	48.29	46.56	12.68	28.91	10.33	15.03	Propyl propionate
37	39.8			2.25		1.08	0.23	1.45	1.29		0.27	1.18	0.79	Octane
38	40.1													Butyl acetate
39	45.4		6.14	1.87	3.12			6.20	6.50		0.69	1.93	0.88	Methyl thiobutyrate
40	46.3	3.40	1.22					3.50			0.33	0.74		Pm- xylene
41	47.0		1.38	5.10				0.99	2.17		0.33	1.92	0.44	lso amyl acetate
42	48.0	3.26		3.85	0.56							1.18	0.41	Heptan-2-one
43	48.5	4.11		3.50	0.26			1.07				0.98	0.31	Heptanal
44	48.8	2.89										0.63	0.16	o- xylene
45	49.2		6.32	9.07	39.24		1.190	4.74	3.93		1.29	31.54	25.19	Propyl butyrate
46	57.0				12.97				2.09			2.33	0.76	Dimetyl trisulfide

# Table I Volatile components identified during the ripening period for Domiati cheese with different fat content

\* Values expressed as area percentages.

T = trace.

- = not detected or not determined.

ripening, acetone showed the highest value among the ketone compounds of karish cheese. Alcohols of karish cheese had the same trend of the ketone compounds. The alcohol compounds were 6.10% for the fresh cheese, then increased during the first and the second months of ripening. The lowest value for alcohols occured at the end of ripening period, being 4.57%. However, ethanol revealed the highest value among the alcohols of the karish sample. On the other hand, propan-1-ol and butan-2-ol were the predominant alcoholes in the ripened karish cheese. The ester compounds in fresh karish cheese recorded 66.81% which is quite a high value. The level of esters fluctuated during the ripening period being 49.34% and 57,22% for the second and third months of ripening, respectively. Propyl propionate was the major ester in fresh karish cheese. During ripening, ethyl butyrate was the major ester in the first month, while ethyl butyrate and ethyl acetate showed higher levels during the second month with propyl butyrate being the highest level at the end of the ripening period. Since the karish cheese curd consists mainly of casein and low content of fat, the substrate applied throughout the ripening period was milk protein. Sulfur compounds were not detected in fresh karish cheese, while marked concentration of 7.19% appeared after one month followed by decrease during the second month and strong increase (16.16%) at the end. Methyl thiobutyrate being responsible in fresh and one mouth samples, in addition, dimethyl trisulfide, and dimethyl disulfide were the sulfur compounds detected at the end of ripening period.

#### Table II

#### Volatile compounds groups and total score of sensory evaluation obtained for three cheese samples during ripening period. (expressed as area percent)

Low fat (Karish) cheese							
-	Low fat cheese						
Volatile groups	Fresh	I	II	111			
Aldehydes Ketones Alcohols Esters Sulfur compounds Hydrocarbons	5.79 5.15 6.10 66.81 0 12.34	1.63 11.51 14.93 58.00 7.19 1.37	12.56 14.76 18.25 49.34 1.87 2.86	3.70 8.29 4.57 57.22 16.16 10.06			
Total organoleptic test score (100)	97	57	10	5			

	Half cream cheese						
Volatile groups	Fresh	I	11	111			
Aldehydes	6.88	13.09	6.93	5.46			
Ketones	11.04	1.25	3.42	3.65			
Alcohols	40.29	23.18	9.78	11.68			
Esters	36.49	61.38	66.03	63.39			
Sulfur compounds	0	0.44	7.52	8.59			
Hydrocarbons	3.20	0.54	5.96	5.59			
Total organoleptic test score (100)	55	60	80	93			

	Full cream cheese						
Volatile groups	Fresh	I	I	III			
Aldehydes	9.88	9.88	29.54	7.39			
Alcohols	38.23	26.77	7.83	6.92			
Esters Sulfur compounds	29.14 0	60.45 0.61	48.91 4.85	53.13 6.35			
Hydrocarbons	0.97	0.82	4.07	1.18			
Total organoleptic test score (100)	55	85	71	35			

Hydrocarbons are found in higher concentrations for fresh and three months ripening samples, while the lower concentrations were observed during the first and the second months. The hydrocarbon compounds were found to be pentane and o-xylene for the fresh sample. Low concentration (0.15%) of pentane was detected in the second month of ripening. Pentane and octane were reported in the sample of two months ripening, while only pentane was detected in higher concentration during the third month. From the previous findings, it can be concluded that, inspite of the low fat content of the cheese, the ripening period led to a chemical or biological degradation of such low fat content, but the presence of the protein as a major substrate led to the higher concentration of the sulfur compounds due to such degradation (21, 22, 15 and 23).

Comparative data of the volatile compounds of fresh and ripened Domiati cheese considering the fat content of two samples revealed several differences not only in their concentrations, but also in the ripening time to obtain a good quality cheese.

Table II shows the volatile groups for the half and full cream cheese.

The concentration of the aldehyde compounds for the fresh half cream cheese were lower than those for fresh full cream cheese, 6.88% and 9.88%, respectively. On the other hand, the trend of aldehydes decreased throughout the ripening period for the half cream cheese, while the full cream cheese sample had the highest level of aldehydes in the second month. Nevertheless, both samples contained the same aldehyde compounds, acetaldehyde, propenal, isobutyraldehyde, 3-methyl butyraldehyde, 2-methyl butyraldehyde, while the later is found in higher ratio in the full cream cheese sample, which might be derived from Strecker or microbiological degradation of amino acids (18).

Ketone compounds of fresh half cream cheese scored 11.04% among the other volatile compounds, leveling up to 17.58% for the full cream fresh cheese. Acetone and pentan-2-one were the ketones detected for the half cream cheese, however pentan-2-one was found in higher ratio for the full cream cheese. The high content of fat in the full cream cheese sample, might be the cause for the presence of butan-2-one due to beta oxidation of fatty acids (24).

The alcohol compounds (table II) were represented in both cheese samples as ethanol, propan-1-ol, butan-2-ol, 2-methylpropan-1-ol, and 3 methyl butan-1-ol. The fresh half cream cheese sample had a higher ratio of ethanol and propan-1-ol than the fresh full cream cheese sample, while the reverse was observed for 3-methyl butan-1-ol. It is likely that these compounds could contribute to the differences in flavour between these two samples (17). Same alcohol compounds were found in both ripened cheese samples, but with variance in values. Butan-2-ol and 2methylpropan-1-ol were the major alcohols during the ripening period of the full cream cheese sample, while the 2-methylpropan-1-ol and propan-1-ol were the major aldehydes for the half cream cheese sample. Ethyl formate, ethyl acetate, propyl acetate, ethyl butyrate, propyl propionate were the esters detected for the full cream cheese. However, esters of fresh half cream recorded 36.49%, compared to 29.14% for the full cream cheese sample. Propyl acetate and propyl propionate were the predominant esters detected for the fresh half and full cream cheese, respectively. Ester compounds of the fresh half cream cheese were significantly increased during the three months of ripening reaching their maximum in the second month. Among these esters, propyl propionate was the major ester detected throughout the ripening period as for the ester compounds of the full cream cheese they showed lower ratio than for the half cream samples. However, the full cream cheese had various esters in higher concentrations; ethyl, butyrate, propyl propionate and propyl butyrate beeing the major esters along the ripening period. The difference between the flavour of the half and full cream cheese samples might be due to the higher content of ethyl butyrate and propyl butyrate in the later samples. This finding is presumably attributable to the high content of fat in the full cream cheese samples, that may facilitate releasing short and medium chain fatty acids. However it has been reported that most of the esters arise from the fat hydrolysis (21, 25, 26, 27).

Various sulfur compounds such as methyl mercaptan, dimethyl disulfide, methyl thiopropionate, methyl thiobutyrate and dimethyl trisulfide were

detected in different ratios. The fresh samples of the half and full cream cheese were clear of the sulfur and compounds. Methyl mercaptan methyl thiopropionate did not appear during the ripening period for the half cream cheese samples. Furthermore, dimethyl trisulfide was detected only at the end of the ripening period (third month), whereas methyl thiobutyrate existed significantly in the second and the third months, while dimethyl disulfide was detected in low percentages in the first and second months. Nevertheless, the samples of half cream cheese had a high score for its sensory evaluation.

Concerning the sulfur compounds existing in the samples of the full cream cheese, methyl mercaptan and methyl thiopropionate were reported only in the third month, while methyl thiobutyrate appeared from the first month to the end of ripening period. Dimethyl trisulfide existed through the second and the third months.

From the previous findings it is concluded that the methyl mercaptan and methyl thiopropionate were the sulfur compounds found in the full cream cheese sample in the third month, which could be the cause that the sample had the lower score in sensory evaluation and the off flavour detected in the cheese sample. Hydrocarbon compounds, mainly pentane and octane, were detected for the half cream cheese samples along the three months of ripening. Some other hydrocarbons existed frequently such as xylene isomers and toluene, when were detected during the second and the third months, respectively. On the other hand, all of the full cream cheese samples contained, pentane, octane, and xylene isomers were observed during the ripening period. However, pentane and octane, were probably produced through the oxidative breakdown of unsaturated fatty acids (28). From the previous discussions it is concluded that, the leakage of fat severely affected the flavour of the cheese during the ripening period so much that the flavour of the cheese becomes bad since the first month of ripening. The half cream cheese reached its maximum good flavour during the third month of ripening, which means that the fat content of half cream cheese was the optimum level for Domiati cheese ripening within three months. The increment of fat content in the cheese as presented in the full cream cheese sample did not give the promising results. The good quality of the full cream cheese did not cross the first month of ripening, when the flavour deterioration of the cheese existed since the second month of ripening.

So, the best time for consuming the Karish cheese, is in fresh state (the usual consumption time), while the best time for consuming the half cream cheese under the manufacturing conditions mentioned previously is accepted up to three month of ripening. The optimum consuming time for the stored full cream cheese is within the second month as maximum.

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