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GC analysis in evaluation of changes in fatty acids content of selected fats during storage and heating

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The purpose of the paper was the comparison of quantitative changes in fatty acids composition in selected fats (soybean, sunflower, rapeseed, and olive oils, as well as margarine, butter and porcine lard) during their storage and heating. Comparison of quantitative analysis results expressed as absolute fatty acids amounts (g 100 g^{-1} – internal standard addition method) with their relative contents after summing up the analyzed components to 100% (internal normalization method), is presented. Experimental data revealed a great differentiation between quantitative results of particular fatty acids groups expressed as absolute amounts (g 100 g^{-1}), and as percentages assuming that the sum of fatty acids in a sample was 100%. In general, the obtained results indicated that long-term storage at both ambient and elevated temperature led to a decrease in SFA, MUFA and PUFA contents (g 100 g^{-1} of sample) in the triacylglycerols fraction. Comparison of absolute and relative contents indicated that more credible results could be achieved only by means of analytical techniques based on internal standard addition or other analytically-equivalent methods that can give a full quantitative representation of the absolute amounts of substances tested.

Keywords: Fats, fatty acid methyl ester, gas chromatography, heating, quality, storage.

INTRODUCTION

Dietary fats are the most concentrated source of energy, fat-soluble vitamins and essential unsaturated fatty acids (UFA). They also build their own tissues and take part in the synthesis of some biologically active substances (eicosanoids), namely prostaglandins counted among tissue hormones.

Fatty acids contained in fats are oxidized in tissues, producing appropriate amounts of energy, or are utilized for building body components. Fatty acids are components of cellular membranes and cellular organelles affecting their permeability for nutrients that are transferred to human organism cells.

Plant oils are essential for human diet due to UFA content [1–8]. High level of unsaturation of fatty acids present in oils is required by nutritionists, and at the same time, it makes great problems for food technologists due to their higher susceptibility to oxidation. Oxidation processes in fats cause worsening of the sensory quality of food products and decrease their nutritional value; final products of these processes cause ageing of the organism, as well as take part in the etiology of such diseases as coronary vessels disturbances and tumors [9]. Moreover, hydrolysis and polymerization processes can occur during fat warming. Intensity of these changes depends on temperature, time and way of heating. Polymerization is the most undesired conversion taking place during fat warming, namely long-term or repeated one. It was found that heating of plant-origin oils containing linoleic acid both in the air and under nitrogen atmosphere, besides dimers and polymers, also formed monomers of cyclic structure (which are counted to agents probably responsible for pathogenesis), and fatty acids of unidentified structure [10]. Products formed during the above mentioned transformations of fatty acids are stable and are not a typical triacylglycerols fraction of fatty acids that may be determined by means of GC technique after saponification and esterification.

There are many studies dealing with changes of fatty acids composition in stored or heated fats [11–17]. Unfortunately, they do not contain full quantitative evaluation of particular fatty acids in triacylglycerols, but only give their percentage assuming the sum in a fat to be 100%. Such quantitative evaluation leads to some result incorrectness, which is particularly apparent in the case of significant amounts of very durable oxidized or polymerized substances formed during fat storage or heating.

Therefore, the purpose of the paper was the analysis of quantitative changes in fatty acids composition in selected fats available on the market during their storage and heating. Moreover, a

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relative comparison of quantitative analysis results expressed as absolute fatty acids amounts (g/100 g – internal normalization method) with their relative contents after totalling the analysed components to 100% (internal standard addition method), is presented in the paper.

EXPERIMENTAL

Fats

Selected fats of plant and animal origin, purchased in a store, were the material for study: soybean oil, sunflower oil, rapeseed oil, olive oil, margarine, butter, porcine lard. Aliquots of 20 g fats were placed in glass bulbs of 50 ml capacity, marked in accordance with Table 1, and then subjected to experimental factors action: heating in a thermostat at 120°C with long-term storage at various temperatures (-22°C in dark; +4°C in dark; ambient with daylight). Samples for analyses were taken from thermostatically processed fats after 120, 192, and 288 h of high-temperature action, and after 70 and 140 days of storage at -22°C, +4°C and ambient temperature.

Table 1. Samples designation

Sample	Sample
1	Raw oil – control
2	Fat heated at +120°C – 120 h
3	Fat heated at +120°C – 192 h
4	Fat heated at +120°C – 288 h
5	Fat stored at ambient temperature – 70 days
6	Fat stored at ambient temperature – 140 days
7	Fat stored at +4°C – 70 days
8	Fat stored at +4°C – 140 days
9	Fat stored at -25°C – 70 days
10	Fat stored at -25°C – 140 days

Analytical methods

Aliquots of 300 μl of heptadecanoic acid (Sigma) solution in hexane (10 mg ml^{-1}) were added to weighed samples of the fats (about 50 mg). Fat saponification and fatty acids esterification (with 14% methanolic solution of BF_3) was performed according to standards IUPAC-AOAC-963.22 [18] and AOAC 969.33 [19]. The quantitative composition of FAMES was determined by GC (FID). The quantitative analysis was performed on the basis of calibration curves for a FAMES standard mixture (C14 – C20) within the concentration range of 0.1 – 80.0 g 100 g^{-1} [20–22]. Water presence in the studied fats was taken into account in the obtained results.

Gas chromatography

GC was performed on a Unicam 610 Series gas chromatograph equipped with a flame-ionization detector and a 60 m (0.25 mm i.d.) column coated with a 0.25 μm film of HP-23. A temperature gradient was applied (160°C for 1 min, then incremented by 2.75°C min^{-1} to 215°C, held at 215°C for 2 min, then incremented by 40°C min^{-1} to 230°C and held at 230°C for 2 min). The injection port and detector temperatures were 270°C; split ratio 1:50. Hydrogen was used as carrier gas at a flow rate of 43 m s^{-1}.

RESULTS AND DISCUSSION

Figs. 1-7 present the results of fatty acids contents in the examined fats during their storage and heating. Data in Table 2 reveal a great differentiation between the quantitative results of particular fatty acid groups presented as absolute amounts (g 100 g^{-1}) and as percentages assuming that the sum of fatty acids in a sample was 100% (internal normalization method).

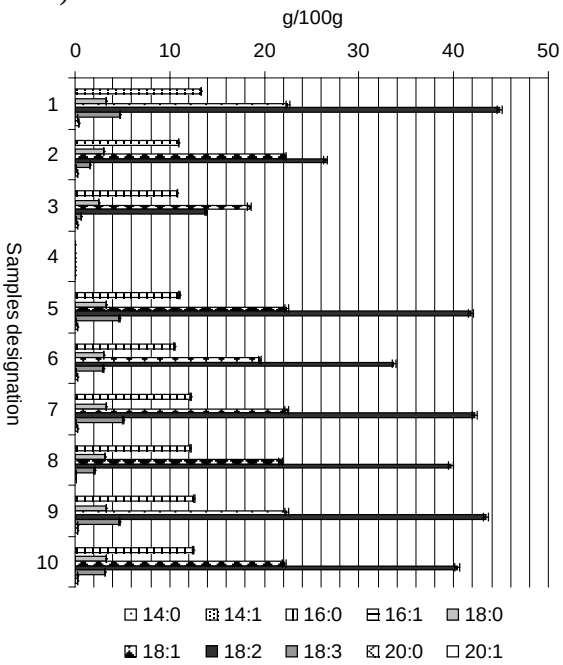

Fig. 1. Fatty acids contents in soybean oil stored under different conditions and heated at +120°C (designations as in Table 1). 14:0, myristic acid; 14:1, myristicoleic acid; 16:0, palmitic acid; 16:1, palmitoleic acid; 18:0, stearic acid; 18:1, oleic acid; 18:2, linoleic acid; 18:3, α-linolenic acid; 20:0, arachidonic acid; 20:1, eicosenoic acid.

In general, the obtained results indicated that long-term storage at ambient temperature and heating led to a decrease in SFA, MUFA and PUFA contents (g 100 g^{-1} of sample) in the triacylglycerols fraction, which is probably associated with the increase in polymerized and oxidized forms levels at the absence of mass exchange between the experimental system and the surroundings – the weight of stored or heated fat remains constant.

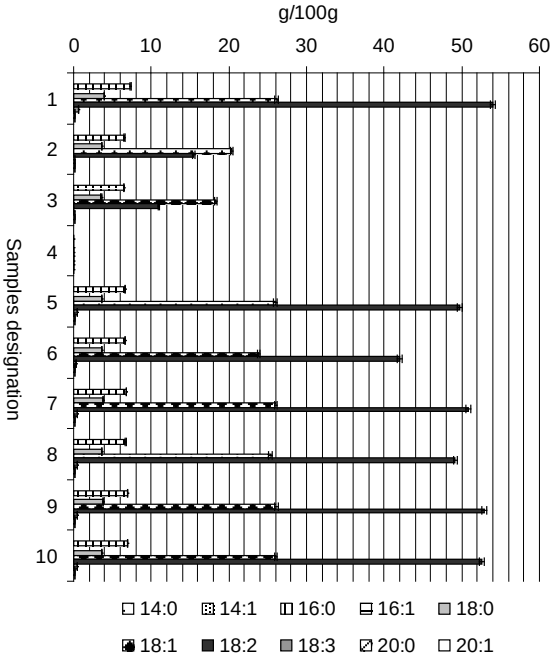

Fig. 2. Fatty acid contents in sunflower oil stored under different conditions and heated at +120°C (designations as in Table 1). 14:0, myristic acid; 14:1, myristicoleic acid; 16:0, palmitic acid; 16:1, palmitoleic acid; 18:0, stearic acid; 18:1, oleic acid; 18:2, linoleic acid; 18:3, α-linolenic acid; 20:0, arachidonic acid; 20:1, eicosenoic acid.

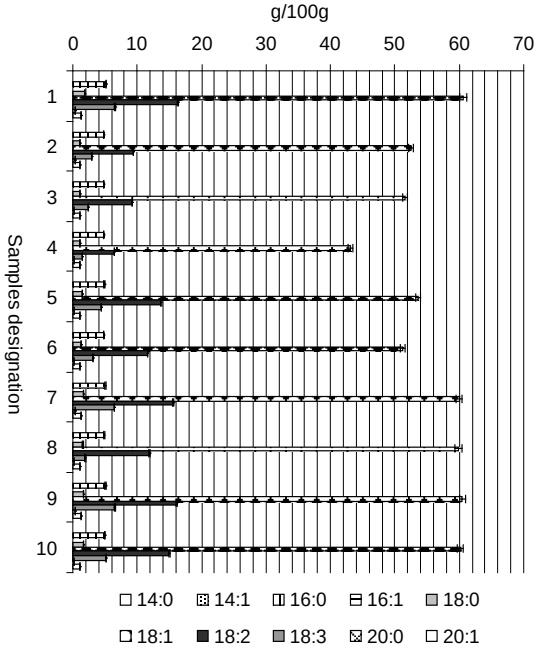

Fig. 3 Fatty acid contents in rapeseed oil stored under different conditions and heated at +120°C (designations as in Table 1). 14:0, myristic acid; 14:1, myristicoleic acid; 16:0, palmitic acid; 16:1, palmitoleic acid; 18:0, stearic acid; 18:1, oleic acid; 18:2, linoleic acid; 18:3, α-linolenic acid; 20:0, arachidonic acid; 20:1, eicosenoic acid.

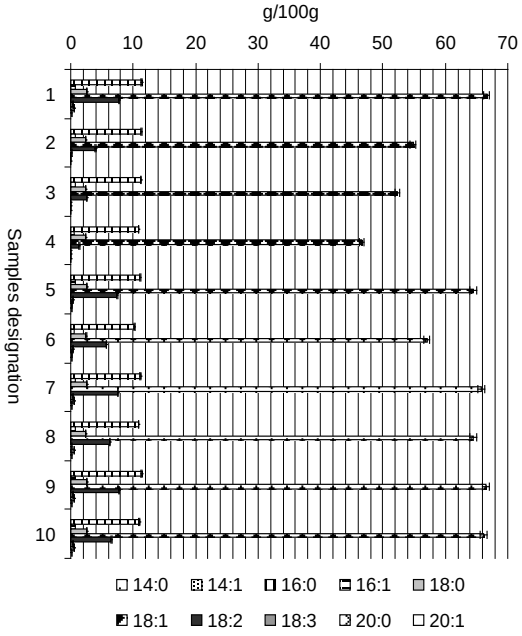

Fig. 4. Fatty acid contents in olive oil stored under different conditions and heated at +120°C (designations as in Table 1). 14:0, myristic acid; 14:1, myristicoleic acid; 16:0, palmitic acid; 16:1, palmitoleic acid; 18:0, stearic acid; 18:1, oleic acid; 18:2, linoleic acid; 18:3, α-linolenic acid; 20:0, arachidonic acid; 20:1, eicosenoic acid.

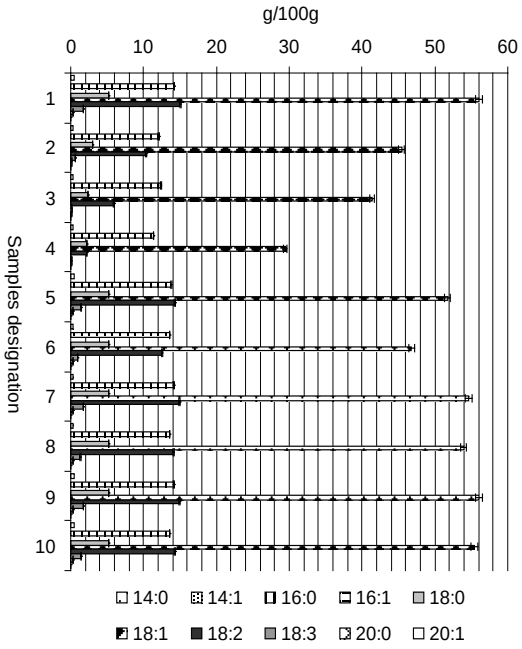

Fig. 5. Fatty acid contents in margarine stored under different conditions and heated at +120°C (designations as in Table 1). 14:0, myristic acid; 14:1, myristicoleic acid; 16:0, palmitic acid; 16:1, palmitoleic acid; 18:0, stearic acid; 18:1, oleic acid; 18:2, linoleic acid; 18:3, α-linolenic acid; 20:0, arachidonic acid; 20:1, eicosenoic acid.

Data on above changes, expressed as a percentage of total fatty acids, indicate that the shares of SFA and MUFA increased, but PUFA percentage in fats shows a decreasing tendency due to experimental conditions.

Relative comparison of both data forms from Table 2 indicates that more credible information can be achieved in that type of experiment applying analytical techniques based on internal standard addition or other analytically-equivalent methods. When interpreting the obtained results from quantitative analysis based on the assumption that the sum of fatty acids in the studied fraction is 100%, a major error is committed, because only semi-quantitative information on fatty acid profile in the studied sample can be obtained in this case.

Table 2. Changes in quantitative composition of particular fatty acids groups in fats stored under different conditions and heated at +120°C (designations as in Table 1). * - sample's state of matter resembling "gum-resin"

Fat	Sample	Fatty acid groups					
		SFA		MUFA		PUFA	
		g 100 g^{-1} of sample	Percentage	g 100 g^{-1} of sample	Percentage	g 100 g^{-1} of sample	Percentage
Soybean oil	1	16.88±0.15	18.89±0.17	22.81±0.30	25.55±0.33	49.57±0.55	55.53±0.61
	2	14.23±0.13	21.90±0.20	22.40±0.29	34.64±0.45	28.03±0.31	43.35±0.48
	3	13.48±0.12	28.76±0.26	18.61±0.24	40.00±0.52	14.43±0.16	31.02±0.34
	4	*	*	*	*	*	*
	5	14.48±0.13	17.28±0.16	22.57±0.29	27.00±0.35	46.55±0.51	55.68±0.61
	6	13.69±0.12	19.47±0.18	19.72±0.26	28.15±0.37	36.66±0.40	52.32±0.58
	7	14.71±0.13	17.35±0.16	22.64±0.29	26.75±0.35	47.27±0.52	55.86±0.61
	8	14.48±0.13	19.73±0.18	20.93±0.27	28.62±0.37	37.72±0.41	51.58±0.57
	9	15.12±0.14	17.56±0.18	22.66±0.29	26.37±0.34	48.13±0.53	56.03±0.62
	10	14.95±0.13	18.45±0.16	22.35±0.29	27.65±0.36	43.53±0.48	53.85±0.59
Sunflower oil	1	11.42±0.10	12.35±0.11	26.37±0.34	28.55±0.37	54.58±0.60	59.09±0.65
	2	10.40±0.09	22.17±0.20	20.52±0.27	44.13±0.57	15.58±0.17	33.51±0.37
	3	10.17±0.09	25.43±0.23	18.52±0.24	46.76±0.61	10.91±0.12	27.56±0.30
	4	*	*	*	*	*	*
	5	10.47±0.09	12.03±0.11	26.19±0.34	30.16±0.39	50.17±0.55	57.79±0.64
	6	10.38±0.09	13.47±0.12	24.06±0.31	31.32±0.41	42.37±0.47	55.16±0.61
	7	10.74±0.10	12.14±0.11	26.24±0.34	29.71±0.39	51.35±0.56	58.13±0.64
	8	10.51±0.09	13.35±0.12	24.35±0.32	31.02±0.40	43.62±0.48	55.58±0.61
	9	11.01±0.10	12.10±0.11	26.32±0.34	28.99±0.38	53.46±0.59	58.89±0.65
	10	10.82±0.10	11.98±0.11	26.24±0.34	29.10±0.38	53.11±0.58	58.90±0.65
Rapeseed oil	1	7.25±0.07	7.85±0.07	61.87±0.80	67.27±0.87	22.86±0.25	24.86±0.27
	2	6.22±0.06	8.50±0.08	53.64±0.70	74.26±0.97	12.37±0.14	17.13±0.19
	3	6.14±0.06	8.61±0.08	52.69±0.68	74.72±0.97	11.68±0.13	16.57±0.18
	4	6.08±0.05	10.32±0.09	44.18±0.57	75.99±0.99	7.88±0.09	13.55±0.15
	5	6.72±0.06	8.37±0.08	54.74±0.71	68.79±0.89	18.12±0.20	22.77±0.25
	6	6.27±0.06	8.49±0.08	52.22±0.68	71.26±0.93	14.79±0.16	20.18±0.22
	7	6.95±0.06	7.81±0.07	59.41±0.77	67.14±0.87	22.13±0.24	25.01±0.28
	8	6.57±0.06	8.27±0.07	58.65±0.76	74.16±0.96	13.87±0.15	17.54±0.19
	9	7.02±0.06	7.74±0.07	60.56±0.79	67.07±0.87	22.72±0.25	25.17±0.28
	10	6.81±0.06	7.89±0.07	59.01±0.77	68.70±0.89	20.08±0.22	23.38±0.26
Olive oil	1	14.53±0.13	16.04±0.14	67.64±0.88	74.92±0.97	8.11±0.09	8.98±0.10
	2	13.79±0.12	18.78±0.17	55.56±0.72	75.65±0.99	4.09±0.04	5.57±0.06
	3	13.66±0.12	19.74±0.18	53.01±0.69	76.59±1.02	2.54±0.03	3.67±0.04
	4	13.32±012	21.52±0.19	47.21±0.61	76.26±0.99	1.38±0.02	2.22±0.02
	5	13.94±0.13	15.95±0.14	65.34±0.85	74.96±0.97	7.89±0.09	9.05±0.10
	6	12.98±0.12	16.80±0.15	57.88±0.75	75.27±0.98	6.04±0.07	7.85±0.09
	7	14.19±0.13	16.13±0.15	65.44±0.85	74.73±0.97	7.94±0.09	9.07±0.10
	8	13.74±0.12	16.44±0.16	62.92±0.82	75.73±1.00	6.42±0.07	7.73±0.09
	9	14.39±0.13	15.92±0.15	67.50±0.88	74.94±1.02	8.18±0.09	9.09±0.10
	10	13.97±0.13	16.05±0.16	65.82±0.86	75.97±1.01	6.85±0.08	7.91±0.09

Table 2. Continued

Fat	Samples	Fatty acid groups					
		SFA		MUFA		PUFA	
		g 100 g^{-1} of sample	Percentage	g 100 g^{-1} of sample	Percentage	g 100 g^{-1} of sample	Percentage
Margarine	1	20.21±0.18	21.72±0.20	56.02±0.73	60.26±0.78	16.73±0.18	18.00±0.20
	2	15.64±0.14	21.77±0.20	45.43±0.59	63.39±0.82	10.95±0.12	15.28±0.17
	3	15.14±0.14	24.30±0.22	41.31±0.54	66.49±0.86	6.04±0.07	9.72±0.11
	4	14.01±0.13	30.65±0.28	29.52±0.38	64.99±0.84	2.19±0.02	4.82±0.05
	5	19.76±0.18	22.73±0.20	51.76±0.67	59.65±0.78	15.68±0.17	18.07±0.20
	6	19.45±0.18	24.45±0.22	46.79±0.61	58.99±0.77	13.46±0.15	16.97±0.19
	7	20.08±0.18	22.68±0.20	52.06±0.68	58.92±0.77	16.61±0.18	18.80±0.21
	8	19.44±0.17	22.72±0.20	50.90±0.51	59.59±0.75	15.47±0.17	18.11±0.20
	9	20.14±0.18	22.53±0.20	52.87±0.41	59.24±0.76	16.66±0.18	18.67±0.21
	10	19.55±0.18	22.40±0.20	52.26±0.39	60.00±0.80	15.70±0.17	18.03±0.20
Butter	1	46.75±0.42	51.37±0.46	39.24±0.33	43.13±0.56	4.99±0.05	5.48±0.06
	2	41.82±0.38	54.56±0.49	31.58±0.46	41.24±0.54	3.17±0.03	4.14±0.05
	3	38.11±0.34	53.73±0.48	30.24±0.41	42.69±0.55	2.49±0.03	3.51±0.04
	4	38.32±0.34	58.92±0.53	25.72±0.41	39.60±0.51	0.92±0.01	1.41±0.02
	5	34.64±0.31	47.07±0.42	35.36±0.39	48.10±0.63	3.52±0.04	4.78±0.05
	6	33.15±0.30	48.78±0.44	31.39±0.48	46.25±0.60	3.33±0.04	4.90±0.05
	7	45.62±0.41	56.00±0.50	31.22±0.46	38.35±0.50	4.56±0.05	5.60±0.06
	8	44.94±0.40	56.94±0.51	30.28±0.53	38.39±0.50	3.64±0.04	4.62±0.05
	9	46.37±0.42	52.65±0.47	36.73±0.48	41.73±0.54	4.93±0.05	5.60±0.06
	10	45.56±0.41	53.80±0.48	35.10±0.46	41.48±0.54	3.97±0.04	4.69±0.05
Porcine lard	1	40.51±0.36	44.56±0.40	40.86±0.53	44.97±0.58	9.49±0.10	10.45±0.11
	2	33.73±0.30	45.79±0.41	37.08±0.48	50.39±0.66	2.78±0.03	3.77±0.04
	3	32.89±0.30	48.51±0.44	33.13±0.43	48.92±0.64	1.71±0.02	2.52±0.03
	4	26.41±0.24	47.53±0.43	28.07±0.36	50.60±0.66	0.99±0.01	1.79±0.02
	5	37.11±0.33	43.58±0.39	39.23±0.51	46.10±0.60	8.75±0.10	10.28±0.11
	6	34.21±0.31	46.04±0.41	33.00±0.43	44.46±0.58	7.01±0.08	9.44±0.10
	7	36.32±0.33	42.26±0.38	40.74±0.53	47.43±0.62	8.82±0.10	10.27±0.11
	8	35.90±0.32	45.19±0.41	35.88±0.47	45.21±0.59	7.58±0.08	9.55±0.11
	9	39.84±0.36	44.32±0.40	40.59±0.53	45.17±0.59	9.42±0.10	10.48±0.12
	10	37.71±0.34	43.57±0.39	39.90±0.52	46.12±0.60	8.89±0.10	10.28±0.11

Such analytical procedure is correct only in routine qualitative evaluation, but it has to take into account other parameters, i.e. color, acidic number, peroxide number, anisidine number, Totox index or oil oxidation stability determined by means of Rancimat test. It should be underlined that the above mentioned parameters not always can characterize the utilization value of a fat. Flaczyk *et al*. [23] and Wroniak *et al*. [24] did not find direct influences of the peroxide content in the studied oils on their oxidation stability. Despite the very high Totox index for olive oil and sunflower oil, those fats were characterized by the highest oxidation stability in the Rancimat test. Therefore, the quantitative analysis by means of GC technique with internal standard and calibration in temperature and storage tests is much simpler and faster, and it supplies more detailed information on the changes in the studied fat composition. However, GC method with internal standard may seem insufficient in routine qualitative evaluation of single fat samples, because there are no reference norms (what levels of fatty acids are desirable for a given product). Thus, in that case, the qualitative and quantitative composition of fresh and stored oils, as well as oils subjected to various thermal processing operations should be determined. It should also be underlined that a proper selection of internal standard or standards, reference material (certified mixture of triacylglycerols) along with validation of the whole analysis is necessary in such procedures. That type of evaluation could be a basis for determining the limiting values for particular fatty acids, which would serve for routine qualitative analysis of various fats.

The experiment revealed that the least changes were found in samples stored at -25°C and +4°C, which depended on the storage time. The most apparent changes in fatty acids contents were

recorded in samples stored at ambient temperature. Fats heated in a thermostat at +120°C were characterized by a dynamic decrease in fatty acids concentrations. Oils containing polyunsaturated fatty acids (sunflower and soybean oils) were particularly susceptible to high-temperature effects: losses of fatty acids contents were respectively about 53% and about 43% after 192 h of heating. Intensive oxidation of these oils, resulting in conversion of the oils into liquids with very high viscosity, like resin, took place after 288 h of samples treatment at high temperature. Besbes *et al*. [14] found that palm oil heated at 120°C for 48 h had significantly higher viscosity than unheated one, which was undoubtedly affected by polymerization and formation of high-molecular-weight compounds including carbon–carbon and carbon–oxygen–carbon bridges between fatty acids [25, 26].

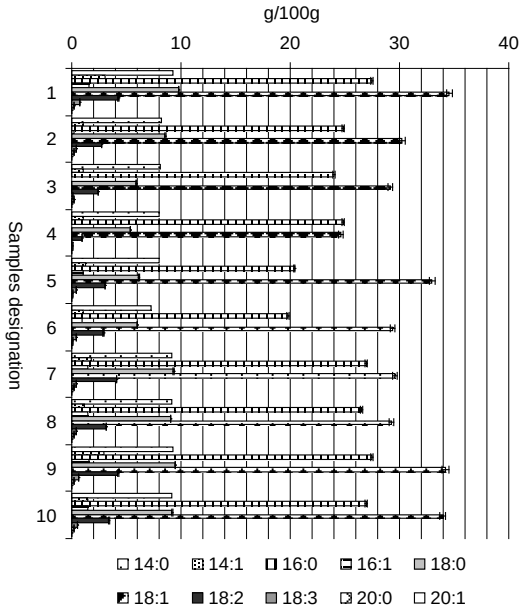

Fig. 6. Fatty acid contents in butter stored under different conditions and heated at +120°C (designations as in Table 1). 14:0, myristic acid; 14:1, myristicoleic acid; 16:0, palmitic acid; 16:1, palmitoleic acid; 18:0, stearic acid; 18:1, oleic acid; 18:2, linoleic acid; 18:3, α-linolenic acid; 20:0, arachidonic acid; 20:1, eicosenoic acid.

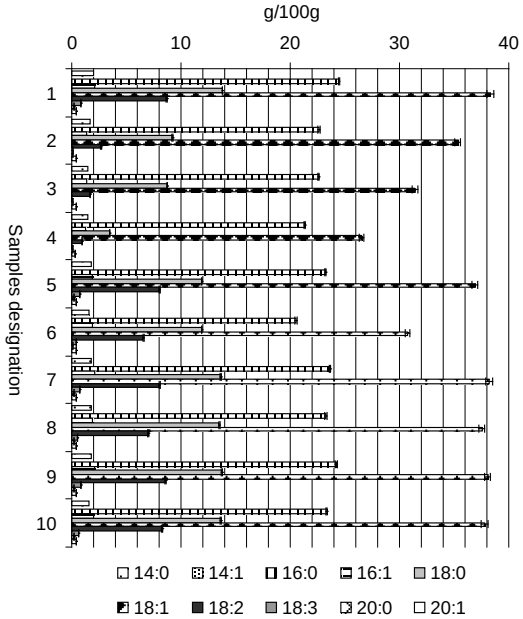

Fig. 7. Fatty acid contents in porcine lard stored under different conditions and heated at +120°C (designations as in Table 1). 14:0, myristic acid; 14:1, myristicoleic acid; 16:0, palmitic acid; 16:1, palmitoleic acid; 18:0, stearic acid; 18:1, oleic acid; 18:2, linoleic acid; 18:3, α-linolenic acid; 20:0, arachidonic acid; 20:1, eicosenoic acid.

As follows from the data in Figs. 1-7 related to the high temperature treatment, the most positive composition of fatty acids was recorded in butter, with loss of fatty acids at about 26.0% after 288 h of heating; in olive oil with loss of about 28.4%; in rapeseed oil with loss of about 33.8%; and in porcine lard with loss of about 35.4% after 288 h of heating. Margarine was characterized by about 47.5% decrease in fatty acids concentration after 288-h heating.

The obtained results confirm the fact that among others, the fatty acid composition and the presence of additional substances (water) and natural antioxidants (phenolic compounds, tocopherols, squalen) determine fat's stability [27–29]. It is well known that fats rich in saturated fatty acids (animal-origin fats) are a group that is best resistant to oxidation, whereas plant-origin oils containing a high percentage of oleinic acid (one double bond) are good for frying, because they are not so susceptible to oxidation as sunflower or soybean oils (rich in linolic acid) [30].

Summarizing, the GC technique with internal standard is suitable for evaluation of the quantitative changes occurring in fatty acids of fats being heated and stored. Furthermore, introducing that method as an alternative in fast qualitative assessment of edible fats is proposed after working out the detailed reference norms, which is going to be the subject of a further study.

CONCLUSIONS

1. Results indicate that the semi-quantitative GC method giving only the percentage of particular fatty acids assuming that their sum is 100%, does not supply correct information for evaluation of changes occurring in stored and heated fats.

2. It was found that the GC method with internal standard is appropriate for assessing the quantitative changes in fatty acids in heated and stored fats.

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ГАЗ-ХРОМАТОГРАФСКИ АНАЛИЗ ПРИ ОПРЕДЕЛЯНЕТО НА ИЗМЕНЕНИЯТА В СЪДЪРЖАНИЕТО НА МАСТНИ КИСЕЛИНИ В ИЗБРАНИ МАЗНИНИ ПО ВРЕМЕ НА СЪХРАНЕНИЕ И НАГРЯВАНЕ

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(Резюме)

Целта на настоящата работа е сравнението на количествените промени в състава на мастни киселини в избрани мазнини (от соя, слънчоглед, рапица, зехтин, както и маргарин, масло и свинска мас) при тяхното съхранение и нагряване. Сравнението е като абсолютни количества (g 100 g^{-1} по метода на стандартната добавка) с тяхните относителни количества след сумиране на анализираните компоненти към 100% (вътрешен метод на нормализация). Експерименталните данни разкриват големи разлики между количествените резултати за определени групи киселини (g 100 g^{-1}) и като процентно съдържание. Общо взето, получените резултати показват, че дългосрочното съхраняване при стайна или повишена температура води до намаляване на съдържанието на наситени (SFA) и ненаситени (MUFA и PUFA) (g 100 g^{-1} проба) във фракцията от триглицеридите. Сравнението на абсолютни и относителни съдържания показва, че по-достоверни резултати може да се постигнат само при използването на аналитична техника, основана на вътрешната стандартна добавка или други еквивалентни методи, даващи пълно количествено представяне на абсолютните количества.