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Research Article

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Determination of Percentage of Laurel Oil in Laurel Soap Using Capillary Gas Chromatographic Analysis

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Abstract

A new analytical method for the quantitative determination of laurel oil in laurel soap by using capillary gas chromatography for detect fraud in the manufacture of laurel soap, which is widely spread in Syria, especially in the city of Aleppo, and is exported to various parts of the world was applied. Assuming that the minimum amount of lauric acid in laurel oil is 20%, provided that the percentage of myristic acid does not exceed 1% (will single out a new research later). Regression equations and correlation coefficient were as the follows: y = 0.2026x + 0.0047 (R^2=0.9996), where y: percentage of lauric acid in fatty acid excreted from soap, and x: percentage of laurel oil in soap.

Keywords: Laurel soap, Fatty acids, percentage of laurel oil in soap, capillary GC analysis.

Introduction

Laurel oil also known as Mediterranean bay laurel, is widely grown in Turkey, Greece, Italy, Spain, Portugal, France, Syria, Morocco, Algeria, Mediterranean Islands, and California. The health benefits of using laurel soap for skin and hair, according to Ohmymag and Healthline were shown. The primary property of Laurel soap (Aleppo soap as it is at Ohmymag and Healthline) is to deeply moisturize the skin [1,2]. Recommended by most dermatologists, thanks to its natural composition, Aleppo soap is suitable for all skin types, whether healthy or problematic. Aleppo soap can help relieve itching for those suffering from psoriasis, acne, and eczema because it will help hydrate the skin. The combination of olive oil and bay laurel oil will help your skin regain suppleness and softness, giving skin a more youthful look. It can also serve as an anti-dandruff shampoo, helping to moisturize the scalp to limit flaking. Placed on the face while allowing it to dry, it makes an excellent mask to minimize imperfections and

rejuvenate tired complexions. Finally, Aleppo soap makes a perfect shaving foam because it facilitates the gliding of the razor and hydrates skin that has been subjected to razor burn. Laurel soap contributes to treating these problems through: 1- Reduces hair loss. 2- It works to give the hair a shiny and healthy appearance. 3- Moisturizing the hair. 4- Eliminates dandruff. 5- It provides the necessary nourishment to the scalp and hair follicles. 6- Improves hair breakage. 7- Helps extend hair. 8- It works to give hair density as a result of nourishing the scalp well. 9- Reduces the appearance of white hair [1- 12].

The effect of ripening time of the laurel fruits (from the region of Kassab in Syria) between October to December on the chemical composition of fatty acids in laurel oil extracted from the fruits (green, brown and black) by hexane using gas chromatographic analysis after converting fatty acids to methyl esters (FAMEs) was studied. It was found that the ratios of fatty acids change with the time of growth and with the maturation of the fruits [13]. The effect of ripening time of the laurel fruits between October to December on the chemical composition of essential oils in laurel oil extracted from the fresh fruits (green, brown and black) by hexane using GC analysis was studied. The ratios of essential oils change with the time of growth and with the maturation of the fruits. Analysis by GC and GC-MS of the essential oils has allowed to identify 26 compounds representing 99.4% of the total content were directly identified after extracting laurel oil [14].

The Syrian national specifications No. 377 [15] for laurel soap, No. 139 [16] for soap and No. 2217 [17] for cosmetic soap specified the necessary conditions for each type of soap.

In the present work, determine laurel oil in laurel soap by using capillary GC analysis to detect fraud in the manufacture of laurel soap (provided that the percentage of myristic acid does not exceed 1%, will single out a new research later) was applied.

Materials and Methods

Instruments and apparatus

A Shimadzu GC-2010 gas chromatograph with capillary column (TRB-WAX 0.5 µm, 30 m ×0.32 mm, Serial: N2068586), auto injecter-AOC-20i and FID detector were used. For microwave digestion of the samples, a high performance microwave digestion apparatus MLS-1200 MEGA with EM-30 unit (Milestone GmbH) was used. An ultrasonic processor model Power sonic 405 was used to sonicine the sample solutions. The diluter pipette model DIP-1 (Shimadzu), having 100 µL sample syringe and five continuously adjustable pipettes covering a volume range from 10 to 5000 µL (model Piptman P, GILSON). Centrifuge (Centurion Scientific Ltd., Model: K2080-Manufactured in the United Kingdom) was used for the preparation of the experimental solutions. SARTORIUS TE64 electronic balance was used for weighing the samples.

Reagents

Hexane, methanol, NaOH, KOH, CH_3COOH (extra pure) were purchased from Merck. Standard of fatty acids (FAME 16 Mix, Cat. No. 722320) was purchased from MACHEREY-NAGEL GmbH & Co. KG, Neumann-Neander-Str. 6-8, D-52355 Duren, Germany. Two standards of essential oils CAN-TERP-MIX 1 & 2, which each contain 21 compounds by focus each 100 $\mu g.mL^{-1}$. Laurel oil (Lauric acid 20.017%, Myristic acid 0.542%, Palmitic acid 16.706%, Oleic acid 36.289%, Linoleic acid 23.990%, Arachidonic acid 0.885% and Linolenic acid 0.548%). Olive oil (Lauric acid 0.0%, Myristic acid 0.002%, Palmitic acid 20.660%, Oleic acid 73.590%, Linoleic acid 8.935%, Arachidonic acid 0.918% and Linolenic acid 0.630%).

Samples preparation

The fruits of the laurel were collected from Kassab area in Syria. Extract the oil with hexane [3]. Studied samples of soap were prepared according to the following: from pure olive oil, from pure laurel oil and from mixture of olive oil and laurel oil in the following proportions: 2.5%, 5.0%, 10.0%, 15.0%, 20.0% and 25.0% laurel oil. It was left in a suitable atmosphere for 36 days. When the tests were conducted on them after adding a quantity of concentrated acetic acid ranging from 0.2 to 1.1 g and heating to the melting point, and converting fatty acids to methyl esters (FAMEs). It was found that the best amount of acetic acid was 0.5 g.

Results and Discussion

Analytical procedure

Using gas chromatographic analysis for studied samples of soap after converting fatty acids to methyl esters (FAMEs) was studied. Programmed column temperature 80°C for 5 min and then increase it to 230°C with increasing temperature rate 10°C/min, FID, flow rate of N_2 carrier gas 1.7 $mL.min^{-1}$, the injection volume 1 µL with split injection mode 1:10, injected port temperature 250°C, and temperature of FID 250°C were applied.

The chemical composition of fatty acids in soap:

Fatty acids were determined in soap samples prepared from (Olive oil only, laurel oil only, and from the mentioned mixtures from 2.5% to 25.0% of laurel oil), see Figures 1-8 and Tables 1-8.

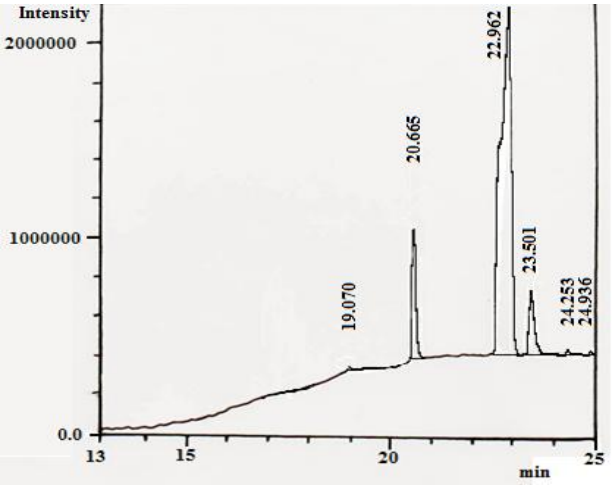

Fig. 1. Gas chromatographic analysis of soap from olive oil only (Programmed column temperature 80°C for 5 min and then increase it to 230°C with increasing temperature rate 10°C/min, FID, flow rate of N_2 carrier gas 1.7 $mL.min^{-1}$, the injection volume 1 µL with split injection mode 1:10, injected port temperature 250°C, and temperature of FID 250°C).

Table 1. The fatty acid components of soap from olive oil only using GC analysis.

Name	Ret. Time, min	Concentrations %
Lauric Acid, **C12**	17.472	0.000
Myristic Acid, **C14**	19.070	0.001
Palmitic Acid, **C16**	20.665	14.918
Oleic Acid, **C18:1**	22.962	73.588
Linoleic Acid, **C18:2**	23.501	8.933
Arachidonic Acid, **C20**	24.253	0.916
Linolenic Acid, **C18:3**	24.936	0.628
Total		**98.984**
Saturated fatty acids, **SFA**	**15.835**	
Unsaturated fatty acids, **USFA**	**83.149**	

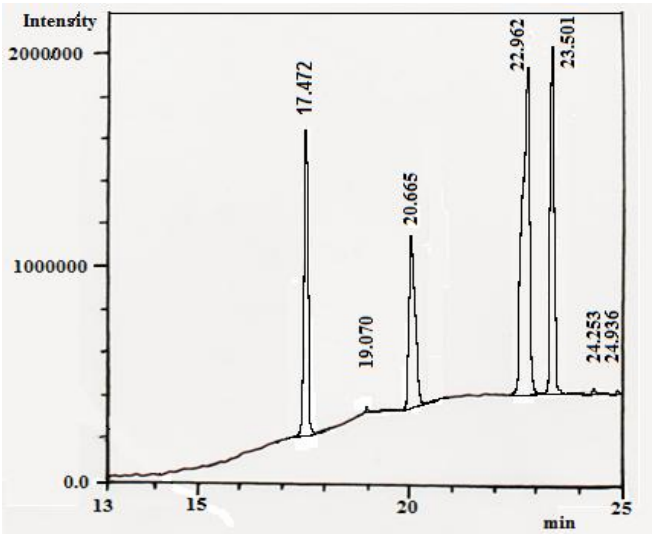

Fig. 2. Gas chromatographic analysis of soap from laurel oil only (Programmed column temperature 80°C for 5 min and then increase it to 230°C with increasing temperature rate 10°C/min, FID, flow rate of N_2 carrier gas 1.7 $mL.min^{-1}$, the injection volume 1 µL with split injection mode 1:10, injected port temperature 250°C, and temperature of FID 250°C).

Table 2. The fatty acid components of soap from laurel oil only using GC analysis.

Name	Ret. Time, min	Concentrations%
Lauric Acid, **C12**	17.472	20.008
Myristic Acid, **C14**	19.070	0.549
Palmitic Acid, **C16**	20.665	16.714
Oleic Acid, **C18:1**	22.962	36.285
Linoleic Acid, **C18:2**	23.501	23.991
Arachidonic Acid, **C20**	24.253	0.880
Linolenic Acid, **C18:3**	24.936	0.550
Total		**98.977**
Saturated fatty acids, **SFA**	**38.151**	
Unsaturated fatty acids, **USFA**	**60.826**	

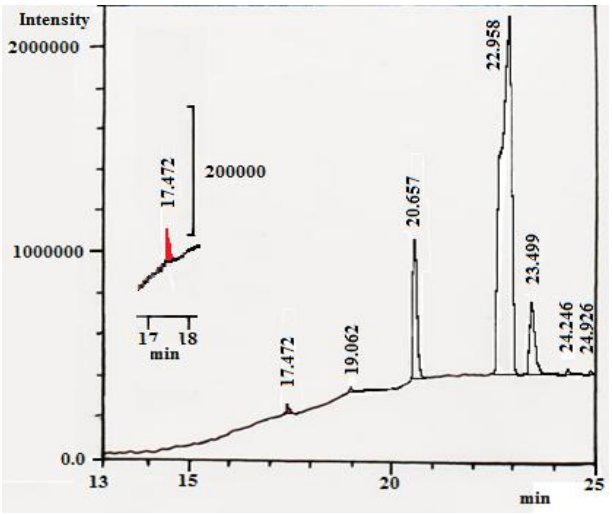

Fig. 3. Gas chromatographic analysis of soap contents 2.5% laurel oil (Programmed column temperature 80°C for 5 min and then increase it to 230°C with increasing temperature rate 10°C/min, FID, flow rate of N_2 carrier gas 1.7 $mL.min^{-1}$, the injection volume 1 µL with split injection mode 1:10, injected port temperature 250°C, and temperature of FID 250°C).

Table 3. The fatty acid components of soap contents 2.5% laurel oil using GC analysis.

Name	Ret. Time, min	Concentrations%
Lauric Acid, **C12**	17.472	0.519
Myristic Acid, **C14**	19.062	0.014
Palmitic Acid, **C16**	20.657	14.962
Oleic Acid, **C18:1**	22.958	72.655
Linoleic Acid, **C18:2**	23.499	9.309
Arachidonic Acid, **C20**	24.246	0.915
Linolenic Acid, **C18:3**	24.926	0.626
Total		**99.000**
Saturated fatty acids, **SFA**	**16.410**	
Unsaturated fatty acids, **USFA**	**82.590**	

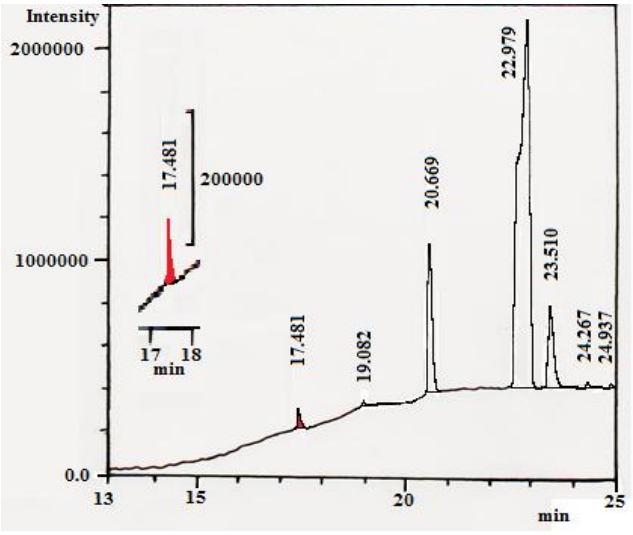

Fig. 4. Gas chromatographic analysis of soap contents 5.0% laurel oil (Programmed column temperature 80°C for 5 min and then increase it to 230°C with increasing temperature rate 10°C/min, FID, flow rate of N_2 carrier gas 1.7 $mL.min^{-1}$, the injection volume 1 µL with split injection mode 1:10, injected port temperature 250°C, and temperature of FID 250°C).

Table 4. The fatty acid components of soap contents 5.0% laurel oil using GC analysis.

Name	Ret. Time, min	Concentrations%
Lauric Acid, **C12**	17.481	0.981
Myristic Acid, **C14**	19.082	0.028
Palmitic Acid, **C16**	20.669	15.007
Oleic Acid, **C18:1**	22.979	71.722
Linoleic Acid, **C18:2**	23.510	9.685
Arachidonic Acid, **C20**	24.267	0.914
Linolenic Acid, **C18:3**	24.937	0.624
Total		**98.961**
Saturated fatty acids, **SFA**	**16.930**	
Unsaturated fatty acids, **USFA**	**82.031**	

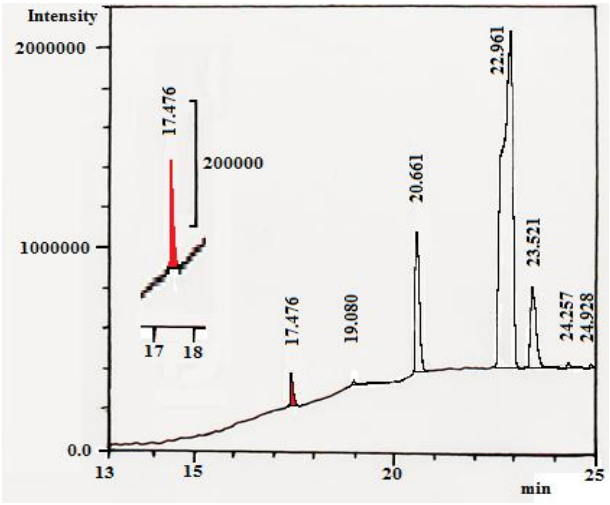

Fig. 5. Gas chromatographic analysis of soap contents 10.0% laurel oil (Programmed column temperature 80°C for 5 min and then increase it to 230°C with increasing temperature rate 10°C/min, FID, flow rate of N_2 carrier gas 1.7 $mL.min^{-1}$, the injection volume 1 µL with split injection mode 1:10, injected port temperature 250°C, and temperature of FID 250°C).

Table 5. The fatty acid components of soap contents 10.0% laurel oil using GC analysis.

Name	Ret. Time, min	Concentrations%
Lauric Acid, **C12**	17.476	2.054
Myristic Acid, **C14**	19.080	0.055
Palmitic Acid, **C16**	20.661	15.097
Oleic Acid, **C18:1**	22.961	69.857
Linoleic Acid, **C18:2**	23.521	10.438
Arachidonic Acid, **C20**	24.257	0.912
Linolenic Acid, **C18:3**	24.928	0.620
Total		**99.033**
Saturated fatty acids, **SFA**	**18.118**	
Unsaturated fatty acids, **USFA**	**80.915**	

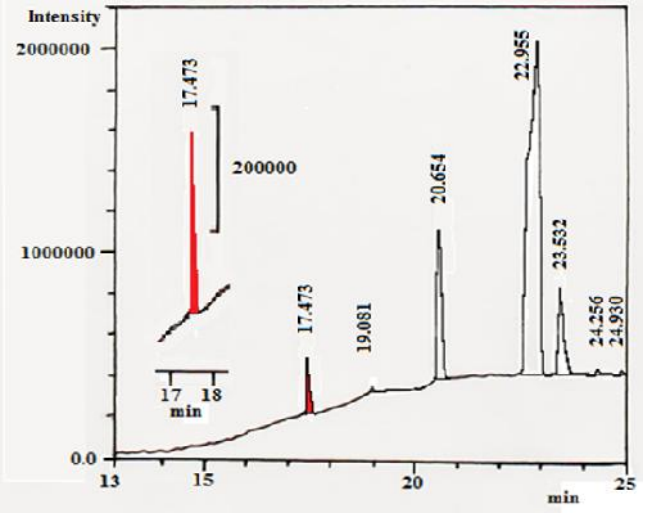

Fig. 6. Gas chromatographic analysis of soap contents 15.0% laurel oil (Programmed column temperature 80°C for 5 min and then increase it to 230°C with increasing temperature rate 10°C/min, FID, flow rate of N_2 carrier gas 1.7 mL.min^{-1}, the injection volume 1 µL with split injection mode 1:10, injected port temperature 250°C, and temperature of FID 250°C).

Table 6. The fatty acid components of soap contents 15.0% laurel oil using GC analysis.

Name	Ret. Time, min	Concentrations%
Lauric Acid, **C12**	17.473	3.106
Myristic Acid, **C14**	19.081	0.083
Palmitic Acid, **C16**	20.654	15.187
Oleic Acid, **C18:1**	22.955	67.992
Linoleic Acid, **C18:2**	23.532	11.191
Arachidonic Acid, **C20**	24.256	0.910
Linolenic Acid, **C18:3**	24.930	0.616
Total		**99.085**
Saturated fatty acids, **SFA**	**19.286**	
Unsaturated fatty acids, **USFA**	**79.799**	

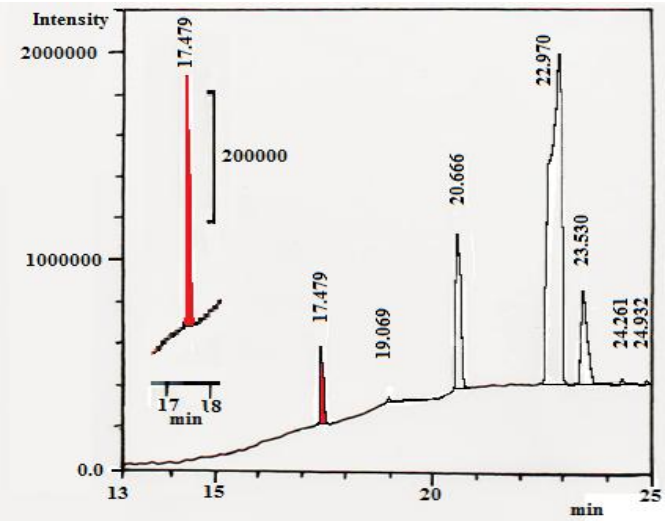

Fig. 7. Gas chromatographic analysis of soap contents 20.0% laurel oil (Programmed column temperature 80°C for 5 min and then increase it to 230°C with increasing temperature rate 10°C/min, FID, flow rate of N_2 carrier gas 1.7 mL.min^{-1}, the injection volume 1 µL with split injection mode 1:10, injected port temperature 250°C, and temperature of FID 250°C).

Table 7. The fatty acid components of soap contents 20.0% laurel oil using GC analysis.

Name	Ret. Time, min	Concentrations%
Lauric Acid, **C12**	17.479	4.001
Myristic Acid, **C14**	19.069	0.110
Palmitic Acid, **C16**	20.666	15.277
Oleic Acid, **C18:1**	22.970	66.127
Linoleic Acid, **C18:2**	23.530	11.944
Arachidonic Acid, **C20**	24.261	0.908
Linolenic Acid, **C18:3**	24.932	0.612
Total		**98.979**
Saturated fatty acids, **SFA**	**20.296**	
Unsaturated fatty acids, **USFA**	**78.683**	

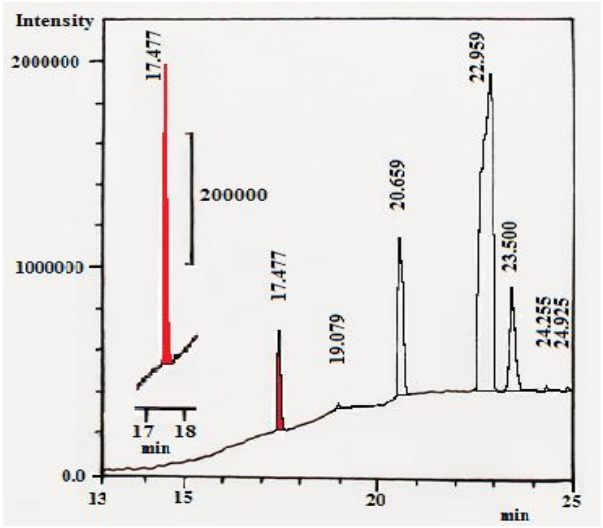

Fig. 8. Gas chromatographic analysis of soap contents 25.0% laurel oil (Programmed column temperature 80°C for 5 min and then increase it to 230°C with increasing temperature rate 10°C/min, FID, flow rate of N_2 carrier gas 1.7 $mL.min^{-1}$, the injection volume 1 µL with split injection mode 1:10, injected port temperature 250°C, and temperature of FID 250°C).

Table 8. The fatty acid components of soap contents 25.0% laurel oil using GC analysis.

Name	Ret. Time, min	Concentrations%
Lauric Acid, **C12**	17.477	5.076
Myristic Acid, **C14**	19.079	0.138
Palmitic Acid, **C16**	20.659	15.367
Oleic Acid, **C18:1**	22.959	64.262
Linoleic Acid, **C18:2**	23.500	12.697
Arachidonic Acid, **C20**	24.255	0.907
Linolenic Acid, **C18:3**	24.925	0.608
Total		**99.055**
Saturated fatty acids, **SFA**	**21.488**	
Unsaturated fatty acids, **USFA**	**77.567**	

The previous results are summarized in Table 9; Whereas soap made from olive oil only or made from laurel oil percentage of less than 5% is considered an soap (not laurel soap); according to the Syrian national specifications No. 377 and No. 139 [3, 4]. Figure 9 showed that the calibration curves for the ratio of laurel oil in soap by gas chromatographic analysis and regression equations and correlation coefficient were as the follows: $y = 0.2026x + 0.0047$ (R^2=0.9996), where y: concentration % of lauric acid in soap, and x: percentage of laurel oil in soap.

Table 9. The fatty acid components of soap contents laurel oil only or 0.0% to 25.0% laurel oil using gas chromatographic analysis with a detector FID.

Name	$\bar{t}$, Ret. Time, min	100 %, Laurel Oil	100 %, Olive Oil	The ratio of concentrations laurel oil in the soap					
				2.5%	5.0%	10.0%	15.0%	20.0%	25.0%
			Soap (According to S.N.S. N° " 139")	Laurel Soap (According to S.N.S. N° " 377" for Laurel Soap)					
Lauric Acid, **C12**	17.472	20.008	0.000	0.519	0.981	2.054	3.106	4.001	5.076
Myristic Acid, **C14**	19.062	0.549	0.001	0.014	0.028	0.056	0.084	0.110	0.138
Palmitic Acid, **C16**	20.657	16.714	14.918	14.962	15.007	15.096	15.187	15.277	15.367
Oleic Acid, **C18:1**	22.958	36.285	73.588	72.655	71.722	69.857	67.992	66.127	64.262
Linoleic Acid, **C18:2**	23.499	23.991	8.933	9.309	9.685	10.438	11.191	11.944	12.697
Arachidonic Acid, **C20**	24.255	0.880	0.916	0.915	0.914	0.912	0.910	0.908	0.907
Linolenic Acid, **C18:3**	24.925	0.550	0.628	0.626	0.624	0.620	0.616	0.612	0.608
Total		**98.977**	**98.984**	**99.000**	**98.961**	**99.033**	**99.085**	**98.979**	**99.055**
Saturated fatty acids, SFA		**38.151**	**15.835**	**16.410**	**16.930**	**18.118**	**19.286**	**20.296**	**21.488**
Unsaturated fatty acids, USFA		**60.826**	**83.149**	**82.590**	**82.031**	**80.915**	**79.799**	**78.683**	**77.567**

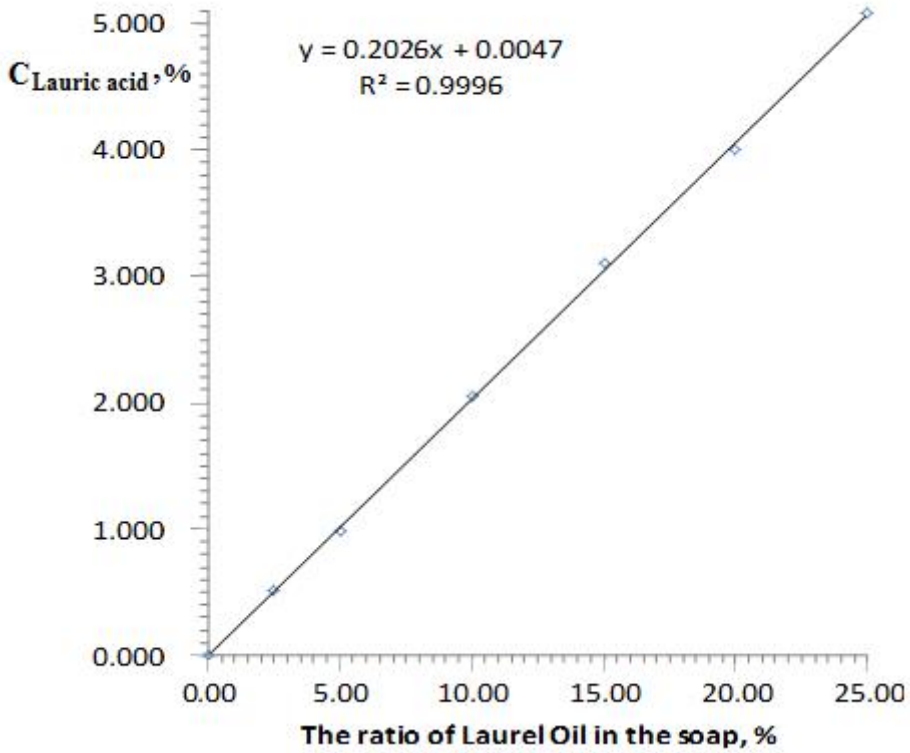

Fig. 9. Calibration curve of the proportion of laurel oil in the oil extracted from laurel soap, depending on the percentage of lauric acid in it by GC analysis (Programmed column temperature 80°C for 5 min and then increase it to 230°C with increasing temperature rate 10°C/min, FID, flow rate of N_2 carrier gas 1.7 $mL.min^{-1}$, the injection volume 1 µL with split injection mode 1:10, injected port temperature 250°C, and temperature of FID 250°C).

Based on the previous results, samples of laurel soap taken from the local market in Aleppo - Syria were identified and classified according to the Syrian standard specifications (S.N.S.) and the degree of their quality and validity, as shown in Table 10.

Table 10. Classification of Laurel soap taken from the local market in Aleppo - Syria classified according to the Syrian standard specifications (S.N.S.) and regression equation: y=0.2026x+0.0047, where y- Concentration% of lauric acid in soap, x- Laurel oil in soap (Provided that myristic acid is does not exceed 1%).

Samples	Myristic acid	Y, Concentrations % of Lauric acid in soap	X, Laurel oil in soap	No of S.N.S.	Classification of soap	Quality of soap	Validity as Laurel soap
A	0.026%	1.04	5.11%	377	Laurel soap	Second degree	Valid
B	0.225%	8.20	40.45%	377	Laurel soap	First degree	Valid
C	0.017%	0.62	3.04%	139	Soap	-	Invalid
D	0.008%	0.28	1.36%	139	Soap	-	Invalid
E	0.020%	0.36	1.75%	139	Soap	-	Invalid
F	0.094%	3.41	16.81%	377	Laurel soap	First degree	Valid
G	2.531%	6.13	-	2217	Cosmetic soap	-	Invalid
H	0.140%	5.06	24.95%	377	Laurel soap	First degree	Valid
I	0.030%	1.24	6.10%	377	Laurel soap	Second degree	Valid
J	0.195%	7.04	34.73%	377	Laurel soap	First degree	Valid
K	2.872%	7.16	-	2217	Cosmetic soap	-	Invalid
L	2.043%	5.42	-	2217	Cosmetic soap	-	Invalid
M	0.192%	7.01	34.58%	377	Laurel soap	First degree	Valid
N	0.116%	3.96	19.52%	377	Laurel soap	First degree	Valid
O	0.013%	0.41	2.00%	139	Soap	-	Invalid
P	0.037%	1.31	6.44%	377	Laurel soap	Second degree	Valid
Q	3.100%	8.00	-	2217	Cosmetic soap	-	Invalid

Conclusion

Determine laurel oil in laurel soap by using capillary gas chromatographic analysis to detect fraud in the manufacture of laurel soap, which is widely spread in Syria, especially in the city of Aleppo, and is exported to various parts of the world was applied. Assuming that the minimum amount of lauric acid in laurel oil is 20% provided that the percentage of myristic acid does not exceed 1% (Will single out a new research later). Regression equations and correlation coefficient were as the follows: y = 0.2026x + 0.0047 (R^2=0.9996), where y: percentage of lauric acid in fatty acid excreted from soap, and x: percentage of laurel oil in soap.

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