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Kishk – a dried fermented milk/cereal mixture. 1. Composition of gross components, carbohydrates, organic acids and fatty acids

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Abstract — An investigation of the compositional quality of 25 commercial samples of Lebanese Kishk was undertaken. The results of chemical analysis ($g \cdot 100\ g^{-1}$ on dry matter basis [DMB]) of the samples were within the following ranges: protein, 14.7–21.4; fat, 2.6–11.5; ash, 4.1–9.3; and carbohydrates, 61.0–76.8. The moisture and salt contents ranged between 6.8 and 10.8, and 0.95 and 4.48 $g \cdot 100\ g^{-1}$, respectively, and the pH averaged 3.8. The carbohydrate content calculated by difference (total solids – [protein + fat + ash]) was slightly greater than by the summation method (galactose + lactose + fibre + starch). Lactic and acetic acids were identified to be the major organic acids present in the Kishk samples, including an appreciable amount of propionic acid. Principal component analysis (PCA) was used to map the relationships between these samples; PC 1 of the organic acid data accounted for only 37 % of the total variation separating the Kishk samples *high* in propionic, lactic and uric/formic acids, and those with *high* acetic, orotic, pyruvic and citric acid contents. Profiling the fatty acid content in the Kishk samples did not conclusively identify the type of milk used, but the ratios of $C_{4:0}/(C_{6:0} + C_{8:0})$ and $C_{12:0}/C_{10:0}$ provided appropriate mapping of Kishk samples made with goat's milk and possibly adulteration with cow's milk at a level of 20 %. © Inra/Elsevier, Paris.

Kishk / composition / organic acids / fatty acids / carbohydrate fraction

Résumé — Le Kishk - un mélange lait/céréales fermenté et séché. I. Composition globale, sucres, acides organiques et acides gras. La composition de 25 échantillons de Kishk libanais du commerce a été étudiée. La composition globale des échantillons était comprise dans les gammes suivante (en $g \cdot 100\ g^{-1}$ de matière sèche) : protéines 14,7 à 21,4 ; matière grasse 2,6 à 11,5 ; cendres

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4,1 à 9,3 et sucres 61 à 76,8. L'humidité et la teneur en sel allaient respectivement de 6,8 à 10,8 et 0,95 à 4,48 g·100 g^{-1} et le pH était en moyenne de 3,8. La teneur en sucre calculée par la différence (matière sèche – [protéines et matière grasse + cendre]) était légèrement supérieure à la valeur obtenue par addition (galactose + lactose + fibre + amidon). Les acides lactiques et acétiques étaient les principaux acide propionique. Une analyse en composante principale a été utilisée pour étudier les relations entre échantillons : la première composante principale des données des acides organiques contribuait pour seulement 37 % aux variations entre les échantillons à forte teneur en acide propionique, lactique et urique/formique et ceux à forte teneur en acide acétique, orotique, pyruvique et citrique. Le profil en acides gras des échantillons de Kishk ne permettait pas d'identifier de façon concluante le type de lait utilisé, mais les ratios de : $C_{4:0}/(C_{6:0} + C_{8:0})$ et $C_{12:0}/C_{10:0}$ permettaient de dresser une cartographie des échantillons de Kishk produit à partir de lait de chèvre et de mettre en évidence une éventuelle adultération avec du lait de vache à un niveau de 20 %.

Kishk / composition / acide organique / acide gras / glucide

1. INTRODUCTION

A wide range of traditional fermented and dried foods are produced in many countries [30]. Recently, Kurmann et al. [19] compiled an international inventory of fresh and dried fermented milk products. One such product, which is widely produced in the rural regions between the Middle East and the Indian subcontinent, is known as Kishk, Kushuk, Keshkeh or Kichk. This product is normally made from low-fat yoghurt or the buttermilk of churned full-fat yoghurt. The yoghurt is mixed with cereal (commonly known as Burghol when made from parboiled cracked wheat), sun-dried and ground to a powder. This fermented product has been investigated by research groups from different countries in the Middle East, and extensive reviews have been reported by El-Gendy [11], Jandal [15] and Tamime and O'Connor [32].

Some pertinent information is available on the chemical composition, microbiological quality and the nutritional value of commercial and laboratory-made Kishk samples [32]. The differences in these parameters were attributed to: (a) the vagaries of the traditional system employed for the manufacture of Kishk; (b) the different ingredients (cow, goat or sheep milk), and the ratio of cereals to fermented milk which may range between 1:2 to 1:4; and (c) the sanitary conditions observed during the manufacture of the product. A survey by von Taleban and Renner [31] on 135 Iranian Kishk samples confirmed that some of these factors affected the chemical composition of the product.

The Lebanese Kishk is normally made from goat's milk, cow's milk or a mixture of the two. In some rare instances, Kishk may be made from sheep's milk. The product may be manufactured by dairy companies for supermarket retail chains, by granaries or may be homemade. The overall objective of this study was to investigate the chemical composition, microbiological quality and nutritional properties of 25 different samples of Lebanese Kishk. The results of the chemical analysis of Kishk are given in detail here.

2. MATERIALS AND METHODS

2.1. Kishk samples

Twenty-five Kishk samples (~1 kg each) were purchased from different retail outlets (dairy companies, supermarkets, granaries and homemade) in the Bekaa Valley and nearby mountain villages. This is the largest dairy and agriculture region in Lebanon. Some of these samples were pre-packed in cloth or plastic bags, but the majority of the Kishk samples were sold in large sacks or metal or wooden bins.

Neither nutritional labelling nor the type of milk(s) used was evident on any of the prepacked Kishk. However, as much information as could be obtained is presented in *table I*, which identifies the region, source of manufacture and the milk used for Kishk-making.

2.2. Compositional analysis

Fat, nitrogen, and ash and moisture contents were determined according to the methods described in BSI [6–8], respectively. In Lebanon, a ratio of Burghol (one part) and fermented milk (four parts) is normally used for the production of Kishk (S. Najar, personal communication); thus, the protein contents in these samples were calculated as follows:

Protein = $[(4/5 \times 6.38) + (1/5 \times 5.70)] \times$ nitrogen (in effect this means that the multiplication factor becomes 6.244).

The total carbohydrate content was calculated by difference (total solids – [protein + fat + ash]), and by the summation method (galactose + lactose + starch + dietary fibre). The free sugars (glucose, galactose and lactose) were extracted with methanol (85 % v/v) according to the method described by Southgate [29], and determined by high performance liquid chromatography (HPLC). Chromatograph Shimadzu (System 6A; Shimadzu Sp., Warsaw, Poland) with refractive index detector, RID-6A and LiChroCart NH_2 column (Merck Sp., Warsaw, Poland) and mobile phase acetonitrile–water (75:25 v/v) were used. The carbohydrate content of the parboiled cracked wheat (Burghol) and the wheat grains were determined using the method as for the Kishk samples, and only before HPLC analysis, the carbohydrate extracts of the Burghol were purified using column chromatography [23]. The starch and dietary fibre contents in the Kishk samples were determined according to the methods described by Statutory Instrument (SI) [27, 28] and Prosky et al. [25].

The pH in the Kishk samples was measured after rehydration of the powder with water at a ratio of 1:2 to produce a porridge-like consistency similar to the way in which the Kishk is prepared for eating. A portable pH stick metre model Check Mate 90 (Mettler Toledo Ltd., Essex, UK), fitted with a standard combined glass electrode, was used to measure the pH value in the reconstituted Kishk samples.

Salt percentage in Kishk was determined according to the method described in BSI [5], which was based on the principle of the reaction between the sodium chloride and silver nitrate in hot acid to form silver chloride. The difference between the titration of the excess silver nitrate with potassium thiocyanate and the blank was taken to calculate the salt percentage in the Kishk sample, e.g. 1 mL of 0.05 $mol \cdot L^{-1}$ potassium thiocyanate ~ 0.00292 g salt.

2.3. Organic acids

The concentrations of organic acids in different Kishk samples were determined by HPLC (Spectra-Physics system, San Jose, CA, USA) using a modification of the technique reported by Marsili et al. [21]. Separation of orotic and citric acid was achieved by isocratic elution with H_2SO_4 (0.00223 $mol \cdot L^{-1}$, 65 °C, 0.7 $mL \cdot min^{-1}$) on a strong cation exchange resin (Aminex HPX-87H, 300 × 7.8 mm; Bio-Rad Laboratories, Richmond, CA, USA) without prejudice to resolution of pyruvic, lactic, uric, propionic, butyric and hippuric acids [20]. Individual organic acids (orotic, citric, pyruvic, lactic, uric/formic, acetic, butyric and hippuric) were determined using the HPLC method as described by Barrantes et al. [4].

Organic acids were extracted from Kishk (5 g) in a 50 mL beaker using 25 mL water-acetonitrile (1:4 v/v) (analytical grade, BDH Chemicals Ltd., Poole, UK). The extract after filtration through a Whatman No. 1 filter paper (Whatman Ltd., Maidstone, UK) was injected (20 μL) into the HPLC column. The flow rate of the solvent was 0.7 $\mu L \cdot min^{-1}$ at 65 °C and the wavelength in the detector was 220 nm.

The chromatograph was calibrated using an aqueous solution of orotic, citric, pyruvic, lactic, uric, acetic, propionic, acids (analytical grade, BDH Chemical Ltd.) at the following concentrations: 20.4, 1 000, 50, 1 680, 6.39, 880 and 925 $\mu g \cdot mL^{-1}$, respectively.

2.4. Fatty acids

The fatty acid content in the Kishk was determined by gas liquid chromatography (GLC) on the lipid extraction by the Rose-Gottlieb method, according to BSI [9], and as described by Barrantes et al. [4]. The chromatograph used was Model 93 equipped with a flame ionisation detector, S 100183 (AI Scientific Cambridge Ltd., Cambridge, UK) and a Model SP 4290 integrator (Spectra-Physics, San Jose, CA, USA). Sam-

Table I. Classification and identification of Kishk samples bought in Lebanon.

Tableau I. Classification et identification des échantillons de Kishk achetés au Liban.

Region	Code number	Brand name	Source	Milk[a]	Type of pack	Visual assessment		
						Flowability	Particle size	Colour[b]
Bekaa Valley								
Mid-Bekaa (Chtoura, Chteta, Kob-Elias, Zahle, Firzol, Saad Nayel, Dier Al-Ghazal)								
	1	Yalco	S-market	—[c]	Open	Poor	Fine	Cream (M)
	2	Masabki	Dairy	—	Bagged	Good	Fine	Cream (D)
	3	Masabni	Dairy	—	Open	Poor	Fine	Cream (L)
	4	Ghazali	S-market	—	Bagged	Fair	Fine	Off-White
	5	Al-Giser	Granary	—	Bagged	Poor	Fine	Cream (M)
	6	Jarjoura	Dairy	—	Open	Good	Fine	Cream (M)
	7	— —[d]	S-market	—	Open	Good	Fine	Magnolia
	8	Najar	Granary	$^{3/4}$G/$^{1/4}$C	Open	Good	Fine	Cream (L)
	9	Kadami	Granary	G	Open	Good	Fine	Magnolia
	10	— —	Home	G	Open	Good	Grainy	White
	11	Choubas	Granary	C	Open	Good	Fine	Cream (M)
	12	Choubas	Granary	C/G	Open	Good	Gritty	Cream (M)
	13	Choubas	Granary	G	Open	Poor	Fine	Magnolia
	14	— —	Granary	-	Open	Poor	Grainy	Magnolia
North Bekaa (Baalbeck)								
	15	— —	Granary	S	Open	Good	Gritty	Golden
South Bekaa (Hasbaya, Mashgara, Rashaya)								
	16	— —	Home	—	Open	Good	Gritty	Golden
	17	— —	Home	—	Open	Good	Fine	Magnolia
	18	W. Al-Kheir	Granary	—	Bagged	Good	Fine	Cream (L)
Mountains								
Mreyjat, Bouwarej								
	19	Al-Dsouki	S-market	—	Open	Poor	Fine	Cream (D)
	20	Hadwan	Dairy	C	Open	Poor	Fine	Magnolia
	21	Hadwan	Dairy	G	Open	Fair	Fine	Magnolia
	22	Falah	Granary	$^{3/4}$G/$^{1/4}$C	Open	Good	Grainy	Cream (M)
	23	C. Antonious	Home	G	Open	Good	Fine	Magnolia
	24	— —	Dairy	G	Open	Good	Grainy	Cream (L)
	25[e]	— —	—	—	Open	Good	Fine	Off-White

[a] C: cow, G: goat, S: sheep; [b] (M): medium, (D): dark, (L): light; [c] not reported; [d] unknown; [e] possibly made in Syria.

[a] C : vache ; G : chèvre ; S : brebis ; [b] (M): moyen ; (D) : sombre ; (L) : clair ; [c] non indiqué ; [d] inconnu ; [e] peut-être produit en Syrie.

ples were injected by an automatic liquid autosampler (Philips Model PU 4700, PYE Unicam Ltd. Cambridge, UK), fitted with a 1 μL syringe. The temperature programme was 50 °C for 2 min (isothermal), then increasing to 200 °C at a rate of 20 °C·min^{-1} and held at 200 °C for 10 min. Nitrogen gas was used as a carrier at a flow rate of about 20 mL·min^{-1}. The flow rate of H_2 was about 20 mL·min^{-1}.

A set of fatty acid standards, dissolved in diisopropyl ether containing 4 % formic acid (BDH Chemicals Ltd.) was used to calibrate the chromatograph. Response factors were automatically determined by a data processor using the *n*-nonanoic acid ($C_{9:0}$) as internal standard. The chromatograms were quantified by the processor by relating the corrected peak areas to the peak area for $C_{9:0}$.

2.5. Statistical analysis

The data were analysed by univariate (analysis of variance) and multivariate (principal component analysis [PCA]) techniques using the Genstat computer program (copyright 1990, Lawes, Agricultural Trust, Rothamsted Experimental Station, UK).

2.5.1. Sample size

The accuracy and precision of the inferences obtained from any survey can be improved by increasing the sample size (n). The precision of the sample mean ($\bar{x}$) follows a Student's *t*-distribution with $(n-1)$ degrees of freedom and is $s/\sqrt{n}$. The precision of sample standard deviation (s) is more complex. The quantity $(n-1)\,s^2/\sigma^2$ has a chi-squared distribution with $(n-1)$ degrees of freedom, where σ^2 is the population standard deviation. The summary below shows how the sample size (n) will affect the estimates of the mean (μ) and population standard deviation (σ). Ultimately, the sample size was limited by the logistics of transporting the Kishk, and it was decided little would be gained by increasing the sample size much beyond 25.

2.5.2. Data analysis

Since the data originate from a survey rather than a designed experiment, the objectives of the statistical analysis were to summarise the data into simple summary statistics. Univariate exploratory data analysis techniques were used to summarise individual variables. Relationships between measured variables were explored using two multivariate methods, namely matrix plots and PCA. A matrix plot is an array of graphs containing an X–Y plot of each variable against every other variable. The merits of a matrix plot are: (a) simplicity and interpretability; (b) the ability to display multivariate outliers in the data set and (c) the ability to assess curvilinear relationships between any two variables.

The disadvantage of a matrix plot is that each individual plot is unable to portray the multivariate relationships between variables. Principal component (PC) bi-plots overcome the limitation of a matrix plot by plotting cross-sectional areas through the multivariate data set. An X–Y plot of the first two PCs is defined by the largest cross section through the data. Unless otherwise stated, the PC bi-plots were produced from the correlation matrix of the original variables. Whenever possible, patterns and clusters of data points observed during the analysis were related back to the known origins of the sample.

3. RESULTS AND DISCUSSION

3.1. Chemical composition of Kishk samples

The average chemical composition of the 25 samples of Kishk is shown in *table II*.

No. of samples	95 % confidence interval for (mean) (*t*-statistic)	95 % confidence interval for (σ) (χ^2 statistic)
	$\bar{x} - t_{(n-1)(\alpha/2)}\, s \le \mu\, \bar{x} + t_{(n-1)(1-\alpha/2)}\, s$	$\sqrt{_{(n-1)}/\chi^2_{(n-1)(1-\alpha/2)}}\, s \le \sigma \le \sqrt{_{(n-1)}/\chi^2_{(n-1)(1-\alpha/2)}}\, s$
12	$\bar{x}$ –0.64 *s* ≤ μ ≤ $\bar{x}$ +0.64 *s*	0.71 *s* ≤ σ ≤ 1.70 s
25	$\bar{x}$ –0.41 *s* ≤ μ ≤ $\bar{x}$ +0.41 *s*	0.78 *s* ≤ σ ≤ 1.39 s
50	$\bar{x}$ –0.28 *s* ≤ μ ≤ $\bar{x}$ +0.28 *s*	0.84 *s* ≤ σ ≤ 1.25 s
100	$\bar{x}$ –0.20 *s* ≤ μ ≤ $\bar{x}$ +0.20 *s*	0.88 *s* ≤ σ ≤ 1.16 s

Table II. Chemical composition and acidity of different samples of Lebanese commercial Kishk.
Tableau II. Composition et acidité de différents échantillons de Kishk libanais du commerce.

Component	Minimum	Maximum	Mean
Proximal composition			
Total solids ($g \cdot 100\ g^{-1}$)	89.23	93.25	91.63
Protein[a]	14.72	21.44	17.75
Fat[a]	2.43	11.52	6.39
Carbohydrates			
Total[b]	61.02	76.73	68.75
Starch[a]	42.60	59.26	50.62
Dietary fibre[a]	6.51	12.24	9.32
Galactose[a]	2.83	10.43	6.11
Lactose[a]	0.56	2.86	1.43
Total[c]	57.13	76.77	67.49
Ash[a]	4.06	9.30	7.03
Salt[d]	0.05	4.48	2.84
pH	3.58	4.12	3.77
Organic acids ($\mu g \cdot g^{-1}$)			
Orotic[e]	1.65	9.54	4.81
Citric[f]	29.00	174.00	68.54
Pyruvic	16.00	81.00	38.16
Lactic ($mg \cdot g^{-1}$)	18.75	43.87	32.47
Uric/formic	13.00	36.00	23.96
Acetic	401.00	856.00	586.56
Propionic	917.00	7452.00	3551.68
Fatty acids (% w/w)			
$C_{4:0}$	1.25	6.66	4.44
$C_{6:0}$	1.25	4.34	3.22
$C_{8:0}$	1.21	3.31	2.30
$C_{10:0}$	2.20	9.18	5.73
$C_{12:0}$	2.02	4.28	3.25
$C_{14:0}$	6.23	11.64	9.98
$C_{16:0}$	27.10	35.80	30.72
$C_{18:0}$	7.12	12.87	9.66
$C_{20:0}$	0.45	1.51	0.77
$C_{14:1}$	1.67	4.09	2.36
$C_{16:1}$	1.16	2.65	1.63
$C_{18:1}$	11.67	25.61	19.26
$C_{18:2}$	2.18	20.89	6.70
$C_{18:3}$	0.16	1.51	0.45

[a] Data were calculated on dry matter basis; [b] carbohydrate = total solids – (protein + fat + ash) or by difference method; [c] carbohydrates = (starch + dietary fibre + galactose + lactose) or by the summation method; [d] salt was calculated from chloride titration; [e] data for sample 24, which was substantially different (17.76 $\mu g \cdot g^{-1}$), was not included; [f] data for sample 25, which was substantially different (504 $\mu g \cdot g^{-1}$), was not included. Results are the average of two determinations performed on each sample.

[a] Données calculées par rapport à la matière sèche ; [b] sucre = matière sèche – (protéines + matière grasse + cendre), ou par la méthode des différences ; [c] la teneur pour l'échantillon 24, [d] qui était considérablement différente (17,76 $\mu g \cdot g^{-1}$) ; [e] n'a pas été incluse dans le calcul ; [f] la teneur pour l'échantillon 25, qui était considérablement différente (504 $\mu g \cdot g^{-1}$), n'a pas été incluse dans le calcul. Les résultats sont la moyenne de deux déterminations par échantillon.

Since the moisture content of these samples ranged between 6.75 and 10.77 g·100 g^{-1}, it was decided to convert the analytical data to a dry matter basis (DMB). Therefore, the total solids (g·100 g^{-1}), protein, fat and ash contents (g·100 g^{-1} [DMB]) ranged between 89.23 and 93.25, 14.72 and 21.44, 2.43 and 11.52, and 4.06 and 9.30, respectively. The variations in these levels could be attributed to many factors such as: (a) the preparation of Kishk and the added ingredients used; (b) the time used for drying the fermented milk/Burghol mixture in the sun; (c) the type or combinations of milk used for fermentation; (d) the efficiency of fat separation after churning the fermented milk; and (e) the amount of salt that is added.

The protein content (g·100 g^{-1} [DMB]) of these Kishk samples averaged 17.75. A ratio of fermented milk to Burghol of 4:1 had been assumed in the calculation; however, if this ratio had been 3:1 or 2:1, then the range of the protein values would be between 14.66 and 21.30 or 14.52 and 21.11, respectively. Although the range in the calculated protein (2:1 to 4:1) found in these samples is substantial, the difference does not depend heavily on which ratio is assumed to be correct. A ratio of 4:1 is assumed to have been used throughout in Kishk-making.

By comparing the results of the chemical analyses shown in *table II* with other published analytical data of commercial Kishk samples [1, 12, 13, 22, 32], it is evident that the Lebanese Kishk samples had a protein content ≥ 16 g·100 g^{-1}. Morcos et al. [22] reported an average protein content of 23.5 g·100 g^{-1} of the Egyptian Kishk samples analysed, but their data totaled 105.3 % and this may suggest inaccuracy in their analyses.

The fat content of the majority of the Lebanese Kishk samples was ≤ 9 g·100 g^{-1} (DMB) with the exception of sample numbers 2, 16 and 24 (*table II*), and these values were similar to fat contents of Kishks made in different countries of the Middle East [32]. The variation in the fat contents of these Kishk samples could be attributed to the efficiency of fat separation from the milk before the production of yoghurt, to the efficiency of recovery of the butter granules from the churned fermented milk, or to the ratio of fermented milk/cereal mixture used. Products similar to Kishk are made in Cyprus (Trahana) and in Saudi Arabia (Madeer and Oggt) which may contain ≥ 15 g fat·100 g^{-1}. The Trahana product is made with sheep's milk while the Saudia Arabian products do not contain the Burghol cereal [32].

The salt content in Kishk samples ranged between 0.95 and 4.48 g·100 g^{-1} (DMB) where the average (2.84) is similar to the data reported by El-Gendy [11] for Egyptian Kishk samples. Higher salt levels (e.g. 4.15–10.76 g·100 g^{-1}) have been reported in commercial Kishk samples made in Egypt [3, 12]. The wide variation in the salt content in Kishk samples is due to the different amounts of added salt during the preparation of Kishk. It is most likely that the salt is used to control the metabolic activity of the microflora in the yoghurt/cereal mixture during the secondary fermentation stage, to extend the shelf-life of the product and contribute to the taste of Kishk [32].

The pH values of the various Kishk samples are shown in *table II*, and averaged 3.77, which reflect the activity of the lactic acid bacteria during Kishk-making.

The carbohydrate contents of the different Kishk samples were calculated by different methods, and they are shown in *table II* along with the overall means. The results of carbohydrate analysis were within the following ranges (g·100 g^{-1} [DMB]): 2.8–10.4 galactose, 0.6–2.9 lactose, 42.6–59.3 starch and 6.5–12.2 dietary fibre including β-glucan. The total content of carbohydrate calculated by difference *vis-a-vis* analysed were within the following ranges (g·100 g^{-1} [DMB]): 61.0–76.7 and 57.1–76.8, respectively. Thus, the analytical data may indicate the following aspects: First, only nine of the 25 samples of Kishk had slightly greater values for the content of carbohydrates when calculated by difference as compared with the summation method (starch + dietary fibre + galactose + lactose), and the rest of the samples (i.e. four had slightly lower values and 12 were similar). The discrepancy in the carbohydrate content was analysed using two-way analysis of variance (ANOVA) and a one-way *t*-test of significance between the means ($P < 0.069$). The differences in the two methods could be attributed to: (a) the exact ratio of Burghol to fermented milk was unknown; (b) the sampling error in which the ground fractions of milk and Burghol ingredients may not be uniformly distributed; and (c) in some instances, strained low-fat yoghurt might have been used rather than low-fat yoghurt, which may have affected the overall analysis. Second, a trace amount of glucose (data not shown in *table II*) was detected in samples 1, 3, 6, 8, 9, 11 and 17. Third, Kishk samples containing substantial amounts of galactose (≥ 3 g·100 g^{-1} [DMB]) may suggest that the starter cultures used to ferment the milk were not able to metabolise galactose when compared with glucose. Four, unknown peaks of sugars were observed in several Kishk samples at low concentrations, and when the Burghol sample was analysed, these carbohydrates (g·100 g^{-1} [DMB]) were confirmed as 0.12 mannose, 2.74 glucose, 0.63 sucrose, 0.24 maltose and 0.53 raffinose. There is no published analytical data on carbohydrates in Kishk to compare the analysis shown in *table II*. Nevertheless, the Kishk samples contain a substantial amount of starch (digestible and non-digestible fractions) which averaged 50.6 g·100 g^{-1} (DMB). In addition, an appreciable amount of dietary fibre (~9.3 g·100 g^{-1} [DMB]) was found in the 25 samples of Kishk, and Burghol is the most likely source of fibre. The variation in the fibre content in these samples (*table II*) suggests that: (a) the Burghol was produced from different varieties of wheat; (b) the efficiency of de-husking of Burghol varied from different sources; and (c) the amount of Burghol used in Kishk-making varied.

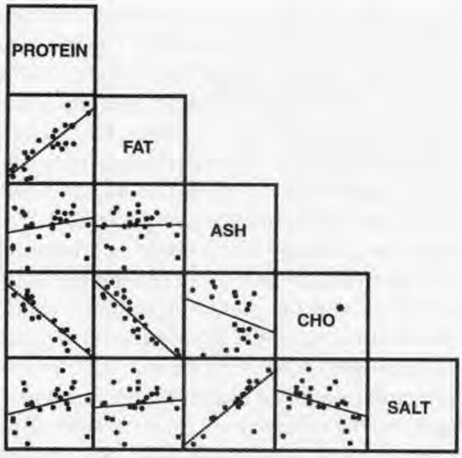

Figure 1. Scatter plots, with linear regression slope, of the main chemical components of Kishk. * CHO: carbohydrate.

Figure 1. Représentation de la dispersion des principaux composants du Kishk, avec les droites de régression. * CHO : sucre.

Figure 1 shows the relationships between the chemical constituents of the Kishk samples. Statistical analysis suggests that: (a) there is a strong positive correlation ($r = 0.865$) between protein and fat and (b) strong negative correlations ($r = -0.950$ and -0.917) between protein and carbohydrate, and between fat and carbohydrate, respectively. Such correlations could be influenced by the fact that most of the fat and much of the protein are derived from the yoghurt, and, therefore, Kishk made with higher yoghurt/Burghol ratio might be expected to be richer in protein and fat, and poorer in carbohydrate. There is no obvious relationship between ash and the other three variables; however, since the amount of salt in each Kishk sample is a component of the ash content [34], it was observed that salt was not related to or was not independent of protein, fat or carbohydrate (*figure 1*).

ANOVA of the main chemical components of Kishk (protein, fat, ash and carbohydrates), along with the information shown in *table I* (mainly geographical region, type of milk used, source/manufacturer or the method of packaging), showed no statistical significant differences. It was decided to use PCA on the correlation matrix of the data, and produce a PC bi-plot (*figure 2*) of the 25 Kishk samples for protein, fat, ash and carbohydrates to try to identify groupings among the samples. The first PC separated the Kishk samples which were relatively high in protein and fat contents from those which were high in carbohydrates. This axis accounts for the majority of the variability (72 %; see *figure 2*). The second PC accounts for a further 25 % of the variation, and separated the Kishk samples by their ash content. Since the bi-plot accounts for a total of 97 % of the total variation, it well represents the chemical data. There are no obvious groupings or clusters of the Kishk samples in the chemical data. The bi-plot pictorially demonstrates the similarity of one product to another; for example, Kishk samples 17, 5, 4 and 21 have low protein, fat and ash contents, whilst products 7 and 8 have high protein, fat and ash contents.

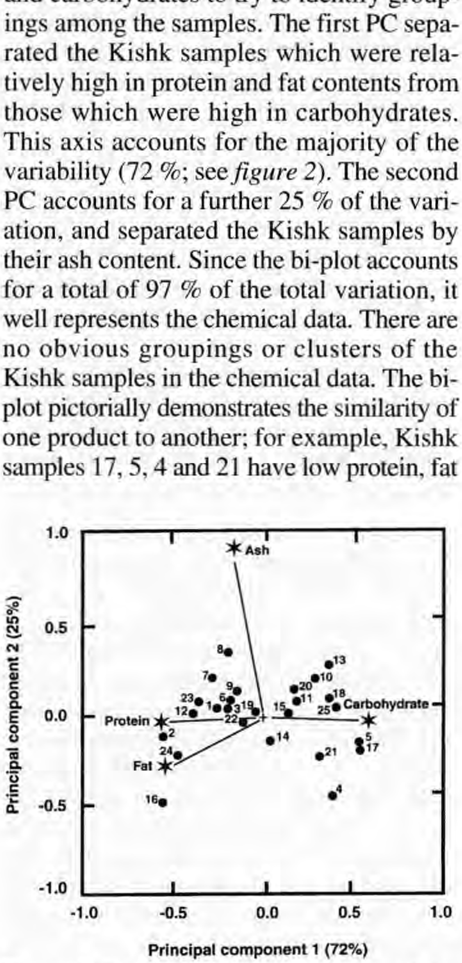

Figure 2. Principal components bi-plot of the chemical constituents of Kishk samples. For sample identification refer to *table I*.

Figure 2. Analyse en composantes principales des constituants des échantillons de Kishk. Voir le *tableau I* pour l'identification des échantillons.

3.2. Organic acids

The ranges of concentration ($\mu g \cdot g^{-1}$) of organic acids in the Kishk samples are shown in *table II*. No published data are available on the organic acids profile in Kishk; however, comparing the results in this survey with the data available on yoghurt [20, 21], the following observations/comparisons can be made: (a) There is a large concentration of lactic acid, which may indicate that the fermentation of milk was achieved by the use of lactic acid bacteria, and the level of lactic acid in Kishk is two-to three-fold higher compared with yoghurt. (b) A small amount of propionic acid (range between 900 and 7 400 $\mu g \cdot g^{-1}$) is found in the majority of the Kishk samples, suggesting the presence of propionic acid bacteria in the starter culture used or possibly acetoin since these organic acids may co-elute; however, much lower propionic acid concentration (< 120 $\mu g \cdot g^{-1}$) has been reported in yoghurt [21]. (c) The orotic, citric and uric/formic acid contents were generally fairly low in Kishk when compared with yoghurt. (d) The pyruvic acid content in yoghurt ranged between 24 and 46 $\mu g \cdot g^{-1}$ [20, 21], and nearly half the Kishk samples contained similar pyruvic acid concentration with the exception of samples 15, 16, 20 and 23 (higher) and only seven Kishk samples contained < 20 $\mu g \cdot g^{-1}$ of pyruvic acid. Finally, (e) the acetic acid content (range between 400 and 850 $\mu g \cdot g^{-1}$) in all the Kishk samples was much higher when compared with yoghurt (~130 $\mu g \cdot g^{-1}$) [20, 21]. The combined levels of organic acids in the different Lebanese Kishk samples were high in all the products and the pH measurement averaged 3.8 (*table II*), thus ensuring the microbiological safety of the product.

Kishk samples 24 and 25 had appreciably high orotic (17.76 $\mu g \cdot g^{-1}$) and citric (504 $\mu g \cdot g^{-1}$) acids content, respectively.

They were different from the rest and were discarded as outliers. This may suggest that the starter culture organisms used by these two manufacturers to ferment the milk did not metabolise orotic and citric acids when compared with the remainder of the samples. These results probably reflect the variation in the metabolic activity of the starter culture.

Correlation coefficients were calculated between the organic acid content for the remaining 23 Kishk samples, and no strong relationship was observed between any pair of organic acids. However, PCA was performed on the organic acid data of these Kishk samples, and a PC bi-plot was plotted (*figure 3*). PC 1 accounted for only 37 % of the total variation, and this was made up by the contrast between the Kishk products with high propionic, lactic and uric/formic acid contents and those with high acetic, orotic, pyruvic and citric acid contents. No clear groupings of the different Kishk samples was evident. PC 2 accounted for a further 19 % of the variation, and the interpretation of this axis is difficult.

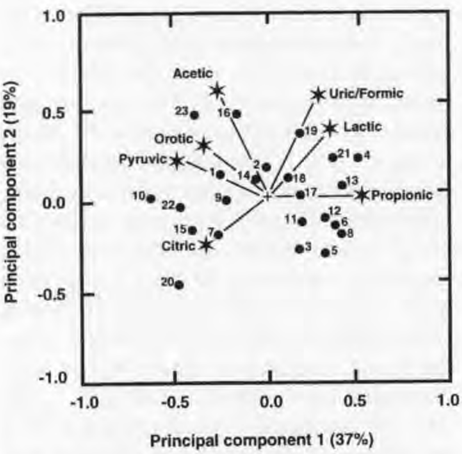

Figure 3. Principal component bi-plot of the organic acid contents of 23 Kishk samples. For sample identification refer to *table I*.

Figure 3. Analyse en composantes principales des teneurs en acides organiques de 23 échantillons de Kishk. Voir le *tableau I* pour l'identification des échantillons

3.3. Fatty acids

Variations in the concentration of different fatty acid contents of 25 Lebanese Kishk samples were observed (see *table II*). These variations are primarily due to the composition of milk fat of different species of mammals, and extensive reviews have been published in the literature [10, 16, 17, 24, 35]. The diet of ruminant animals (cow, goat or sheep) can influence the fatty acid composition of milk fats [33]. The fatty acid $C_{18:2}$ content of Kishk samples 13, 17, 19 and 25 was excessively high (*figure 4*), which may possibly suggest: (a) a higher level of Burghol was used, but unlikely to be the major source; (b) a high level of vegetable oil was used in the diet of ruminants in which the milk was subsequently used in Kishk-making; and/or (c) that the de-hydrogenation may have occurred during the secondary fermentation stage of Kishk-making (the fermented milk/Burghol mixture is left at ambient temperature for 1 week) [32].

Studies of the composition of fatty acids of milk fats of goat's and sheep's milk suggest the following differences: (a) goat's milk contains twice the amount of $C_{8:0}$, $C_{10:0}$ and $C_{12:0}$ compared with cow's milk [18]; and (b) sheep's milk contains twice the amount of $C_{4:0}$ to $C_{12:0}$ present in cow's milk [2]. The data shown in *table II* does not confirm the 'true' origin of the milk used for Kishk-making despite the fact that some products have been identified by the manufacturers (*table I*). The lack of proper 'mapping' of fatty acid contents of Kishk may suggest that sheep's and/or goat's milks have been adulterated with cow's milk. Many research workers in this field have suggested that correlations of certain fatty acids ($C_{4:0}/(C_{6:0} + C_{8:0})$, $C_{10:0}/C_{8:0}$, $C_{14:0}/C_{8:0}$, $C_{12:0}/C_{10:0}$, $C_{14:0}/C_{12:0}$ and/or $C_{15:0}/C_{14:1}$) may be useful in detecting adulteration of sheep's milk with cow's milk at a level 15–20 %

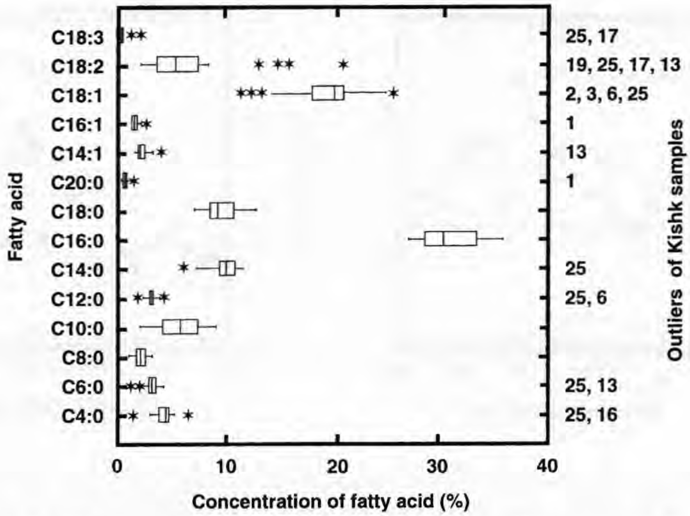

Figure 4. Box plot of concentration of fatty acids of the Lebanese Kishk samples. For sample identification refer to *table I*. Data were calculated on weight of fat. The vertical line inside the box represents the median and the vertical ends of the box represent the 1st and 3rd quartiles. Asterisks represent outliers which are further out than 1.5 × inter-quartile range from the 1st and 3rd quartiles, respectively.

Figure 4. Concentrations en acides gras des échantillons de Kishk libanais, avec les intervalles d'incertitude. Voir le *tableau I* pour l'identification des échantillons. Données calculées à partir du poids de la matière grasse. Les lignes verticales à l'intérieur des boîtes représentent la valeur médiane et les extrémités verticales de boîtes les 1er et 3e quartile. Les astérisques représentent les échantillons extrêmes qui sont au-delà de 1,5 fois la variation inter-quartile entre le 1er et 3e quartile.

rather than individual fatty acid contents [26]. Furthermore, the ratio of fatty acids $C_{12:0}/C_{10:0}$ has been recommended to identify the milk origin in different cheeses for which ratios of 1.2 and 0.46 for cow's and goat's cheeses, respectively [14]. The ratio of 0.46 becomes proportionally greater with increased adulteration of goat's milk with cow's milk.

Such ratios of fatty acids have been used to discriminate the Kishk samples and to identify whether goat's or sheep's milk has been adulterated with cows' milk. Some of the results are shown in *figure 5* which provided the most appropriate groupings of cow, goat or mixed Kishk products. It is difficult to confirm the type of milk used because this approach has its limitation where the level of adulteration of cow's milk with goat's milk should be 15–20 % [18]. Nevertheless, Kishk samples 13, 15, 16, 18 to 21 and 25 are made from cows' milk, and the remaining samples are made from mixed milks (see *figure 5*). It is possible that combined analysis of fatty acids and profiling of protein fractions in Kishk may confirm which of the sample(s) is made from pure goat's milk. The latter results will be published separately [34].

PCA was performed on the correlation matrix of the fatty acid data of the Kishk samples, and a PC bi-plot was plotted (*figure 6*). The first and second PCs contained 73 % of the total variation; hence, the bi-plot is a reasonable summary of the correlation matrix. PC 1 accounted for 48 % of the total variation and contrasted samples of Kishk high and low in C18:2. Samples

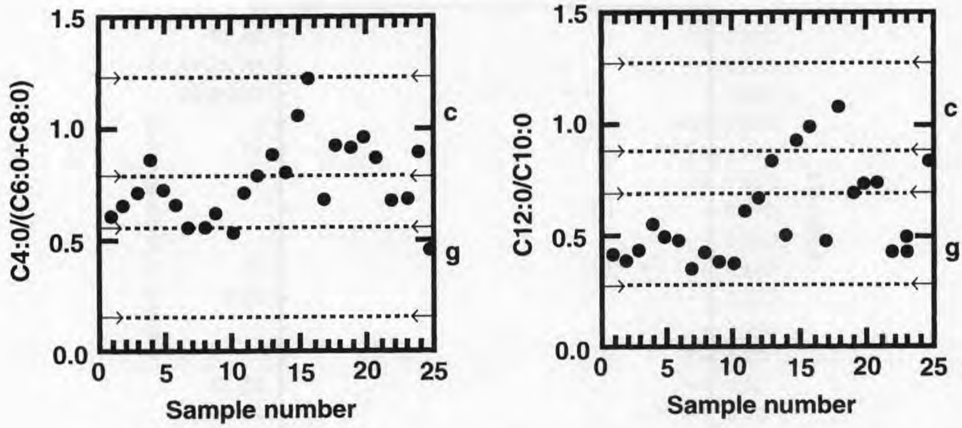

Figure 5. Ratios of certain fatty acids in Lebanese Kishk samples which have been proposed for detecting the adulteration of goats' milk with cows' milk. For sample identification refer to *table I*. c: cow, g: goat. The fatty acid correlations were based on data reported by Ramos and Juárez [26].

Figure 5. Ratios en certains acides gras d'échantillons de Kishk libanais choisis pour la détection d'adultération du lait de chèvre par du lait de vache. Voir le *tableau I* pour l'identification des échantillons. c : vache ; g : chèvre.

13, 17, 19 and 25 were very high in $C_{18:2}$ when compared with the rest of the samples. PC 2 accounted for a further 25 % of the variation and has no clear interpretation.

4. CONCLUSION

Differences in Lebanese Kishk composition were observed that could mainly be attributed to the different levels of yoghurt and Burghol used during preparation. The chemical analysis of 25 Kishk samples were similar to those previously reported in the literature. Profiling of fatty acids in Kishk did not provide conclusive evidence regarding the type of milk used, with the exception of some samples where the goat's milk had been adulterated with cow's milk at a rate of ~20 %.

No statutory instruments exist in Lebanon concerning the chemical composition of Kishk, and in such circumstances it is possible to propose that the yoghurt/Burghol ratio should be 4:1.

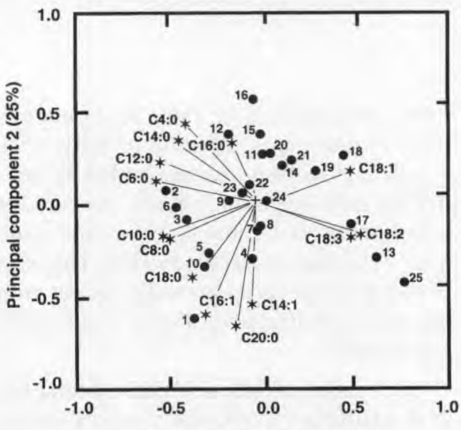

Figure 6. Principal component bi-plot of fatty acids of Kishk samples. For sample identification refer to *table I*.

Figure 6. Analyse en composantes principales des acides gras des échantillons de Kishk. Voir le *tableau I* pour l'identification des échantillons.

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