



Relationships between the evolution of the percentage in weight of polar compounds and that of the molar percentage of acyl groups of edible oils submitted to frying temperature

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ABSTRACT

The evolution of the molar percentage of several kinds of acyl groups of extra virgin olive, sunflower and virgin linseed oils was monitored throughout heating at frying temperature by means of ¹H nuclear magnetic resonance. Likewise, the evolution of the percentage in weight of the polar compounds of the same oils under the same heating conditions was also determined. Relationships between both sets of parameters, in each oil and in the oils as a group, were studied. An equation which is able to accurately predict the percentage in weight of the polar compounds, throughout the heating at frying temperature, of any one of these three oils, from the molar percentage of triunsaturated, diunsaturated and monounsaturated acyl groups, was obtained. In this way both molar percentage of acyl groups and percentage in weight of polar compounds can be obtained in a few minutes that registration of the ¹H NMR spectrum of the oil takes, in addition to the rest of information provided by this technique. The study reveals the close relationships between percentage in weight of polar compounds and the composition expressed in terms of molar percentages of acyl groups in edible oils heated at frying temperature.

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1. Introduction

The degradation of food lipids has, as a consequence, not only the formation of compounds with undesirable organoleptic properties but also of compounds with well-known toxicity (Guillén & Goicoechea, 2008). Heat and the presence of oxygen or other pro-oxidants are both responsible for lipid degradation. Deep-frying is one of the most widely used culinary processes in which lipid degradation is produced. Nowadays, when food safety is established as one of the pillars of public health, most countries have limits for the presence of toxic compounds in foods. The safety of edible oils used in deep-frying is monitored in most countries by their contents of polar compounds (Stier, 2004). These are defined as those compounds present in the oil that are retained on a silica gel column, their nature and identity not being totally well-known. The determination of this parameter was well-established (Walking & Wessels, 1981); however, faster and simpler methods have been developed. These are based on physical properties measurements, such as viscosity, density, surface tension, or dielectric constant, or on chemical tests directed to the monitoring of certain compounds formed during the oil degradation (Stier, 2004). Some

of these methods provide very closely related results, a reason for which they have been considered equivalent (Marmesat, Rodrigues, Velasco, & Dobarganes, 2007).

In a previous study (Guillén & Uriarte, 2011), relationships between the percentage in weight of polar compounds of non-heated oil and the oil composition, expressed as molar percentage of acyl groups, were tested. A great variability in the polarity of non-heated oils of different botanical origins was shown, that of the oils rich in polyunsaturated groups being higher than that of those rich in monounsaturated or saturated groups. Likewise, very close relationships between the time at which edible oils reach 25% in weight of polar compounds and the original oil composition (expressed as molar percentage of their acyl groups) were also found. The equations that relate these parameters have predictive value, enabling the prediction of the safe heating time of oils of very different nature.

In this context, this study concerns the evolution of the percentage in weight of polar compounds of three oils of very different nature submitted to frying temperature and also to the evolution of their main acyl groups. The oils involved in the study were extra virgin olive oil, sunflower oil and virgin linseed oil. The aim was to explore possible relationships between percentage in weight of polar compounds and oil composition, taking into account that the latter is responsible for the oil dielectric constant and for its percentage in weight of polar compounds.

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2. Materials and methods

2.1. Oil samples

The oils subjected to study were extra virgin olive oil, sunflower oil and virgin linseed oil. All of them were acquired in local supermarkets. The percentages of oleic or monounsaturated groups (MU), of linoleic or diunsaturated groups (DU), and of linolenic or triunsaturated groups (TU) in the original oils were determined from their ^1H nuclear magnetic resonance spectra, as in previous studies (Guillén & Uriarte, 2012a, 2012b, 2012c). The obtained average data are shown in Tables 1, 3 and 4.

2.2. Thermodegradation of the oils

Four litres of the above cited oils were heated at frying temperature (190 °C) for periods of 8 h/day, in a discontinuous industrial fryer (Franke ECO4, 230 V, 10A, 2.3 kW) for 38 and 40 h in the case of extra virgin olive and sunflower oils, respectively, and for 20 h in the case of virgin linseed oil. The temperature was periodically tested by a calibrated thermometer. The dimensions of the stainless steel tank of the fryer were 12 cm wide \times 30 cm long \times 17 cm high. Throughout the heating no amount of oil was replenished and the cover was kept closed. The oil was stored at room temperature between the heating episodes. Periodically, throughout the heating, the percentage in weight of polar compounds was determined; at the same time samples were taken in order to determine, by ^1H NMR, the molar percentages of the several kinds of acyl groups.

2.3. Registration of the ^1H nuclear magnetic resonance spectra and determination of the molar percentage of the acyl groups

The spectra were registered in a Bruker Avance 400 spectrometer operating at 400 MHz. The oil sample (200 μl) was mixed in a 5 mm diameter tube with 400 μl of deuterated chloroform, which contained 0.2% of non deuterated chloroform and a small amount (0.03%) of tetramethylsilane (TMS) as internal references. In order to select the most appropriate values to obtain accurate quantitative results in the smallest possible period of time, a very broad range of recycling times and relaxation delays was tested in the acquisition of the ^1H NMR spectra, not only of the original oils but also of the oils later to be submitted to different heating times. In this way, the acquisition parameters used were: spectral width 5000 Hz, relaxation delay 3 s, number of scans 64, acquisition time 3.744 s, and pulse width 90°, with a total acquisition time of 12 min 54 s. The experiment was carried out at 25 °C. The determination of the molar percentage of the several acyl groups of the oils

Table 1

Average molar percentage of monounsaturated (MU), diunsaturated (DU), and triunsaturated (TU) acyl groups and percentage in weight of polar compounds (PC) in extra virgin olive oil at different times of heating at 190 °C.

Heating time (h)	MU%	DU%	TU%	PC%
0	82.0 \pm 1.20	4.95 \pm 0.25	0.59 \pm 0.01	7.00
2	81.9 \pm 2.98	4.62 \pm 0.21	0.59 \pm 0.05	7.75
10	81.4 \pm 2.23	3.92 \pm 0.16	0.41 \pm 0.03	12.5
17	80.6 \pm 3.10	3.74 \pm 0.21	0.28 \pm 0.01	16.0
20	79.5 \pm 1.13	3.70 \pm 0.25	0.31 \pm 0.07	20.0
24	79.3 \pm 0.88	3.37 \pm 0.24	0.24 \pm 0.06	20.0
26	79.2 \pm 1.52	3.29 \pm 0.16	0.22 \pm 0.05	21.3
28	79.0 \pm 1.48	3.34 \pm 0.35	0.28 \pm 0.03	22.5
31	77.8 \pm 0.85	3.38 \pm 0.25	0.35 \pm 0.05	22.5
33	77.9 \pm 2.25	2.98 \pm 0.21	0.24 \pm 0.01	23.5
36	77.9 \pm 1.32	2.74 \pm 0.16	0.19 \pm 0.04	26.3
38	78.1 \pm 2.22	2.65 \pm 0.25	0.18 \pm 0.03	26.5

Table 2

Coefficients of monoparametric [$\text{PC}\% = a + b (\text{AG}\%)$] ($\text{AG} = \text{MU}, \text{DU}$ or TU) or of biparametric [$\text{PC}\% = a + b (\text{MU}\%) + c (\text{TU}\%)$] equations that relate percentage in weight of polar compounds PC% and the molar percentage of certain acyl groups (monounsaturated (MU), diunsaturated (DU), triunsaturated (TU) acyl groups) in the oils throughout the heating, the correlation coefficient (R), and the number of points involved (n).

Oil	Eq. no.	Variables	a	b	c	R	n
Extra virgin olive	1	MU%	340.59	-4.04	-	0.9646	12
Extra virgin olive	2	DU%	51.85	-9.29	-	0.9714	12
Extra virgin olive	3	TU%	32.93	-43.75	-	0.9225	12
Extra virgin olive	4	MU%, TU%	236.90	-2.66	-19.05	0.9917	12
Sunflower oil	5	MU%	-255.65	7.96	-	0.9086	11
Sunflower oil	6	DU%	127.33	-2.13	-	0.9942	11
Virgin linseed oil	7	DU%	-7.68	1.61	-	0.9332	11
Virgin linseed oil	8	TU%	73.09	-0.95	-	0.9663	11

Table 3

Average molar percentage of monounsaturated (MU), diunsaturated (DU), and triunsaturated (TU) acyl groups and percentage in weight of polar compounds (PC) in sunflower oil at different times of heating at 190 °C.

Heating time (h)	MU%	DU%	PC%
0	33.5 \pm 2.51	54.7 \pm 3.20	11.0
2	33.7 \pm 1.85	53.8 \pm 2.89	13.0
4	34.0 \pm 2.11	53.0 \pm 2.19	13.5
8	34.2 \pm 3.11	52.4 \pm 3.02	16.0
12	34.3 \pm 1.26	51.1 \pm 2.06	18.5
16	35.4 \pm 2.85	50.0 \pm 2.51	19.5
20	35.0 \pm 1.08	49.2 \pm 1.13	21.8
28	34.9 \pm 0.98	48.2 \pm 1.02	25.8
32	35.4 \pm 1.13	47.6 \pm 1.26	25.8
36	35.3 \pm 0.85	46.4 \pm 1.98	29.3
40	35.7 \pm 1.06	45.3 \pm 2.00	30.0

Table 4

Average molar percentage of monounsaturated (MU), diunsaturated (DU), and triunsaturated (TU) acyl groups and percentage in weight of polar compounds (PC) in virgin linseed oil at different times of heating at 190 °C.

Heating time (h)	MU%	DU%	TU%	PC%
0	19.4 \pm 0.95	19.8 \pm 0.99	52.3 \pm 3.25	23.5
1	19.8 \pm 0.65	19.6 \pm 0.78	51.1 \pm 3.10	23.5
2	19.3 \pm 0.84	20.0 \pm 1.15	51.0 \pm 2.98	24.8
4	18.7 \pm 1.06	20.7 \pm 1.86	50.5 \pm 3.01	25.3
7	18.6 \pm 0.58	20.7 \pm 1.87	49.4 \pm 2.03	26.8
8	19.2 \pm 1.26	21.6 \pm 2.00	48.5 \pm 4.02	26.8
10	17.8 \pm 0.98	22.6 \pm 1.98	47.8 \pm 3.58	27.5
12	20.2 \pm 1.35	21.5 \pm 2.00	46.9 \pm 1.22	27.5
15	19.5 \pm 1.12	23.0 \pm 0.97	45.9 \pm 2.86	28.3
18	18.8 \pm 1.24	24.1 \pm 1.02	44.8 \pm 3.02	30.5
20	19.3 \pm 0.98	23.4 \pm 1.11	44.3 \pm 2.98	32.0

submitted to frying temperature was carried out from spectral data, using approaches developed in previous studies (Guillén & Uriarte, 2009, 2012a, 2012b, 2012c). The average obtained data are shown in Tables 1, 3 and 4.

2.4. Determination of the percentage in weight of polar compounds

This parameter was determined throughout the heating by the Testo 265 instrument. This device measures the dielectric constant of the oil and transforms this value directly into percentage in weight of polar compounds. Measurement is carried out by immersing the sensor in hot oil and provides the % in weight of polar compounds, together with the temperature. Accuracy values are: $\pm 2.0\%$ PC (+40 to +190 °C) and ± 1.5 °C. Values obtained at several heating times are given in Tables 1, 3 and 4.

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