



## Occurrence of furan in coffee from Spanish market: Contribution of brewing and roasting

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### ABSTRACT

In this work, we evaluated the occurrence of furan in brews obtained from regular, decaffeinated, and instant coffee and commercial packed capsules. For this purpose, a previously developed automated headspace solid-phase microextraction method coupled to gas chromatography–mass spectrometry (HS-SPME-GC–MS) was used. Initially, the influence of HS-SPME conditions on furan formation was evaluated. In addition, the effect of roasting conditions (temperature and time) used for coffee beans on furan formation was also studied. We found that low temperature and long roasting time (140 °C and 20 min) decreases the final furan content. Furan concentrations in regular ground coffee brews from an espresso coffee machine were higher (43–146 ng/ml) than those obtained from a home drip coffee maker (20 and 78 ng/ml), while decaffeinated coffee brews from a home drip coffee maker (14–65 ng/ml) showed a furan concentration similar to that obtained from regular coffee. Relatively low concentrations of this compound (12–35 ng/ml) were found in instant coffee brews, while commercial packed coffee capsules showed the highest concentrations (117–244 ng/ml). Finally, the daily intake of furan through coffee consumption in Barcelona (Spain) (0.03–0.38 µg/kg of body weight) was estimated.

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### 1. Introduction

In recent years considerable attention has been given to the study of heat-induced toxicants in food as a result of their potential harmful effects on human health (Wenzl, Lachenmeier, & Gökmen, 2007). Among these chemicals, furan (C<sub>4</sub>H<sub>4</sub>O) is one of the main undesirable contaminants formed during thermal treatment of food and drink. Although the presence of furan in foods has been known for years (Maga, 1979), interest in this compound has increased recently because of its classification as a possible human carcinogen (Group 2B) by the international Agency for Research on Cancer (IARC) (FDA, 2004; IARC, 1995; NTP, 2004). Therefore, monitoring programs have been launched by several food organisations such as the US Food and Drug Administration (FDA), the Swiss Federal Office of Public Health (SFOPH) and the European Food Safety Authority (EFSA) (EFSA, 2004; FDA, 2008; SFOPH, 2004), to determine the occurrence of furan in food commodities. Several mechanisms that can explain furan formation in foods are well documented. These mechanisms are related mainly to thermal degradation of carbohydrates, pyrolysis of sugars at high temperature, oxidation of polyunsaturated fatty acids and decomposition of ascorbic acid and its derivatives (Crews & Castle, 2007).

Coffee contains higher concentrations of furan (up to 199 ng/ml) than other processed foods (EFSA, 2004; FDA, 2008; SFOPH, 2004; Zoller, Sager, & Reinhard, 2007). Moreover, given that coffee is highly consumed (ICO, 2008), exposure to furan may be a potential risk to human health. Furan in coffee is generated during the roasting of green coffee beans, which are rich in carbohydrates and amino acids (Murkovic & Derler, 2006). The temperature and time used during roasting are the main factors responsible for furan formation in coffee. In addition, the brewing procedure can also affect the furan content. However, there is little information about the influence of roasting and brewing processes on furan formation in coffee (Kuballa, Stier, & Strichow, 2005; Zoller et al., 2007). Moreover, the measurement of human exposure to furan is hampered by a scarce data about its occurrence in this beverage (EFSA, 2007), which is the main source of furan for adults. In order to obtain reliable data to assess the exposure of humans to furan through coffee consumption, more information is required.

The analysis of furan is currently performed by headspace (HS) (Crews, Roberts, Laurysen, & Kramer, 2009; FDA, 2006; Morehouse, Nyman, Mcneal, Dinovi, & Perfetti, 2008; Zoller et al., 2007) or headspace solid-phase microextraction (HS-SPME) (Altaki, Santos, & Galceran, 2007, 2009; Bianchi, Careri, Mangia, & Musci, 2006; Ho, Yoo, & Tefera, 2005; La Pera et al., 2009) both coupled to gas chromatography–mass spectrometry (GC–MS). However, furan can be generated during the analysis and the extraction temperature and time must be controlled to prevent its formation (Crews & Castle, 2007). For instance, furan formation

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has been reported during HS analysis of ground green coffee beans at a temperature as low as 40 °C (Senyuva & Goekmen, 2005). Moreover, the HS method proposed by the FDA in 2004 was updated in 2006, decreasing sampling temperature from 80 to 60 °C because of the observation of furan formation at the former temperature (Nyman, Morehouse, McNeal, Perfetti, & Diachenko, 2006, 2008). One advantage of using HS-SPME instead of HS is that lower extraction temperatures (~30 °C) can be commonly used and consequently the generation of furan can be reduced.

Here we examined the furan content in brews obtained from regular, decaffeinated and instant coffee commercialised in Spain. We used a fully automated HS-SPME-GC-MS method. In addition, the occurrence of furan in coffee brews from commercially packed capsules was evaluated for the first time. The influence of HS-SPME conditions, such as temperature and time, on furan formation during the analysis was also studied. Moreover, the effect of roasting conditions and brewing procedures on the furan content of the final brews was examined. By means of a 24-h recall questionnaire, we also estimated furan intake from coffee consumption in a small population sample in Barcelona (Spain).

## 2. Materials and methods

### 2.1. Chemicals and standards

Furan and [<sup>2</sup>H<sub>4</sub>] furan (furan-d<sub>4</sub>) at a purity >99% were purchased from Sigma–Aldrich (Munich, Germany). Water of organic trace grade, methanol of GC grade and sodium chloride of analytical grade were all obtained from Merck (Darmstadt, Germany). Individual stock standard solutions of furan and furan-d<sub>4</sub> at a concentration of 12 mg/g in methanol were prepared by transferring 20 µl of pure compounds to a 2-ml amber-sealed vial previously filled with methanol. Intermediate standard solutions of furan and furan-d<sub>4</sub> were prepared by weight at a concentration of 90 µg/g from stock standard solutions by appropriate dilution with methanol. All these solutions were stored at 0 °C and prepared weekly. Aqueous working standard solutions of furan and furan-d<sub>4</sub> at a concentration of 90 ng/g were individually prepared daily by spiking 20 ml of water with appropriate volumes of their intermediate standard solutions. For furan determination, eight-calibration standard solutions at concentrations ranging from 0.05 to 10 ng/g were prepared by adding in weight, through the septum, an appropriate amount of the aqueous working standard solution of furan into a 20-ml HS vial. In addition, 100 µl of the furan-d<sub>4</sub> aqueous working solution was added to each calibration solution to achieve a concentration of 0.9 ng/g.

### 2.2. Coffee sample preparation

A total of 23 samples of ground coffee (15 caffeinated and 8 decaffeinated) and five samples of instant coffee, selected from the most consumed brands in Spain, were obtained from the major markets in Barcelona (Spain). In addition, 12 Nespresso® coffee capsules (Nestlé Nespresso® SA, Paudex, Switzerland) (*Ristretto*, *Arpeggio*, *Roma*, *Livanto*, *Capriccio*, *Volluto*, *Cosi*, *Decaffeinato*, *Decaffeinato intenso*, *Vivalto Lungo*, *Decaffeinato Lungo* and *Finezza Lungo*) were purchased. Before use, commercial coffee samples were stored at 4 °C in their sealed packages to prevent loss of furan. To study the effect of roasting on furan formation, green coffee beans from Brazil were purchased from a local roasting factory in Barcelona, Spain. Portions of these beans (ca. 100 g) were then individually roasted at temperatures of 140, 170 and 200 °C for 6 min in an electric rotating laboratory roaster (Probat-Werke Prä 1Z, Probat-Werke, Germany). Moreover, to determine the influence of temperature and time on the formation of furan, several

portions of the beans were roasted to the same degree under three temperature–time conditions: high temperature–short roast (200 °C, 6 min), intermediate temperature–intermediate roast (170 °C, 12 min) and low temperature–long roast (140 °C, 20 min). All coffee samples were then ground at room temperature to a commonly commercialised particle size (200–250 µm). To determine the degree of roasting, the international standard colour space parameters were used. These parameters, proposed by the International Commission on Illumination (CIE) (Papadakis, Abdul-Malek, Kamdem, & Yam, 2000), allow the characterisation of coffee colour by means of  $L^*a^*b^*$  parameters, where  $L^*$  is the luminance or lightness component and  $a^*$  and  $b^*$  are the chromaticity coordinates. These measurements were performed using a Chromameter CR-410 spectrophotometer (Konica Minolta, Osaka, Japan).

The regular ground coffee samples (9 g) were brewed using an espresso coffee machine and a home drip coffee maker to obtain approximately 60 ml of brew in both cases. For the coffee roasted in the laboratory, only the automatic espresso machine was used to obtain brews. Decaffeinated ground coffees were prepared with a home drip coffee maker using the same coffee/water ratio as that used for the brewed regular ground coffee. Instant coffee was obtained following the manufacturer's recommendations, mixing 2 g of powder with 60 g of boiling water. For preparation of Nespresso® coffee, all espresso blends, ca. 5.5 g of coffee, were brewed with an Essenza Nespresso coffee machine to obtain 40 ml of coffee, while for the Lungo coffee capsules, 110 ml of brew was obtained from ca. 7 g of coffee. After preparation, all brews were placed immediately in a closed vial with no headspace and then stored at 4 °C until analysis.

### 2.3. GC–MS conditions

GC–MS analyses were performed on a Varian 3800 gas chromatograph coupled to a Saturn 2200 ion-trap MS system (Varian, Mississauga, Canada) and equipped with a CTC Combi-Pal auto-sampler (CTC Analytics AG, Zwingen, Switzerland), which is configured with a sample tray for 32 vials of 20 ml, a SPME fibre conditioning station, and a temperature-controlled single magnet mixer tray. Separation was performed on a BX-volatile (cyanopropylphenyl polysilphenylene-siloxane), 60 m × 0.25 mm i.d., fused-silica capillary column (SGE Europe, Villebon, France) with a film thickness of 1.4 µm. The temperature programme was 35 °C (held for 2 min) to 230 °C at 20 °C/min (held for 5 min). The injector was fitted with an SPME inlet liner (i.d., 0.75 mm, SGE Europe) and a 23-gauge Merlin Micro-seal septum (Supelco, Bellefonte, PA, USA) and operated in splitless injection mode (3 min). Helium was used as carrier gas at a constant flow-rate of 1.7 ml/min. The ion trap mass spectrometer was tuned using perfluorotributylamine (FC-43) following the manufacturer's recommendations. Electron ionisation (70 eV) with 30 µA of emission current and temperatures of 200, 80 and 280 °C were set for the trap, manifold and transfer line, respectively. For MS acquisition, EI full-scan mode was used over the mass range  $m/z$  35–100 at 0.75 s/scan (7 µscan per scan). Quantification of furan was performed by isotope dilution using  $m/z$  68 and  $m/z$  72 for furan and furan-d<sub>4</sub>, respectively. For confirmation,  $m/z$  39 [M–CHO]<sup>+</sup> for furan was monitored. A Varian Saturn Workstation software (Version 6.42) was used for control, general operation and data acquisition of the GC–MS system, while the Cycle composer software (Version 1.5.3) was used to control and program the CTC Combi-Pal autosampler.

### 2.4. Automated HS-SPME method

Furan analysis in coffee samples was performed using an automated HS-SPME method that was previously developed and

validated (Altaki et al., 2009). Briefly, an aliquot (0.5 ml) of brewed coffee was quickly transferred from the storage vial, immersed in an ice/water bath (4 °C) to a 20-ml headspace vial containing a PTFE-coated stir bar, 2 g of sodium chloride and an adequate amount of water (up to 8 ml). The vial was immediately sealed and then spiked with 100 µl of furan-*d*<sub>4</sub> aqueous working standard solution. For HS-SPME analysis, a 75-µm carboxen-polydimethylsiloxane fibre (CAR/PDMS) (Supelco, Bellefonte, PA, USA), which was conditioned following the manufacturer's recommendations (300 °C under helium flow for 1 h) was used. Before analysis, the sample vial was equilibrated for 5 min in the single magnet mixer tray at 30 °C and 750 rpm. The fibre was cleaned for 1 min at 275 °C in a conditioning station and then immediately exposed to the headspace of sample vial for 20 min at 30 °C. Thermal desorption of furan was accomplished by exposing the fibre in the GC injector port at 275 °C for 2 min.

### 2.5. Quality control and quality criteria

Quality control of the HS-SPME method was performed through the routine analysis of procedural blanks and quality control standards and samples to ensure the absence of contaminants and possible carryover between samples, and to assess the quality of the results. In addition, a daily sensitivity test was carried out to check the possible changes in the absorption capacity of the SPME fibre and the GC-MS response. Although no certified reference materials are available, the method was assessed in an interlaboratory study on furan in baby food (*z*-score < 0.6), organised by the Institute for Reference Materials and Measurements (IRMM) of the European Commission's Joint Research Centre (JRC) (JRC, 2008). The identification criteria for furan were based on its retention time and the intensity ratios of furan and furan-*d*<sub>4</sub> ions. A deviation of the ion intensity ratios within 20% of the mean values of the calibration standards was considered acceptable.

### 2.6. Coffee consumption data and furan intake

Data on coffee consumption habits in Spain were obtained from the Spanish Coffee Federation (FEC) and the Spanish Ministry of Agriculture, Fisheries and Food (MAPA) (FEC, 2007; MAPA, 2007). Furthermore, coffee consumption data were obtained using a 24-h recall questionnaire on 488 participants (219 females and 269 males) aged from 19 to 58 years from five zones in the city of Barcelona. Each participant was asked about the number of cups of coffee had in the previous 24 h. Furan intake per day and capita from brewed coffee was estimated from the average amount of coffee consumed and the minimum and maximum concentration of furan detected in espresso and drip coffee brews.

## 3. Results and discussion

### 3.1. Evaluation of furan formation during HS-SPME analysis

Here we used an automated HS-SPME-GC-MS method previously developed for the analysis of furan in food (Altaki et al., 2009). The method provided good linearity ( $r^2 > 0.999$ ) over a wide range of concentrations (0.01–10 ng/g) and low limits of detection (LODs, 0.02–0.12 ng/g) with good precision (RSD% ≤ 10%). However, since the formation of furan from green coffee has been reported during headspace analysis at an extraction temperature of 40 °C (Senyuva & Goekmen, 2005), experiments were conducted to check whether furan is effectively formed during the HS-SPME method. The effects of extraction time and the sample size on furan formation were evaluated at the HS-SPME extraction temperature (30 °C). For this purpose, two aliquots (0.5 and 2 ml) of a natural

coffee brew obtained using an espresso coffee machine were analysed in triplicate using the proposed HS-SPME method (Section 2.4). No significant differences between the furan concentration in the two aliquots (57 ± 4 ng/ml for 0.5 ml of coffee and 55 ± 3 ng/ml for 2 ml) were observed, thereby demonstrating that the amount of precursor, which is directly related to the amount of coffee, did not affect the formation of this compound at 30 °C. In addition, an aliquot of 0.5 ml of the same coffee brew was analysed using an equilibration time of 100 min and the same extraction time (20 min). In this case, the concentration of furan (58 ± 4 ng/ml) was not statistically different to that obtained previously using a 5-min equilibration at a confidence level of 95%. These findings indicate that furan is not formed during HS-SPME analysis at 30 °C during the equilibration and extraction. Moreover, the stability of the sample in the vials before analysis was examined. For this purpose, four sample vials were placed in the sample tray of the automatic injector at laboratory temperature (20 ± 2 °C) and analysed following the proposed HS-SPME method after 0, 1, 6 and 15 h. For all standing times, good agreement in the results (RSD% < 12%) was obtained, thereby demonstrating the stability of the coffee samples in the vials before analysis.

### 3.2. Influence of the roasting process on furan formation

The properties of roasted coffee, such as aroma and taste, are highly influenced by the roasting degree, which particularly depends on the temperature and time applied during the process. However, there is little information about the effect of roasting conditions on furan formation. For instance, Zoller et al. (2007) found that furan concentration increases from 22 to 1792 ng/g (semi-quantitative data) when the roasting temperature is increased from 150 to 250 °C in an open headspace vial (small-scale in the laboratory). In our study, we addressed the effect of roasting on the furan content of green beans from Brazil, which were roasted under conditions commonly used in the coffee manufacturing industry (roaster, roasting degree, cooling and grinding). First, the effect of temperature was evaluated by roasting the coffee beans in an electric rotating roaster for 6 min at 140, 170 and 200 °C. After roasting, the beans were ground and the roasting degree was measured (Section 2.2). Green coffee was not analysed because it is not directly consumed. An Espresso machine was used to prepare a brew from each sample (9 g of coffee powder in 60 ml of water) and the furan content was determined in triplicate using the proposed HS-SPME GC-MS method. Furan levels increased from 22 ± 2 to 138 ± 8 ng/ml with the roasting degree ( $L^*$  from 47.4 to 23.16) (Table 1). These findings can be explained by the increase in roasting temperature.

The combined effect of both temperature and time on furan formation was evaluated by roasting several portions of Brazilian green coffee beans to a degree ( $L^* = 21 \pm 0.7$ ), commonly used for commercialised regular roasted coffees, but applying different temperature–time conditions. The following three conditions were tested: high temperature-short roast (200 °C, 6 min), intermediate

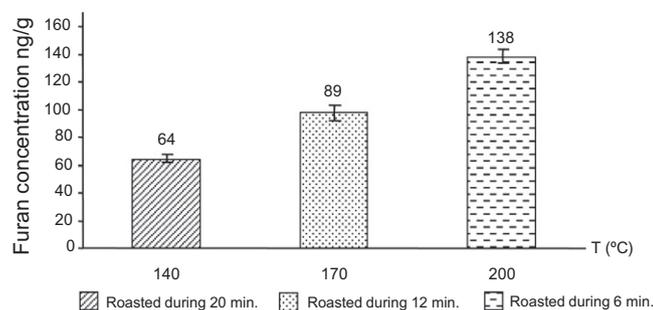
**Table 1**  
Effect of roasting temperature on furan level in coffee brews<sup>a</sup>.

Temperature (°C)	Lightness ( $L^*$ ) <sup>b</sup>	Furan level in brewed coffee	
		Mean ± sd (ng/ml) <sup>c</sup>	RSD (%)
140	47.4	22 ± 2	9
170	39.98	53 ± 3	6
200	23.16	138 ± 8	6

<sup>a</sup> Roasting time 6 min. Brewed with an automatic espresso machine at same conditions.

<sup>b</sup>  $L^* = 100$  (White), 0 (Black).

<sup>c</sup>  $n = 3$ .



**Fig. 1.** Influence of temperature–time roasting conditions on the furan content of brewed coffee (roasting degree,  $L^* = 21 \pm 0.7$ ).

temperature-intermediate roast (170 °C, 12 min) and low temperature-long roast (140 °C, 20 min). After roasting and grinding, coffee powder was brewed in an espresso machine to obtain 60 ml of coffee brew which was analysed by HS-SPME-GC-MS. The lowest furan concentration ( $64 \pm 5$  ng/ml) was found for low temperature and slow roasting (140 °C and 20 min) conditions, while for intermediate and quick roasting processes at higher temperatures, the concentration of this compound increased to  $89 \pm 7$  and  $138 \pm 8$  ng/ml, respectively (Fig. 1). These observations could be attributed to the combination of two effects, namely the decrease in furan formation at low temperature and the increase in the loss of furan by evaporation during the long roast at 140 °C.

### 3.3. Influence of coffee type and brewing procedure on furan content

The effect of the brewing procedure on the furan content of the coffee brews was studied using a professional espresso coffee machine and a home drip coffee maker, the most common machines used to prepare coffee in Europe. For this study, brews obtained from 15 commercial ground coffees selected from the most consumed brands in Spain were analysed in triplicate using the proposed HS-SPME GC-MS method. Furan concentration in brews from the two brewing methods ranged from  $20 \pm 3$  to  $78 \pm 6$  ng/ml for brews obtained with a home drip coffee maker and between  $43 \pm 4$  and  $146 \pm 12$  ng/ml for those obtained using espresso machine with a relative standard deviation lower than 15% (Table 2). These concentrations are consistent with those reported in the literature for brewed coffees of diverse origins (3–199 ng/ml) (EFSA, 2004; FDA, 2008; SFOPH, 2004; Zoller et al., 2007). Compar-

ing the results obtained using the two brewing methods and the same coffee/water ratio, the furan content in espresso brews was always higher, between 32% and 85%, than that obtained with a home drip coffee maker. These results can be explained by the higher pressure applied in the espresso machine (900–1000 kPa) than that in the home drip maker (100 kPa), which increases the extraction of furan from the coffee powder. Another factor that must be considered is the loss of furan in the cup or in the pot by evaporation during brewing. Preparation of a cup of coffee often requires 30 s using an espresso machine, while a home drip coffee maker takes about 100 s. This time difference may explain the higher furan content found in espresso coffee brews. High concentrations of furan in brews prepared with an espresso machine have been reported previously (Kuballa et al., 2005; Zoller et al., 2007).

Decaffeinated and instant coffee samples were also analysed. Decaffeinated brews were prepared using a home drip coffee maker while instant coffee samples were obtained following the manufacturer's instructions (Section 2.2). Furan was found in all samples at concentration levels ranging from 14 ng/ml to 65 ng/ml for decaffeinated coffees and between 12 ng/ml and 35 ng/ml for instant coffee ( $RSD\% \leq 12\%$ ) (Table 3). These concentrations

**Table 3**  
Furan concentration (ng/ml) in decaffeinated and instant coffee brews.

Coffee sample	Furan concentration (ng/ml)	
	Mean $\pm$ sd <sup>a</sup>	RSD (%)
<i>Decaffeinated coffee<sup>b</sup></i>		
Natural decaffeinated coffee #1	24 $\pm$ 3	12
Natural decaffeinated coffee #2	28 $\pm$ 2	7
Natural decaffeinated coffee #3	20 $\pm$ 2	12
Natural espresso coffee	35 $\pm$ 3	9
Natural cream espresso coffee	65 $\pm$ 2	3
Natural 100% Arabica coffee	14 $\pm$ 1	8
Natural low caffeine content coffee	18 $\pm$ 2	11
Coffee blend (50% natural and 50% torrefacto coffee)	40 $\pm$ 2	5
<i>Instant coffee<sup>c</sup></i>		
Natural instant coffee #1	12 $\pm$ 1	8
Natural instant coffee #2	20 $\pm$ 2	10
Natural decaffeinated instant coffee	28 $\pm$ 3	11
Natural pure 100% Colombian coffee	35 $\pm$ 2	6
Natural 100% Arabica coffee	32 $\pm$ 2	6

<sup>a</sup>  $n = 3$  replicates.

<sup>b</sup> Brewed coffee prepared using a drip coffee maker (9 ml/60 of boiling water).

<sup>c</sup> Instant coffee prepared following the manufacturer directions.

**Table 2**  
Furan concentration (ng/g) in brewed regular coffees obtained by drip coffee maker (filter) and espresso coffee machine.

Regular coffee	Furan concentration in coffee brew (ng/ml) <sup>a</sup>			
	Drip coffee maker		Espresso machine	
	Mean $\pm$ sd	RSD (%)	Mean $\pm$ sd	RSD (%)
Natural coffee #1	40 $\pm$ 3	8	70 $\pm$ 5	7
Natural coffee #2	29 $\pm$ 3	10	65 $\pm$ 5	8
Natural coffee #3	28 $\pm$ 3	11	43 $\pm$ 4	9
Natural coffee #4	48 $\pm$ 3	6	93 $\pm$ 6	6
Natural espresso coffee	30 $\pm$ 3	10	60 $\pm$ 5	9
Natural cream espresso coffee	39 $\pm$ 3	8	72 $\pm$ 6	8
Natural filter coffee	20 $\pm$ 3	15	50 $\pm$ 5	10
Natural Italian style coffee	43 $\pm$ 3	7	110 $\pm$ 8	7
Natural pure 100% Colombian coffee	66 $\pm$ 4	6	125 $\pm$ 6	5
Natural pure 100% African coffee	46 $\pm$ 3	7	82 $\pm$ 5	6
Natural pure 100% Brazilian coffee	78 $\pm$ 6	8	146 $\pm$ 12	8
Natural 100% Arabica coffee	24 $\pm$ 3	13	56 $\pm$ 5	8
Natural black 100% Arabica coffee	52 $\pm$ 3	6	101 $\pm$ 5	5
Coffee blend (70% natural and 30% torrefacto coffee)	35 $\pm$ 3	9	74 $\pm$ 6	8
Coffee blend (50% natural and 50% torrefacto coffee)	32 $\pm$ 3	9	59 $\pm$ 5	8

<sup>a</sup>  $n = 3$ .

were similar to those obtained for regular ground coffee and are in agreement with data described in the literature (Crews & Castle, 2007; FDA, 2008). For instance, the FDA reported furan concentrations from 34 to 53 ng/ml in decaffeinated coffee, while Crews et al. (2009) found levels ranging between 8 and 66 ng/ml. For instant coffee, concentrations ranging from 1 to 51 ng/ml have been reported (Crews & Castle, 2007; Crews et al., 2009; Zoller et al., 2007). Given that decaffeinated coffee is produced from green coffee, our results confirm that the roasting process is responsible for the formation of furan. In addition, the slightly lower concentrations of furan in instant coffee can be attributed to the lower coffee/water ratio (2 g coffee powder/60 ml of water) used for brew preparation.

#### 3.4. Analysis of furan in Nespresso coffee capsules

Recent years have witnessed a large increase in the consumption of coffee from packed capsules worldwide. This system guarantees fresh espresso coffee brews with a high-quality aroma and taste, and protects coffee from the damaging effects of light, air and humidity until brewing. The capsules in this system are filled with ground roasted coffee and are hermetically plastic-wrapped. A specialised coffee machine is required for each capsule brand. To determine the furan content in brews obtained with this system, eleven espresso coffee capsules from Nespresso (Table 4), one of the most widely consumed coffee capsules in the world, were selected and analysed in triplicate following the proposed method. For brewing coffee, a Nespresso coffee machine (Essenza) was used to obtain 40 ml of brews for coffee blends (*Ristretto*, *Arpeggio*, *Roma*, *Livanto*, *Capriccio*, *Volluto*, *Così*, *Decaffeinato* and *Decaffeinato intenso*) and 110 ml for Lungo coffees (*Vivalto Lungo*, *Decaffeinato Lungo* and *Finezza Lungo*). Furan concentrations for these coffee capsules ranged from 117 ng/ml for *Capriccio* to 244 ng/ml for *Decaffeinato intenso* (Table 4). These results were higher than those obtained in espresso brews of regular ground coffee (43–146 ng/ml), although similar coffee powder/water ratios were used (5.5 g/40 ml for Nespresso capsule and 9 g/60 ml for espresso coffee). This observation could be attributable to two factors, namely prevention of furan loss during handling as a result of the hermetic seal of the capsules, and the higher pressure applied by the Nespresso coffee machine (1850 kPa), which favours the extraction of this compound from the ground coffee. Regarding the *Lungo* coffee capsules, furan concentrations ranged from 68 to 102 ng/ml and were lower than those obtained with espresso capsules. This finding may be due to the higher dilution of the coffee brew (7 g coffee powder/110 ml of water) and the longer time period of brewing, which may increase the loss of furan.

**Table 4**  
Furan levels (ng/ml) in brewed coffee of Nespresso capsules.

Coffee blend	Nespresso capsule	Furan concentration (ng/ml)	
		Mean $\pm$ sd <sup>a</sup>	RSD (%)
Espresso blend <sup>b</sup>	<i>Ristretto</i>	230 $\pm$ 7	3
	<i>Arpeggio</i>	127 $\pm$ 8	6
	<i>Roma</i>	149 $\pm$ 8	5
	<i>Livanto</i>	193 $\pm$ 8	4
	<i>Capriccio</i>	117 $\pm$ 5	4
	<i>Volluto</i>	146 $\pm$ 6	4
	<i>Così</i>	132 $\pm$ 8	6
	<i>Decaffeinato</i>	158 $\pm$ 8	5
	<i>Decaffeinato intenso</i>	244 $\pm$ 12	5
	Lungo blend <sup>c</sup>	<i>Vivalto Lungo</i>	83 $\pm$ 5
<i>Decaffeinato Lungo</i>		102 $\pm$ 6	6
<i>Finezza Lungo</i>		68 $\pm$ 4	6

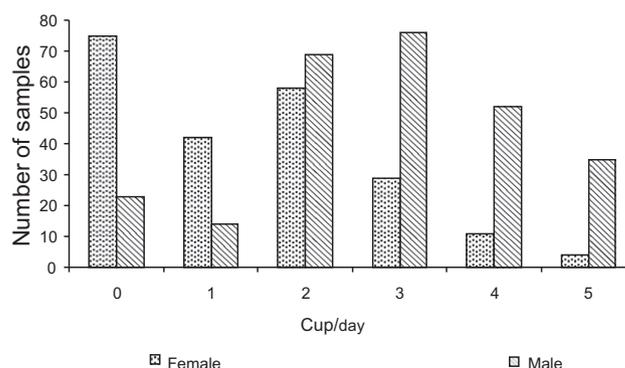
<sup>a</sup> n = 3 replicates.

<sup>b</sup> Final volume of brewed coffee: 40 ml.

<sup>c</sup> Volume of coffee brew 110 ml.

#### 3.5. Furan intake from coffee consumption

At present, the average coffee consumption in Spain reported by the Spanish Coffee Federation is 599 cups of coffee per person and year (FEC, 2007), which corresponds to 1.64 cups of coffee per person and day. These values are consistent with data provided by the Spanish Government (MAPA, 2007) and the International Coffee Organization (ICO, 2008), which reported an average coffee consumption of 4.5 kg of coffee beans per person and year for the last three years in Spain. On the basis of these data and on the furan content obtained in this study for espresso and drip coffee brews, we estimated the daily furan intake to be from 1.97 to 14.4  $\mu$ g/person, which implies exposure of between 0.03 and 0.21  $\mu$ g/kg body weight (bw)/day of this compound, assuming an average adult weight of 70 kg. Although these data are useful to establish the overall exposure of the total population to furan, we considered it of interest to obtain a more detailed picture of coffee consumption habits of people living and working in a large city. For this purpose, a 24-h recall questionnaire was completed from a representative sample of 488 participants aged from 19 to 58 years from five districts of Barcelona (Spain) (219 females and 269 males). Each participant was asked about his/her coffee drinking habits, such as the amount of coffee (cups of coffee/day) ingested in the previous 24 h, type of coffee and brewing procedure, and also some personal information, such as age, sex, address, citizenship and body weight. Fig. 2 shows the results of the coffee consumption survey. Around 20% of the participants were not habitual coffee drinkers. The average daily coffee consumption was 3.1 and 2.1 cups of coffee for males and females, respectively. This consumption rate implies a daily furan intake for males from 3.6 to 26.3  $\mu$ g/person and for females between 2.4 and 17.5  $\mu$ g/person. These findings indicate exposure to furan of 0.05 to 0.38  $\mu$ g/kg bw/day for males and between 0.03 and 0.25  $\mu$ g/kg bw/day for females. These data are only slightly higher than those obtained using the official Spanish average consumption data. These results agree with those reported by the US FDA (0.15  $\mu$ g/kg bw/day) (Morehouse et al., 2008) and EFSA (<0.03–1.65  $\mu$ g/kg bw/day) (EFSA, 2004) and are much lower than the non-observed adverse effect level (NOAEL), which was established at 2 mg/kg bw/day for experimental animals (Moser, Foley, Burnett, Goldsworthy, & Maronpot, 2009). Since the acceptable daily intake (ADI) was estimated to be 2  $\mu$ g/kg bw/day within a safety factor of 1000 (Kuballa et al., 2005), the maximum furan intake found in this study (0.38  $\mu$ g/kg bw/day) was around five times lower than this value. Consequently, we conclude that the estimated risk from coffee consumption, which is the biggest source of furan exposure for adults, is low.



**Fig. 2.** Coffee consumption in Barcelona (Spain) obtained from a 24-h recall questionnaire (488 participants).

#### 4. Conclusions

Furan formation was not observed during the analysis of brewed coffee by automatic HS-SPME combined with GC-MS using a low extraction temperature (30 °C) and a short time (20 min). The roasting time and temperature applied to green coffee beans affect the final furan content of coffee. Therefore, we recommend gently roasting conditions (140 °C for 20 min) to reduce furan formation. The furan concentration levels found in regular ground coffee (20–146 ng/ml), decaffeinated (14–65 ng/ml) and instant coffee (12–35 ng/ml) brews commercialised in Spain were in agreement with those reported in the literature, showing the highest concentrations in espresso brews. Nevertheless, it must be mentioned that furan concentrations in brews from Nespresso coffee capsules (117–244 ng/ml) were higher than those obtained for regular ground coffee. The daily furan intake from coffee consumption was estimated to be 0.25 and 0.38 µg/kg bw for males and females, respectively. These values are lower than the established acceptable daily intake of 2 µg/kg bw.

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