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# 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.

## 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 ($C_4H_4O$) 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, Lauryssen, & Kramerc, 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 [$^2H_4$] furan (furan-$d_4$) 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-$d_4$ 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-$d_4$ 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-$d_4$ 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-$d_4$ 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 intense, Vivalto Lungo, Decaffeinato Lungo and Finezzo 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 Pré 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 autosampler (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-$d_4$, respectively. For confirmation, $m/z$ 39 $[M–CHO]^+$ 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