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Original research article

Furan, 2-methylfuran and 3-methylfuran in coffee on the Canadian market

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ABSTRACT

Forty samples of commercially brewed coffee samples from 3 national chains and 48 samples of non-brewed coffee samples were collected from retail outlets located in a single Canadian city and analyzed for furan, 2-methylfuran and 3-methylfuran by headspace gas chromatography–mass spectrometry. Non-brewed samples were analyzed "as is" and some were also analyzed after brewing in the laboratory using several preparation techniques. The three analytes were detected in all samples. The rank order of concentrations for all samples was 2-methylfuran >furan > 3-methylfuran. Ground coffee types sampled included regular ground, decaffeinated and cartridge type coffee; mean furan concentrations for these coffee types were 2200, 2450 and 2360 ng/g; 9470, 10400 and 10700 ng/g for 2-methylfuran; and 447, 463 and 508 ng/g for 3-methylfuran respectively. Mean levels for both regular and decaffeinated instant coffee powders were lower with mean furan concentrations of 233 and 327 ng/g; 1600 and 1800 ng/g for 2-methylfuran and 72.9 and 75.2 ng/g for 3-methylfuran respectively. Commercially brewed coffee types sampled included regular ground, decaffeinated and espresso coffee; mean furan concentrations for these coffee types were 38.7, 53.1 and 157 ng/g; 172, 184 and 583 ng/g for 2-methylfuran; and 6.4, 6.7 and 19 ng/g for 3-methylfuran respectively. Brewing coffee samples in laboratory as per manufacturers' instructions resulted in 27–85% loss of furans—as compared to not brewed samples, loss of methyl furans exceeded that of furan by 10–15%. Brewed coffee stored/standing for up to 30 min resulted in further losses of furans, from 3 to 47%. Degree of loss was not analyte dependent but was highly influenced by storage conditions.

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1. Introduction

Furan has been found to form in foods that undergo heat treatment (Maga, 1979; U.S. Food and Drug Administration, 2009). Furan is ubiquitous in heat-treated foods at ng/g levels due to the multiple paths of formation from precursors naturally present in the food(Becalski and Seaman, 2005; Limacher et al., 2007, 2008; Mark et al., 2006; Perez-Locas and Yaylayan, 2004).

Occurrence, analysis and formation of furan has been subject to numerous reviews, *e.g.* (Crews and Castle, 2007; Blank, 2008; Guenther, 2012; Moro et al., 2012).

Coffee consumption is likely a main contributor to furan exposure from dietary sources for adults. Despite temperatures exceeding 200 °C during the roasting process, furan with its boiling point of 31 °C has been found in roasted coffee in amounts above 5 μg/g (European Food Safety Authority, 2011)

In consideration of the above, in the last decade several investigations focused on levels of furan in roasted and brewed coffee were undertaken (Zoller et al., 2007; Weizenegger et al., 2012; Food Standards Agency, 2013; Crews et al., 2009). Some of these studies also attempted to quantify losses of furan upon brewing and storage (Mesias and Morales, 2014; Altaki et al., 2011; Arisseto et al., 2011; Guenther et al., 2010; Kim et al., 2009).

Furan is classified as possibly carcinogenic to humans (Group 2B) (International Agency for Research on Cancer, 1995). The harmful effects of furan results from cytochrome P450 catalyzed furan ring oxidation which yields a reactive *cis*-butene-1,4-dial

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metabolite which in turn can bind to various cellular components (Peterson, 2013)

The 2- and 3-methyl substituted furan are metabolically activated in a similar fashion as the parent furan yielding α, β-unsaturated dialdehydes/aldehydoketone (Ravindranath et al., 1984). These methyl analogs of furans are known to occur in foods and especially coffee (Maga, 1979; Test, 2004) and were shown to form in model reactions, similar to furan, from precursors commonly found foods (Adams et al., 2011; Becalski and Seaman, 2005; Limacher et al., 2008). Yet, despite these findings, scarce, compared to furan, attention was drawn to the issue of presence of methyl furans in foods. Recently, a 28-day gavage toxicity studies have shown that the main target of 2-methylfuran and 3-methylfuran in rats is the liver as is the case in furan itself (Gill et al., 2014, 2015). Our previous survey of furan and 2- and 3-methylfurans in foods indicated that coffee might be an important source of methyl furans in the diet (Becalski et al., 2010). In this work we are presenting results of a pilot survey for furan, 2-methylfuran and 3-methylfuran in 40 samples of commercially brewed coffee samples from 3 national chains and 48 samples of various types of retail non-brewed coffee samples which were collected from outlets located in a single city and analyzed by headspace gas chromatography–mass spectrometry. Non-brewed samples were analyzed "as is" and some were also analyzed after brewing in the laboratory using several preparation techniques. Selected samples were also subjected to simulated storage to ascertain loss of analytes over time. Our survey represents the most detailed investigation of coffee for the presence of furan, 2-methylfuran and 3-methylfuran to date.

After this work was completed, results of the two investigations of furan and alkylfurans in canned foods and coffee were published. The alkylfurans included 2-methyl, 2-ethyl-, 2-pentyl furan and 2,5-dimethylfuran (Fromberg et al., 2014) and 2- methyl, 2-vinyl-, 2-methoxymethyl furan and 2,5-dimethylfuran (Chaichi et al., 2015). Both investigations used methods which utilized d_4-furan as the sole internal standard.

2. Materials and methods

2.1. Standards

Furan 99%, 2-methylfuran 99% and d_4-furan, isotopic purity >99 atom% D were from Sigma–Aldrich (St. Louis, MO). 3-Methylfuran 98% was obtained from TCI America (Portland, OR) while 2-methylfuran-d_3 (methyl-d_3) 95% and 3-methylfuran-d_3 (methyl-d_3) 95% were supplied by Toronto Research Chemicals Inc. (Toronto, ON, Canada).

2.2. Method

The samples were analyzed by headspace gas chromatography–mass spectrometry (Agilent 6890/5975C Inert XL) with CTC Analytics CombiPAL injector equipped with a 2.5 mL headspace syringe (Palo Alto, CA, USA) using stable isotope dilution approach for all three analytes as described previously (Becalski et al., 2010). The limit of detection (LOD) in brewed coffee was 0.10 ng/g and in ground coffee was 3 ng/g for all analytes. The method employed a calibration range of approximately 0.4–250 ng/mL.

2.3. Commercial foods

a. Retail ground and instant coffee samples were analyzed as is, without brewing.

A total of 48 coffee samples were purchased from retail locations in Winnipeg, Manitoba between March and July, 2012, principally based on marketing research company (AC Nielson) market share data. Samples included regular ground coffee ($n = 17$), decaffeinated ground ($n = 10$), instant powder ($n = 9$), decaffeinated instant powder ($n = 9$) coffee, and single use cartridges ($n = 3$). Roasted whole bean coffee was not included at this stage of the survey. Samples were stored in the original containers at room temperature prior to analysis. All samples were analyzed prior to the stated best before dates.

a. Retail ***brewed*** coffee samples

Forty commercially brewed coffees were collected over 4 sampling days from each of three outlets in July, 2012. Two separate locations of each outlet were sampled. Samples included drip style regular roast and decaffeinated coffee and espresso coffee. One outlet was also sampled for a drip brewed regular coffee advertised as "bold" style. No information was available on how long the brewed coffee samples were stored at the retail locations prior to sample collection. Brewed coffee samples were transferred from the original, covered container into pre-weighed, air tight, 125 mL containers containing a magnetic stir-bar within 5–10 min after retail purchase. The brewed coffee samples were then transported back to the laboratory in a cooler over ice, stored at 4 °C and analyzed within 48 h.

2.4. Typical sample preparation

Non-brewed ground and instant coffee samples (0.5 g) were weighed directly into 20 mL headspace vials containing sodium sulfate. 12.5 mL of water was added to the vial, vial was sealed with a septum and internal standard of labeled furans was added.

Randomly selected coffee samples were also brewed in the laboratory as per manufacturers' instructions using: (a) Bodum 8 cup French press brazil coffee maker model 10938, (50 g of coffee/1000 mL water) this brewing method incorporated a 5 min brewing time, (b) Black and Decker 5 cup drip coffee maker, model DCM675BFC (32 g of coffee/740 mL water), (c) Flavia single cup maker (7–9 g of coffee/210 mL water). Brewed coffee samples were immediately transferred into sample jar with a lid (~120 mL) containing a magnetic stirring bar. Instant coffee was brewed directly in the jar (1.4 g coffee/120 mL). A labeled furans internal standard was added and the sample was equilibrated by stirring in a closed jar at room temperature for 30 min. A 12.5 mL aliquot was pipetted to a 20 mL headspace vial containing sodium sulfate, sealed, and transferred to GC for analysis. Some samples were diluted with deionized water and re-analyzed if their peak area for 2-methylfuran exceeded the peak area of the employed calibration curve.

2.5. Positive samples

Each analytical *non-brewed* samples batch included positive control samples of regular ground coffee "A" (furan, 2-methylfuran and 3-methylfuran concentration; 1680, 6930 and 350 ng/g, respectively) or regular instant coffee powder "B" (furan, 2-methylfuran and 3-methylfuran concentration; 101, 278 and 20.2 ng/g, respectively)

Each analytical *brewed* samples batch included a positive control sample of regular instant coffee powder "B" which was brewed in duplicate with each sample set in accordance with manufacturer instructions. One duplicate was spiked with furan, 2-methylfuran and 3-methylfuran at a level of approximately 10 ng/g.

2.6. Spiking experiments

Recovery experiments were performed with each non-brewed sample set using a spiked control sample consisting of regular

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