



Furan in coffee: Pilot studies on formation at roasting and losses during production steps and consumer handling

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Furan in coffee: Pilot studies on formation during roasting and losses during production steps and consumer handling

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Abstract

The occurrence of furan in some food products has been known for a few decades already and it has been re-confirmed in more recent investigations that furan is present in a variety of foodstuffs. This list of products includes roasted coffee which has been shown to generate furan as a result of the heat treatment during the roasting reactions which are applied to achieve the desired aroma and flavor profile of a roasted coffee. The objective of this study was to provide data to better understand the available data of furan in coffee, to understand the kinetics of furan generated during roasting and to estimate the reduction of furan levels afterwards due to subsequent processing steps and consumer handling. Finally, the study was meant as a contribution to establish exposure data on the basis of scientific data at the stage of coffee consumption. This paper shows that the formation of furan during roasting is dependent on roasting conditions and is therefore directly linked to achieving targeted flavor profiles. Furthermore it is demonstrated that modifications in process conditions to potentially reduce furan levels may have the opposite effect on other undesired reaction products of the roasting chemistry like e.g. acrylamide. Due to the high volatility of furan any subsequent processing step or consumer handling has an impact on the level of furan. As a guidance from this study and in consideration of the identified losses of each process and handling step on basis of the trial conditions it is estimated that only approximately 10% of the initially generated furan during roasting gets into the cup of coffee for consumption.

Keywords: furan, coffee, formation, analysis, exposure

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Introduction

It has been known to the scientific community for a number of years that furan is present in coffee as part of the coffee volatile aroma components which are generated during roasting. More recent risk assessments have been initiated by a report from the US Food and Drug Administration in 2004. Amongst others due to improved analytical methodology its surveys have identified the occurrence of furan in a number of food products which have undergone a heat treatment. This range of foodstuffs includes among others coffee products. The presence of furan is a potential concern as, based on animal trials, furan is considered possibly carcinogenic to humans (IARC 1995).

There has been little knowledge available on the relationship between coffee roasting conditions and resulting furan formation. Moreover, the impact of subsequent processing steps and consumer handling of the product on levels of furan is not well understood. Finally, due to the high volatility of furan, sampling has been shown to be crucial to obtain reliable results. Therefore it has been difficult to interpret individual data on furan in coffees which have been produced under different conditions and sampled at different process steps. As such, the contribution of coffee as consumed to the human exposure to furan has been very difficult to assess from analysis data for roasted whole beans or roast & ground coffee and only been possible with analysis of coffee brews ready for consumption.

This paper is meant to increase knowledge on formation and disappearance of furan in different stages of coffee roasting and handling. Intentionally the investigations have been limited to bench top or pilot plant screening trials to estimate the magnitude of impact from individual process steps. The trials have been designed to simulate typical commercial plant conditions and average consumer handlings but are not covering the full range of all process variables and conditions. Overall, it is meant as contribution to establish exposure data on basis of scientific data at the stage of coffee consumption.

Materials and methods

Green coffee

The roasting experiments to investigate furan formation during roasting were performed with the three major types of green coffees: wet processed Arabica coffee (*Coffea arabica*) (represented by a sample of a Colombian green coffee); dry processed Arabica coffee (*C. Arabica*, represented by a sample of a Brazilian green coffee); and dry processed Robusta coffee (*Coffea canephora robusta*, represented by a Vietnamese green coffee). Studies to investigate the losses of furan in subsequent processing steps were typically performed by using a sample of a Brazilian green coffee.

Pilot plant roaster

The roasting experiments were executed on a Neuhaus Neotec RFB-10 pilot plant roaster with a batch size of typically 1.5 kg.

Analysis of degree of roast

The degree of roast was measured by light reflectance using a Lange colorimeter. No harmonized methodology across the European coffee industry is available and the coffee industry is applying individual calibration curves taking the roast color as characterization of the degree of roast. LRUs (Lange reflectance units) as used for this study would be 100-110 LRUs for a very light roast and 40-50 LRUs for a very dark roast.

Furan analysis

Furan was analyzed according to the FDA-method „Determination of furan in foods” using headspace gas chromatography mass spectrometry (GC/MS). Here, furan is quantified by using a standard addition curve, where the concentration of furan in fortified test portions is plotted versus the furan/d4-furan response factors. The amounts of furan and/or d4-furan added to the test portions are based on an estimate (x_0) of the furan concentration in the food.

Chemicals

Furan was purchased from Acros Organics (Niederau, Germany) and d4-furan from Isotec (Miami, USA). Methanol was from Sigma-Aldrich (Seelze, Germany). All other chemicals used were of analytical grade.

Pre-treatment

Whole coffee beans are frozen at -20 °C before milling. The homogenization is performed under cooling with liquid nitrogen using a Retch mill, mesh size 0.5 µm.

Estimation of the furan level in the coffee sample

For the one-point estimate of the concentration of furan in the sample 0.5 mg of coffee is weight into a 20 ml headspace vial. After addition of 4.5 ml water the headspace vial is sealed. The sample is fortified through the septum of the seal with 50 µl of internal standard (IS) solution (d4-furan, $c = 30 \mu\text{g ml}^{-1}$). After shaking, the sample is analyzed by headspace GC/MS as described below. By using the integrated response ratio for furan/d4-furan (m/z 68/72) and the ng amount of the IS, the ng amount of furan in the coffee sample is estimated.

Standard addition procedure

0.5 g of sample is weight into seven 20 ml headspace vials respectively. To every vial 4.5 ml of water is added and the vials are than sealed. IS solution is added ($c = 30 \mu\text{g ml}^{-1}$) to all of the seven vials at the level of the estimated furan level (x_0) in the sample. To two of the samples furan (furan standard, $c = 30 \mu\text{g ml}^{-1}$) is added at a level of 0.5 times of the estimated furan level ($0.5 x_0$) through the septum of the seal. Two further vials are fortified with furan at 1 time ($1 x_0$) and 2 times ($2 x_0$) the estimated furan level of the coffee sample, respectively. Three of the seven vials were not fortified with furan (x_0). After shaking all seven vials are transferred to GC/MS analysis as described below.

GC/MS analysis

GC/MS analysis is performed using a Varian Saturn 2100 GC/MS (Varian, Darmstadt, Germany) coupled to a CTC Analytics Combi PAL Autosampler-Headspace with incubator oven (CTC Analytics AG, Zwingen, Switzerland).

The vials are incubated for 30 min in the headspace oven at 50 °C before injection (transfer line: 230 °C, injection volume: 1000 μl , injector temperature: 200 °C, 1 min splitless, split ratio 1:20, carrier gas: helium). The chromatographic separation is made on a HP-PLOT Q column (15 m x 0.32 mm ID x 20 μm) (Agilent Technologies, Waldbronn, Germany). The following temperature profile is used: 0.1 min at 50°C, 10°C/min to 190°C, 30°C/min to 225 °C, hold for 1 min. The detection is made in the scan mode, scan range: m/z 35 to 85, MSD delay time: 5 min, trap temperature: 150 °C. The following ions are used for quantification: D4-furan: m/z 72 and 42, furan: m/z 68 and 39.

For calculation of the furan content of the coffee sample the integrated responses for m/z 68 (for furan) and m/z 72 (for d4-furan) are determined and the response ratio is calculated, m/z 68 divided by m/z 72. All test portions ($0 x_0$) and calibration standards ($0.5 x_0$, $1 x_0$, $2 x_0$) are subjected to a linear regression analysis where the x-axis equals the ng amount of furan added to the test portion and the y-axis equals the response ratios. The slope and intercept of the calibration curve is determined and the equation is solved for $0 x_0$ at y equal to zero. In order to determine the $\mu\text{g kg}^{-1}$ amount of furan in the sample $0 x_0$ is divided by the test portion amount in grams.

Validation data

The expanded measurement uncertainty (level of confidence 95%, coverage factor $k=2$) for the determination of furan in coffee ($x = 3200 \mu\text{g/kg}$) using the described method is calculated to be 20 %. The recovery rate of furan spike to a sample is usually between 90 and 120 %.

In a ring trial organised by the German § 64 working group "furan analysis", a z-score of 0.48 was achieved for the analysis of furan in coffee ($x = 2840 \mu\text{g/kg}$) using the described method. The repeatability and the reproducibility of the analysis of furan in coffee was calculated to be 9 and 19 %.

Artifacts of the furan analysis

Sample weight. According to the FDA method „Determination of furan in foods” 5 g of sample is used for the analysis. However, it could be shown that for samples containing higher amounts of furan, like coffee, the sample weight has to be reduced to 0.5 g in order to receive robust and comparable results.

Isotope dilution method versus standard addition procedure. It could be shown that for coffee the calculation of the furan content can lead to significant higher or lower amounts (up to 50 %) when using the simple isotope dilution method with d4-furan as internal standard. It seems that for the analysis of samples with high furan content (higher than 500 $\mu\text{g kg}^{-1}$), like coffee, the standard addition procedure is essential to obtain reliable results.

Choose of the analytical column. Besides the HP-PLOT Q column the use of a HP-INNOWax column (Agilent Technologies, Waldbronn, Germany) was tested. For some samples, significant higher furan levels (up to 50%) were calculated when using the HP-INNOWax column. The background of this phenomenon could not be identified. In order to obtain comparable results, the column proposed by the FDA for the analysis of furan was used in our experiments.

Results and discussion

Formation of furan at roasting

Green Coffee

The selected green coffees representing the major types of green coffee have been roasted under varying roasting conditions to result in a range of roast colors for a constant roast time. The resulting furan levels in the roasted coffees are depending on the roast colors (Figure 1). In contrast, the results do not indicate that the furan formation potential is significantly varying for different green coffee types. The results for the different green coffee types at constant roast times are within a range which represents the measurement uncertainty (95% confidence level) of the analytical methodology of $\pm 20\%$. The conclusion is consistent with the investigations of La Pera et al. (2009). This study has covered green coffees from 6 producing countries and no correlation was observed between furan levels after roasting and provenance of green coffee. On this basis no further studies have been made to investigate in differentiation among green coffee origins of the same type.

Here Figure 1

Roast conditions

1 European roast coffees cover a broad range of flavor profiles according to the different
2 regional preferences and are designed to meet specific consumer groups' acceptances. The
3 individual flavor profiles are controlled by specifying the green coffee blend, the degree of
4 roast (typically measured by light reflectance or color measurements) and roast time. When
5 analyzing pilot plant samples which have been roasted to cover qualities from light to dark
6 roasted and from fast to long roasted coffees it can be seen that darker roast colors
7 (indicated by lower LRU-values) and longer roast times in tendency result in higher levels of
8 furan (Figure 2). Statistical analysis through modeling shows that both the roast colour effect
9 and the roast time effect are statistically significant at 99% confidence level. Options to
10 modify process conditions with the intent to reduce to furan levels after roasting are very
11 limited because from one side the mentioned parameters need to be set in tight ranges to
12 maintain the product identity of an existing product and to achieve its typical flavor profile. On
13 the other side it needs to be considered that modifying process conditions to reduce one
14 undesired constituent may increase at the same time the level of another undesired
15 constituent. Lantz et al. (2006) have shown that acrylamide levels in roast coffee are
16 decreasing with increasing degree of roast and with longer roast time. These results have
17 been confirmed in this study (Figure 3). Acrylamide is showing the opposite relationships with
18 the same process parameters compared to those which have been found for furan in this
19 study.
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33 Here Figure 2 und Figure 3
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36 Accordingly, the indications from this study are that process conditions to result in low
37 acrylamide levels tend to lead to higher furan levels (Figure 4)
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44 The full range of furan levels for roast whole beans from this study of 2000-7000 $\mu\text{g kg}^{-1}$ is
45 consistent with published data which are reporting levels of 3510-6100 $\mu\text{g kg}^{-1}$ (Kuballa et al.
46 2005).
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52 Plant processing steps
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55 The purpose of this study was to assess the impact of coffee production processes and
56 consumer handling on furan levels in a cup of coffee. Therefore pilot scale trials have been
57 conducted, simulating typical conditions of commercial plant processes and consumer
58 handling. In the scope of these investigations it was not intended to cover the full range of all
59 variables and technology configurations. Additional investigations may be required for
60 specific cases and to fully understand the levels of furan in all commercial coffee products.

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Roasting in commercial plants is typically followed by storage of the roasted whole beans of up to several hours before grinding - with maximum tolerable storage time depending on individual quality criteria. For the purposes of these tests the effect of this storage was simulated by storing freshly roasted coffee in open bags at defined temperatures and sealing the bags after defined periods to stop potential furan losses. Analyzing samples with up to 96 hours of storing at ambient temperatures and at 40 °C shows that results are constant over time within a range of +/- 20% which represents the measurement uncertainty (95% confidence level) of the analytical methodology. This indicates that storage of whole beans of up to 96 hours seems not influenced by temperature. Moreover, such a storage does not seem to significantly impact the furan levels in an open system (Figure 5).

Here Figure 5

Grinding

In order to ensure a consistent brew performance and extractability the roast whole beans are ground to controlled mean particle sizes. This grinding process opens the cell structures of the beans and it is known that grinding potentially causes significant losses of volatile aroma compounds. The level of aroma loss depends on specific process conditions. It was therefore to be expected that furan levels will be reduced as well during grinding. Tests to estimate the magnitude of furan losses at grinding have been done with two different types of laboratory grinders (Mahlkönig: Model DK5LS; Modern Process Equipment Inc: Model MPE 4555). It is shown that in parallel to achieving smaller mean particle size the furan losses are increasing (Figure 6). At typical European drip filter grind sizes of 350–500 µm, furan losses are estimated to be in the order of magnitude of 40%. With starting levels of ca. 2000–7000 µg kg⁻¹ in roast whole beans depending on roasting conditions this would lead to levels of 1200–4200 µg kg⁻¹ after grinding. This range is in line with levels as published previously (Kuballa et al. 2005; Zoller et al. 2007) with 790–3390 µg kg⁻¹ and 959–5938 µg kg⁻¹ respectively.

Here Figure 6

Degassing

CO₂ is generated in coffee beans as part of the roasting reactions. Roast & ground coffees which are intended to get packed in vacuum packs need to degas before packing. This is to allow the CO₂ to escape from the beans in order to avoid that this takes place inside the vacuum bag which would result in a pressure build up and softening of the pack. The required degassing times are mainly depending on bean type, roast conditions, moisture and particle size. For this study, two roast and ground samples were placed into open plastic

1 bags directly after roasting. After defined degassing periods at ambient temperatures the
2 bags were sealed and prepared for analysis. Results show that degassing of up to four
3 hours resulted in a furan reduction of app. 20% (Figure 6).
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7 The degree of reduction for commercial plant degassing processes will additionally depend
8 on product temperature after grinding. Furthermore and depending on specific facilities
9 configurations the effect of the transport from the degassing silo to the packaging machine
10 needs to be assessed as a potential basis for additional furan losses.
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15 Shelf life

16 It was investigated if furan levels in packed coffee products would change during shelf life.
17 For this, vacuum packed roast & ground coffee was stored at 4 °C and at ambient
18 temperatures (Figure 7). As shown all results are within a range of +/- 20% which represents
19 measurement uncertainty (95% confidence level) of the analytical methodology. It therefore
20 can be concluded that levels of furan for roast coffee are not significantly changing during
21 the first three month of the shelf life.
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33 Consumer handling

36 Kitchen life

37 Typically, in Europe roast coffee is packed and sold in vacuum packs or soft packs with pack
38 quantities for more than one brewing / serving. The packs are being used up over a longer
39 time period. Therefore a product sample taken out of a freshly opened product should not be
40 seen as representative for an exposure estimate. It needs to be understood, how the
41 'kitchen life' –the typical house hold use up period- impacts the furan levels. There are
42 different consumer practices in place from leaving packs open to tightly closing packs during
43 the use-up period. Specifically and for quality preservation reasons it is often recommended
44 to store opened pack in a refrigerator to maintain the initial and desired aroma level.
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50 In a simulation of a typical consumer handling a 500 g pack of a roast coffee the product
51 was opened, followed by taking a defined quantity of product (25 g) out of the pack,
52 reclosing the pack tightly (with a clip) and storing under different conditions (ambient or in a
53 refrigerator). This procedure was repeated once every day.
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58 The furan level in the refrigerated coffee remained almost constant over the usage period. In
59 contrast, the products which have been stored at ambient temperatures (both whole beans
60 and ground coffee) are showing a significant decrease in furan levels in the order of
magnitude of 20-25% over a seven days usage period.

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6 Brewing

7 Furan exposure estimates require realistic brews as made for consumption. It has to be
8 understood that preparing a cup of coffee for consumption is not harmonized at all and a
9 number of different aspects need to be considered, especially as furan is very volatile and
10 has low water solubility properties. The most important aspects cover:
11

- 12 - *Type of preparation.* Brewing of roast & ground coffee on drip filter machines is still
13 typical for the majority of the European coffee consumption. Other methods like fully
14 automatic machines (which include grinding of roast whole beans), espresso brewing
15 (with typically higher coffee usage per beverage volume) and machines based on
16 coffee pads, disks or capsules are preparing single cups for immediate consumption.
17 For these coffees storage / warming of the beverages on hot plates until
18 consumption does not need to be considered.
19
- 20 - *Brew recipe.* The brew recipe is varying significantly from consumer to consumer and
21 typically consumers are preparing their coffees according to individual preferences.
22 There may also be regional trends to be considered. Espresso beverages are
23 typically based on higher brew recipes (*i.e.* same amount of coffee powder is used
24 for a lower beverage volume) resulting in higher levels of coffee solubles (aroma,
25 volatile compounds etc.) per volume of beverage. For exposure estimates of furan
26 these higher Espresso levels should not be seen as typical for the total coffee intake.
27 Furan levels should only be considered together with the corresponding consumption
28 rates and volumes of the specific type of coffee preparation.
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41 The effect of brewing and the corresponding transfer of furan from a roast & ground coffee
42 product into the beverage have been investigated intensively by Kuballa et al. (2005) and La
43 Pera et al. (2009). Both studies showed significant losses for furan at the brewing step. On
44 basis of this existing data base for the effect of brewing, in this study only one brewing
45 experiment per type of brewing equipment has been performed to confirm the magnitude of
46 the effect. On basis of the data from this study together with the data from the other studies
47 it can be assumed that in average and across different brewer types 50-60% of the furan in
48 the roast & ground coffee is lost when brewing coffee.
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56 Here Table 1
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59 Our results are within the reported variation from earlier studies (Table 1). Variability of
60 results for data from different brewing methodologies are probably due to differences in
design of equipment in terms of brewing temperatures, brewing time and how closed the

1 system is to retain volatile components, including furan. For a specific brewing system the
2 sampling procedure needs to be specified in detail to allow reproducible data. In order to get
3 relevant data for consumer exposure estimates it is recommended to take the brew samples
4 after a defined cooling period to get to typical drinking temperature of the beverage.
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7 Published data for furan in the prepared beverage from roast coffee (Kuballa et al. 2005;
8 Zoller et al. 2007; Morehouse et al. 2008) show a range from 8-199 $\mu\text{g l}^{-1}$. The upper end of
9 the range has been analyzed from espresso type beverages which –due to the higher
10 recipes of typically 100-140 g coffee per liter compared to 30-60 g coffee per liter for a
11 standard cup of coffee– results in higher levels of furan. For an exposure estimate from an
12 European perspective it has to be considered though that the average consumption rate and
13 volume for espresso coffee is much lower than the consumption rate of a standard cup of
14 coffee.
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22 Hot plate / cup

23 Coffee beverages brewed on drip filter machines are typically prepared to be consumed over
24 a longer time period after brewing while the beverage is kept hot on a heating plate. This
25 may cause furan losses depending on time, temperature and design of the coffee machine.
26 For this study a trial was conducted to analyze the change in furan level in the brew within
27 the first 60 min. This period covers the timeframe which is typically provided as guidance on
28 how long coffee brew can be kept without significantly compromising in quality. After 60 min
29 the furan level in the brew has decreased to app. 50% of the initial level as measured
30 immediately after brewing. Assuming that brews in average are kept hot for 30 min, this
31 would lead to reduction rates of 35%.
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40 The effect on furan loss by pouring the beverage into a cup including the cooling of the
41 beverage in the cup to normal drinking temperatures was estimated in a separate trial. This
42 showed that furan in a cup is reduced by 25% after 30 min. As coffee is typically consumed
43 within the first few minutes, a reduction could be assumed in the order of magnitude of 10%.
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48 Here Figure 9
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51 These findings are consistent with results reported from other studies. Goldmann et al
52 (2005) and Zoller et al. (2007) have analysed brews when exposed to the atmosphere at
53 room temperature. With higher losses in the beginning and diminishing losses with time after
54 1 hour the furan content in the brew was reduced to 30% respectively 50% of the initial level
55 after brewing. Higher losses have been analyzed by La Pera et al. (2009) when maintaining
56 the temperature of the brew. Depending on the temperature 50% losses of the initial furan
57 content have been achieved after 10 min or less.
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Conclusions

In order to provide consumers with products which they can enjoy due to their levels of volatile aroma compounds the industry has implemented technologies and processes to protect products against aroma losses. As furan is a volatile component as well, all these measures will also reduce furan losses during production

The studies described in this paper have shown that furan is generated as a function of the process parameter 'degree of roast' and 'roast time' with no significant options to modify these process parameters to achieve lower furan levels. Firstly, these parameters have to be kept in narrow ranges to maintain the quality/flavor profile of a product. Secondly, these same parameters are known factors to influence the levels of acrylamide in coffee in an opposite direction. Acrylamide is another, undesired component generated when roasting coffee.

During subsequent processing and consumer handling the furan levels will decrease significantly and all factors need to be assessed when trying to estimate typical levels of furan in coffee when consumed. For this assessment it needs to be noted that 'best practices' for commercial plant processes and advice to consumers to retain the desired volatile aroma component in the coffee product will also reduce the losses for furan in parallel. As a guidance from this study and in consideration of the estimated typical impact factors of each process and handling step the total furan losses from roasting to final cup for consumption are indicated to be around 90%.

Here Table 2

On basis of the full range of furan levels after roasting of this study and assuming a brew recipe of 50 g coffee per liter it would calculate to in-cup furan levels of 10–35 $\mu\text{g l}^{-1}$, which is at the low end of published data (ranging from 8– 199 $\mu\text{g l}^{-1}$). The discrepancy can be explained as the published data includes brews based on higher brew recipes and may not consider all impacting factors from a typical consumer handling in their results as outlined in this study.

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4 Figure captions
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7 Figure 1. Effect of different types of green coffee on furan level at different roast colours
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10 Figure 2. / Figure 3. Effect of roast color and roast time on acrylamide level and on furan
11 level
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14 Figure 4. Correlation between furan and acrylamide levels formed in roasted coffee at
15 different roasting conditions
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18 Figure 5. Effect of storage time and temperature on furan levels in roast coffee whole beans
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21 Figure 6. Effect of grinding and degassing time to different mean particle sizes on furan level
22 in the ground coffee
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25 Figure 7. Effect of shelf life on furan level in vacuum packed roast & ground products
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28 Figure 8. Effect of a simulated 'kitchen life' after first opening of a pack on furan level
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31 Figure 9. Effect of keeping brewed coffee hot on a hot plate and effect of pouring the brewed
32 coffee into a cup
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For Peer Review Only

Figure 1

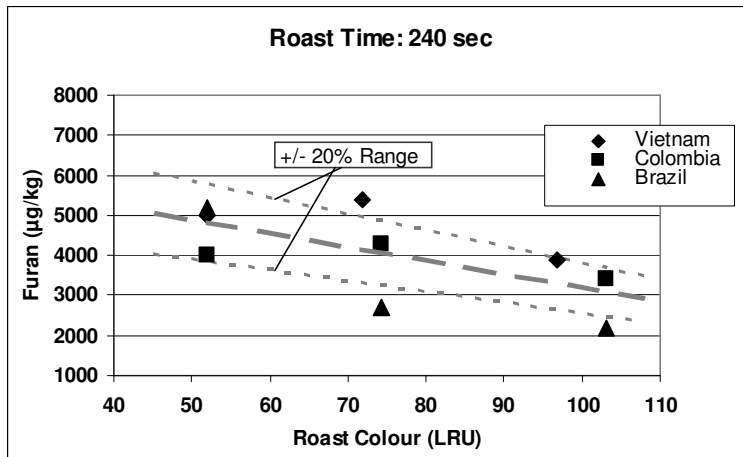


Figure 2 and Figure 3

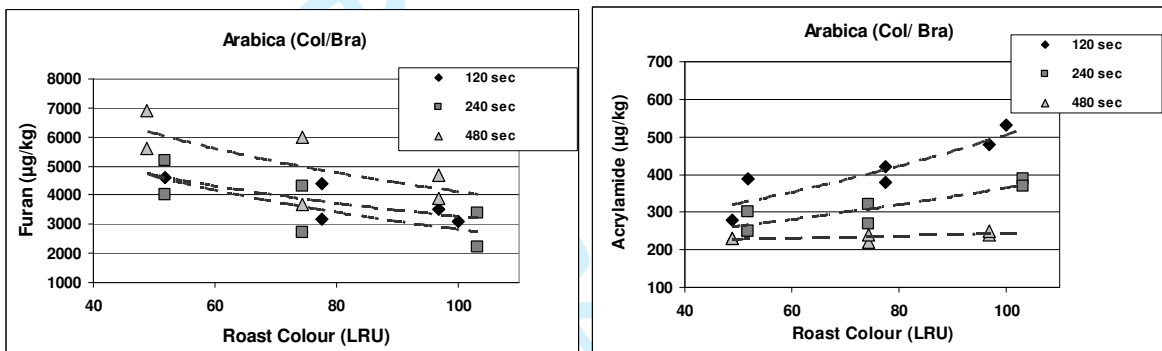


Figure 4

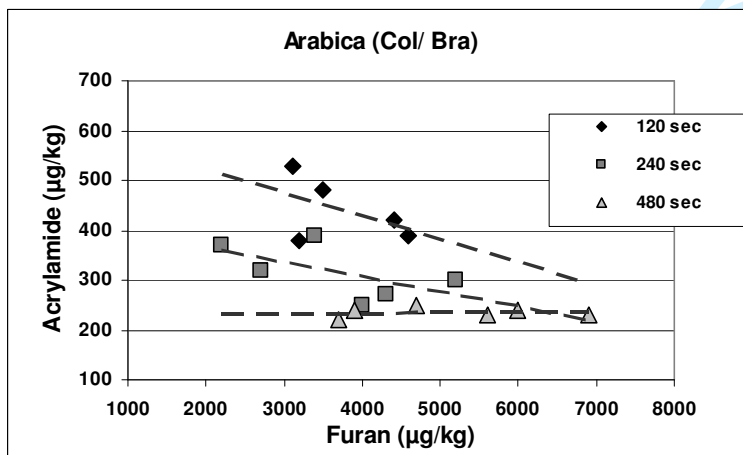


Figure 5

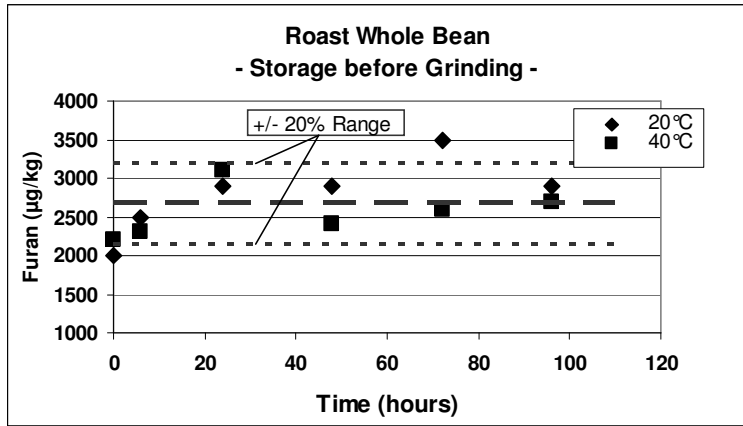


Figure 6

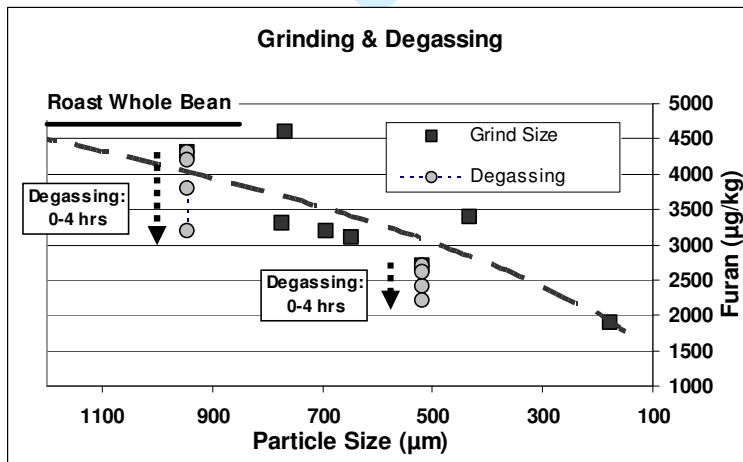


Figure 7

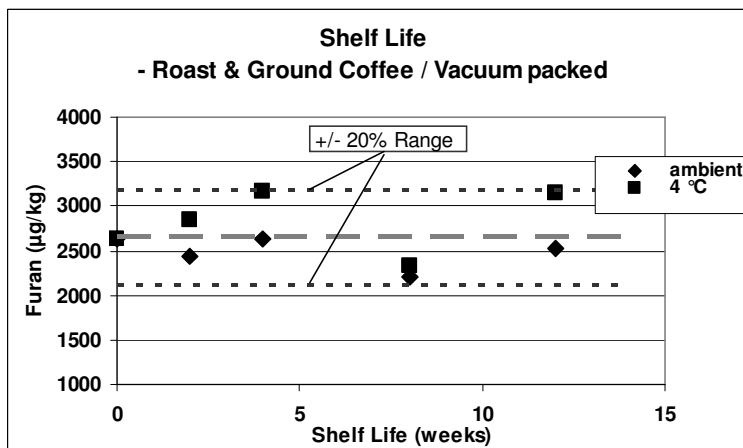


Figure 8

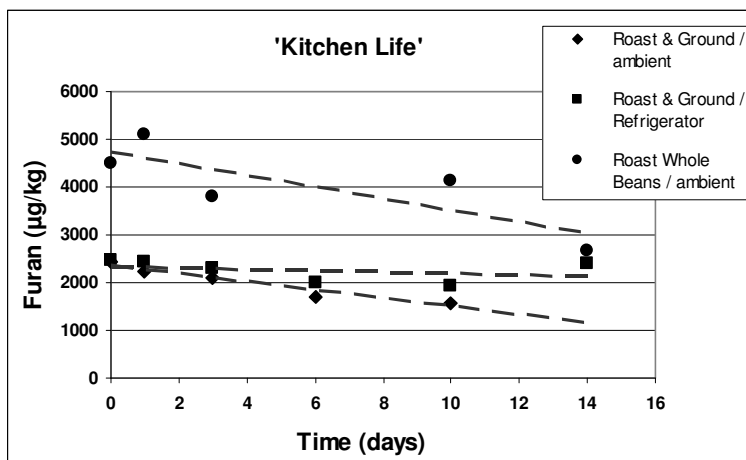


Figure 9

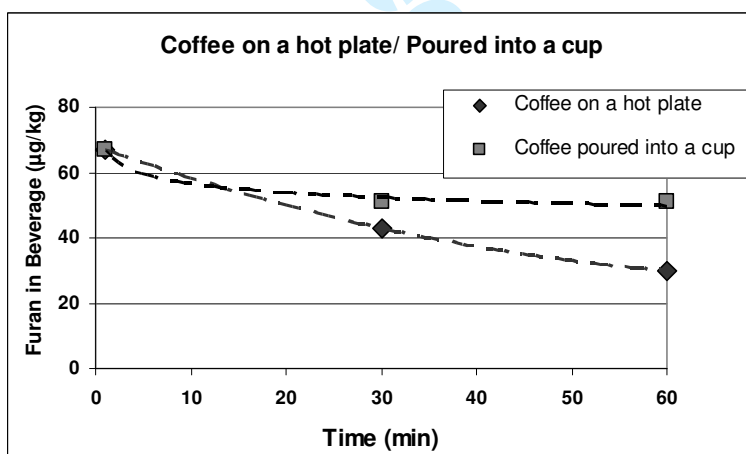


Table 1. Effect of brewing method on furan extraction using different brewing techniques. Data presented show the amounts of furan in the brew expressed as percentage of the initial furan levels in roast and ground respectively instant coffee.

	This study (n=1)	Kuballa et al. 2005	La Pera et al. 2009
Drip Filter	50%	Mean: 26% Range: 11-91%	
Cafetière/ Infusion in hot water	65%		43.0 +/- 6.9%
Pads	50%		
Espresso	33%	Mean: 40% Range: 23-57%	36.7 +/- 7.9
Instant Coffee	81%	Mean: 70% Range: 32-122%	

Table 2. Summary of influencing factors on furan from roasting to coffee as consumed.

Process/ Handling Step	Key influencing factors	Assumed typical reduction per step	Remaining furan level after step
Roasting	- Roast Time - Degree of roast	-	100%
Grinding	- Grind size - Temperature	40%	60%
Degassing	- Particle Size - Duration/ - Temperature	20%	48%
In Pack Shelf Life		0%	48%
'Kitchen Life'	- Type of packaging - Degree of re-closing - Temperature - Duration	25%	36%
Brewing	- Type of Brewer - Temperature	55%	16%
Keeping coffee hot	- Temperature/ - Duration	35%	10%
Coffee in cup for consumption	- Pouring into cup - Cooling	10%	9%