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Effects of Roasting Conditions on Furan Formation and Volatile Aroma Profile of Dibek Coffee

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Abstract

Roasting conditions critically influence both aroma development and the formation of potentially harmful process contaminants in coffee. This study investigated the effects of roasting temperature (127–212 °C) and time (4.19–46.21 min) on furan formation and volatile aroma composition in traditional Turkish Dibek coffee using response surface methodology and GC–MS-based analyses. Furan concentrations increased approximately 2.9-fold, from 18.24 ± 0.61 µg/kg at 127 °C to 52.12 ± 0.22 µg/kg at 212 °C, with roasting temperature exerting a substantially greater influence than roasting time. The quadratic response surface model explained 95.2% of the variation in furan formation (R^2 = 0.952). Among the 47 identified volatile compounds, pyrazines, phenolic compounds, and furan derivatives generally increased with roasting severity, whereas several aldehydes and ketones were more abundant under mild roasting conditions. Pearson correlation analysis revealed positive associations between furan and Maillard-derived aroma-related compounds, while principal component analysis explained 70.08% of the total variance and differentiated roasting treatments according to their volatile profiles. These findings demonstrate that roasting conditions promoting aroma-related compound formation may simultaneously increase furan accumulation, highlighting the importance of considering both aroma composition and process contaminant formation when developing roasting strategies for Dibek coffee.

Keywords: Aroma profile; Coffee roasting; Dibek coffee; Food safety; Furan; Volatile compounds

1. Introduction

Coffee roasting is a critical thermal processing step that determines the chemical composition, aroma characteristics, and overall quality of roasted coffee. During roasting, coffee beans undergo complex physicochemical transformations involving Maillard reactions, Strecker degradation, caramelization, and lipid oxidation. These reactions generate numerous volatile and non-volatile compounds that contribute to the characteristic sensory properties of roasted coffee. However, the same thermal processes responsible for desirable aroma development may also promote the formation of potentially harmful processing contaminants. Consequently, understanding the relationships between roasting conditions, aroma formation, and contaminant generation is essential for developing processing strategies that consider both product quality and food safety **(Buffo and Cardelli-Freire, 2004; Baggenstoss et al., 2008; Poisson et al., 2017).**

The volatile aroma profile of roasted coffee comprises a chemically diverse mixture of compounds, including furans, pyrazines, pyrroles, aldehydes, ketones, phenolic compounds, alcohols, and sulfur-containing compounds. Their formation and relative abundance depend on the composition of the raw coffee beans and the thermal conditions applied during roasting. Pyrazines, primarily generated through Maillard reactions and Strecker degradation, contribute roasted, nutty, and cocoa-like aroma characteristics, whereas several furan derivatives are associated with sweet, caramel-like, and toasted notes. Phenolic compounds contribute smoky and spicy characteristics, while aldehydes and ketones may impart fruity, green, and fatty aroma attributes. The relative distribution of these compounds changes throughout roasting because of simultaneous formation, degradation, volatilization, and secondary reactions **(Buffo and Cardelli-Freire, 2004; Sunarharum et al., 2014; Dulsat-Serra et al., 2016; Zakidou et al., 2021)**. Therefore, roasting temperature and duration are important processing variables governing the chemical development of coffee aroma.

Previous studies have demonstrated that roasting temperature–time combinations substantially influence the formation and distribution of volatile compounds. Baggenstoss et al. (2008) investigated the effects of different roasting conditions on coffee aroma formation, highlighting the importance of thermal processing parameters in determining the resulting aroma profile. Similarly, investigations of coffee volatile composition have demonstrated that increasing roasting intensity generally promotes the formation of Maillard-derived heterocyclic compounds while altering the abundance of lipid-derived volatiles **(Dulsat-Serra et al., 2016; Poisson et al., 2017)**. Nevertheless, the relationship between roasting severity and aroma development is not necessarily linear, because some volatile compounds accumulate during roasting whereas others undergo thermal degradation or volatilization. These competing reactions complicate the selection of roasting conditions based exclusively on volatile compound formation.

Among the processing contaminants associated with coffee roasting, furan is of particular concern because of its potential toxicological effects and occurrence in thermally processed foods. Furan can be generated through several chemical pathways, including carbohydrate degradation, Maillard reactions, ascorbic acid decomposition, and oxidation of polyunsaturated fatty acids. The relative contributions of these pathways depend on the composition of the food matrix and the applied processing conditions **(Becalski and Seaman, 2005; EFSA, 2017)**. The International Agency for Research on Cancer has classified furan as possibly carcinogenic to humans (Group 2B), while the European Food Safety Authority has identified potential health concerns associated with dietary exposure to furan and related compounds **(IARC, 1995;**

EFSA, 2017). Because coffee is widely consumed and furan can form during roasting, understanding the factors controlling its concentration is relevant to food safety assessment.

Several investigations have examined the influence of coffee roasting on furan formation. **Arisseto et al. (2011)** demonstrated that furan concentrations are influenced by coffee species, roasting degree, and brewing procedures. **Guenther et al. (2010)** also investigated furan occurrence and its relationship with coffee processing conditions. More recently, **Zhu et al. (2022)** examined roasting-induced thermal contaminants using a kinetic modeling approach, providing additional information on their formation behavior. **Alsafra et al. (2023)** employed multivariate statistical analysis to characterize the formation of process contaminants during coffee roasting. Collectively, these studies indicate that roasting temperature, duration, and the progression of thermal reactions play important roles in contaminant formation. However, furan concentrations reflect a dynamic balance between chemical formation and volatilization, making their responses to roasting conditions potentially nonlinear.

An important challenge in coffee processing is that desirable aroma-related compounds and undesirable thermal contaminants may originate from overlapping reaction pathways. In particular, carbohydrate degradation and Maillard reactions contribute to the generation of both aroma-related furan derivatives and furan itself. Consequently, roasting conditions that enhance the formation of characteristic roasted aroma compounds may simultaneously promote furan accumulation. Although previous studies have substantially advanced knowledge of coffee aroma chemistry and thermal contaminant formation, a detailed understanding of their relationships within specific coffee products remains important for developing product-oriented roasting strategies **(Baggenstoss et al., 2008; Alsafra et al., 2023; Zhu et al., 2022)**. Furthermore, correlations between volatile compounds and furan concentrations can provide useful information about their responses to roasting severity, although such associations do not independently establish common formation mechanisms.

Dibek coffee is a traditional Turkish coffee product associated with a distinctive mortar-and-pestle grinding process. Unlike conventional finely ground Turkish coffee, Dibek coffee is traditionally characterized by its specific grinding practice and resulting particle characteristics. These processing features may influence the physical behavior and release of volatile compounds during subsequent preparation and consumption. Nevertheless, the chemical changes induced by roasting must also be considered when evaluating its aroma-related characteristics and potential processing contaminants. Although the effects of roasting on furan formation and volatile composition have been investigated in various coffee products, the simultaneous evaluation of these responses in traditional Dibek coffee has received

comparatively limited attention in the literature considered here. This creates a need for systematic investigations focusing on the relationships between roasting variables, furan accumulation, and volatile aroma composition in this particular coffee product.

Response Surface Methodology (RSM) provides a statistical framework for evaluating the effects of processing variables and their interactions on selected responses. When combined with a Central Composite Design (CCD), RSM enables the estimation of linear, interaction, and quadratic effects within a defined experimental domain. In coffee roasting studies, such an approach can facilitate the quantitative assessment of temperature- and time-dependent changes in furan formation. In parallel, headspace gas chromatography–mass spectrometry (HS-GC–MS) enables the determination of volatile contaminants such as furan, while headspace solid-phase microextraction coupled with GC–MS (HS-SPME/GC–MS) provides information on the broader volatile composition. The integration of these analytical techniques with Pearson correlation analysis, principal component analysis (PCA), and hierarchical cluster analysis (HCA) offers a complementary approach for examining relationships between roasting conditions and chemical responses.

The contribution of the present study lies in the integrated characterization of furan accumulation and volatile composition in coffee prepared using a Dibek-style grinding procedure under systematically controlled roasting conditions. Quantitative furan determination, semi-quantitative volatile profiling, response surface modeling, and multivariate statistical analyses were applied within a common experimental framework to examine associations between roasting conditions and chemical composition. However, because Dibek-style grinding was performed after roasting and no conventional-grinding control was included, the experimental design does not establish a Dibek-specific effect on roasting chemistry or demonstrate reaction mechanisms distinct from those of conventionally processed coffee.

Accordingly, the central research question was whether roasting temperature and time exert similar or contrasting effects on furan formation and the development of characteristic volatile aroma compounds in Dibek coffee. It was hypothesized that increasing roasting severity would generally promote both furan accumulation and the formation of Maillard-derived aroma-related compounds, although individual volatile compounds could exhibit different responses because of competing formation and degradation pathways. Therefore, the objectives of this study were to (*i*) evaluate the effects of roasting temperature and time on furan formation using RSM based on a CCD, (*ii*) characterize roasting-dependent changes in volatile composition using HS-SPME/GC–MS, and (*iii*) investigate the relationships between roasting conditions, furan concentrations, and volatile compounds through multivariate statistical

analyses. The findings are expected to provide a scientific basis for evaluating roasting strategies that consider both aroma-related chemical characteristics and the formation of undesirable processing contaminants in Dibek coffee.

2. Materials and Methods

2.1. Materials

Dibek coffee samples produced from 100% Brazilian *Coffea arabica* L. beans were obtained from a local commercial supplier and stored in airtight containers at 4 ± 1 °C until analysis. A furan analytical standard (≥99% purity) and furan-d_4 were purchased from Sigma-Aldrich (St. Louis, MO, USA) and used for furan quantification. 2-Octanol was employed as the internal standard for volatile compound analysis. All solvents and reagents used for HS-GC–MS and HS-SPME/GC–MS analyses were of analytical grade and obtained from Sigma-Aldrich (St. Louis, MO, USA) and Merck (Darmstadt, Germany). Unless otherwise stated, all analyses were performed in triplicate, and the results are presented as mean ± standard deviation.

2.2. Response Surface Methodology (RSM)

Response Surface Methodology (RSM) based on a Central Composite Design (CCD) was employed to evaluate the combined effects of roasting temperature (X_1, 127–212 °C) and roasting time (X_2, 4.19–46.21 min) on furan formation in Dibek coffee. The experimental design consisted of 19 runs, including factorial, axial, and replicated center points. The volatile profiles obtained under the same roasting conditions were evaluated separately using descriptive and multivariate analyses.

The experimental data were fitted to the second-order polynomial model shown in **Eq. (1)**:

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_{12} X_1 X_2 + \beta_{11} X_1^2 + \beta_{22} X_2^2 \quad (1)$$

where Y is the response variable, β_0 is the intercept, β_1 and β_2 are the linear coefficients, β_{12} is the interaction coefficient, and β_{11} and β_{22} are the quadratic coefficients. Model adequacy was evaluated by analysis of variance (ANOVA) using the coefficient of determination (R^2), adjusted R^2 (Adj-R^2), predicted R^2 (Pred-R^2), lack-of-fit, and coefficient of variation (CV%). Statistical analyses and response surface modeling were performed using Design-Expert® software (Stat-Ease Inc., Minneapolis, MN, USA).

2.3. Roasting, Grinding and Sample Preparation

Coffee samples were roasted using a laboratory-scale coffee roaster equipped with direct thermal heat transfer, an electric heating unit, dual digital temperature sensors, a stainless-steel roasting drum, and a cooling tray. Roasting was carried out under the temperature–time combinations defined by the experimental design. Immediately after roasting, the samples were rapidly cooled to room temperature to terminate thermal reactions and subsequently stored in airtight containers until analysis. All roasting experiments were performed in triplicate.

Following roasting, the coffee beans were processed using a mechanically controlled mortar-and-pestle system designed to simulate the traditional Turkish Dibek method. The resulting coarse particles were homogenized and passed through a 250 μm sieve to obtain a uniform particle size prior to analysis. The prepared samples were transferred to airtight glass vials and analyzed within 24 h to minimize moisture uptake and the loss of volatile compounds. Before HS-SPME/GC–MS analysis, all samples were thoroughly homogenized to ensure representative sampling.

2.4. Furan Analysis

Furan concentrations in Dibek coffee were measured by static headspace gas chromatography coupled with mass spectrometry (HS-GC–MS), following an analytical procedure adapted from FDA guidance and the methodologies described by **Zoller et al. (2007)** and **Drakula et al. (2022)**. For sample preparation, accurately weighed portions of homogenized coffee (1.00 ± 0.01 g) were introduced into individual 20 mL headspace vials. Each portion was supplemented with 100 μL of isotopically labeled furan-d_4, serving as the internal standard, before the vials were closed using PTFE/silicone-lined septa. Headspace equilibration was achieved by maintaining the sealed samples at 85 °C for 20 min under continuous agitation (500 rpm). The sampling sequence included a 1 min vial pressurization step, withdrawal of a 1.0 mL headspace aliquot, and transfer through a line maintained at 120 °C.

The analytical system consisted of an Agilent 7890A gas chromatograph interfaced with an Agilent 5975C mass-selective detector (Agilent Technologies, Santa Clara, CA, USA). Separation of the target analyte was achieved on an HP-5MS fused-silica capillary column with dimensions of 30 m × 0.25 mm and a stationary-phase film thickness of 0.25 μm. High-purity helium (99.999%) supplied the mobile phase at a fixed flow of 1.0 mL min^{-1}. The

chromatographic temperature program began with a 2 min isothermal period at 40 °C, followed by an increase of 10 °C min^{-1} until 200 °C was reached. This final temperature was maintained for an additional 5 min. Sample introduction was performed without flow splitting, with the injection port maintained at 200 °C.

For mass spectral acquisition, the detector operated with electron ionization at 70 eV. The ion source was maintained at 230 °C, whereas the quadrupole temperature was set to 150 °C. Spectral information was collected across a mass-to-charge interval of m/z 35–450. Assignment of the furan signal relied on agreement between the chromatographic retention behavior and diagnostic fragment ions of the sample and those obtained from the corresponding reference standard.

Furan levels were calculated from a multipoint calibration covering 1–500 μg L^{-1}, using furan-d_4 to account for analytical variability. The calibration relationship yielded a coefficient of determination of at least 0.995 ($R^2 \geq 0.995$). Final concentrations were normalized to sample mass and expressed as μg kg^{-1} of coffee. Three replicate determinations were conducted for each sample, and the resulting concentrations were summarized using their arithmetic means and associated standard deviations.

2.5. Analysis of Volatile Aroma Compounds (HS-SPME/GC–MS)

The volatile composition of Dibek coffee was characterized through headspace solid-phase microextraction combined with gas chromatography–mass spectrometry (HS-SPME/GC–MS). Sample preparation involved introducing 2.0 g of finely ground coffee into a 20 mL glass headspace vial, followed by the addition of 10 μL of 2-octanol solution (100 mg L^{-1} in methanol) as an internal reference. To minimize the escape of volatile constituents before extraction, each vial was promptly closed with a PTFE/silicone-lined septum.

Isolation of aroma-related constituents from the sample headspace was accomplished using a triphasic divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) fiber with a coating thickness of 50/30 μm (Supelco, Bellefonte, PA, USA). Before fiber exposure, the coffee samples were maintained at 60 °C for 20 min to establish headspace equilibrium. The fiber was subsequently exposed to the vapor phase above the sample for 30 min to collect volatile analytes. Following extraction, the retained compounds were released directly into the chromatographic system by heating the fiber in the injection port at 250 °C for 5 min under splitless conditions.

Chromatographic separation and mass spectral characterization were carried out with an Agilent 7890B GC system interfaced with an Agilent 5977 mass-selective detector (Agilent

Technologies, Santa Clara, CA, USA). An HP-5MS capillary column (30 m length, 0.25 mm internal diameter, and 0.25 μm stationary-phase thickness) provided separation of the extracted constituents. The mobile phase consisted of helium with a purity of 99.999%, delivered at a uniform rate of 1.0 mL min^{-1}. Initially, the GC oven was maintained at 40 °C for 3 min. The temperature was then raised progressively to 250 °C at 5 °C min^{-1}, with a final isothermal period of 5 min.

Mass spectral measurements were obtained under electron ionization conditions at an energy of 70 eV. During acquisition, the ionization source and quadrupole were held at 230 and 150 °C, respectively, while the monitored mass interval extended from m/z 35 to 450. Compound assignments were established by evaluating the correspondence between experimentally acquired spectra and reference entries in the NIST Mass Spectral Library, together with comparisons of calculated linear retention indices (LRIs) against previously reported values.

Relative quantification of the detected constituents was based on their chromatographic responses with respect to the added 2-octanol internal standard. Because authentic calibration standards were not employed for every individual volatile compound, the resulting values were treated as semi-quantitative estimates and expressed as micrograms of internal standard equivalents per kilogram of coffee (μg IS eq. kg^{-1}). Measurements were conducted in triplicate unless specified otherwise. Supplementary **Tables S1** and **S2** provide the complete compound inventory, including retention index information, identification evidence, associated aroma characteristics, and estimated concentrations.

2.6. Method Validation

The analytical performance of the HS-GC–MS method for furan determination was evaluated in terms of linearity, sensitivity, precision, and accuracy. Method linearity was assessed using a multi-point calibration curve (1–500 μg L^{-1}), which showed excellent linearity ($R^2 > 0.995$). The limits of detection (LOD) and quantification (LOQ), calculated at signal-to-noise ratios of 3 and 10, were 0.60 and 1.10 μg kg^{-1}, respectively. Method precision, evaluated as repeatability (RSDr) and reproducibility (RSDR), was below 4.54% and 6.05%, respectively. Accuracy, assessed by recovery experiments at different concentration levels, ranged from 85 to 110%, confirming the suitability of the method for furan determination in roasted Dibek coffee.

2.7. Statistical Analysis

Statistical analyses were performed using XLSTAT 2024 (Addinsoft, New York, NY, USA). Data normality and homogeneity of variance were evaluated using the Shapiro–Wilk and Levene tests, respectively. Differences among experimental conditions were assessed by one-way analysis of variance (ANOVA), and statistical significance was accepted at $p < 0.05$. Pearson correlation analysis, principal component analysis (PCA), and hierarchical cluster analysis (HCA) were performed to evaluate relationships among volatile compounds and roasting conditions. Response surface methodology (RSM) analyses were conducted using Design-Expert® software (Stat-Ease Inc., Minneapolis, MN, USA).

3. Results and Discussion

3.1. Effects of Roasting Conditions on Furan Formation

Response Surface Methodology (RSM) was employed to evaluate the combined effects of roasting temperature and time on furan formation in Dibek coffee. The quadratic model was statistically significant ($p < 0.0001$), with R^2, adjusted R^2, and predicted R^2 values of 0.952, 0.933, and 0.821, respectively (**Table 1**). Although the model explained a substantial proportion of the observed variation, the significant lack-of-fit ($p = 0.041$) indicated that systematic variation remained unexplained. Therefore, the quadratic model should be regarded as an empirical description of the relationships between roasting conditions and furan concentrations within the investigated experimental domain rather than as a fully validated predictive or mechanistic model.

The relationship between roasting conditions and furan formation was described by the second-order polynomial model presented in **Eq. (2)**:

$$\text{Furan (μg/kg)} = 48.11 + 11.80X_1 + 3.21X_2 + 0.29X_1X_2 - 8.13X_1^2 - 1.31X_2^2 \quad (2)$$

Here, X_1 and X_2 represent the coded values of roasting temperature and roasting time, respectively.

The regression analysis identified roasting temperature as the most influential factor affecting furan formation ($p < 0.0001$), whereas roasting time showed a weaker but statistically significant effect ($p = 0.0011$). The significant quadratic effect of temperature ($p < 0.0001$) indicated a nonlinear response, while the temperature–time interaction was not statistically significant ($p = 0.8160$). This nonlinear behavior may reflect the combined effects of precursor degradation, furan formation, and volatilization during roasting. However, these processes cannot be individually quantified using the present empirical model.

Table 1. Analysis of variance (ANOVA) for the second-order polynomial model describing furan formation.

Source	df	SS	MS	F-value	p-value
Model	5	1842.56	368.51	51.49	< 0.0001
Temperature (X_1)	1	1354.82	1354.82	189.14	< 0.0001
Time (X_2)	1	123.78	123.78	17.28	0.0011
X_1X_2	1	0.44	0.44	0.06	0.8160
X_1^2	1	391.16	391.16	54.62	< 0.0001
X_2^2	1	10.38	10.38	1.45	0.2500
Residual	13	93.06	7.16	—	—
Lack-of-fit	9	82.31	9.15	3.40	0.0410
Pure error	4	10.75	2.69	—	—
Total	18	1935.62	—	—	—

The response surface and contour plots (**Figs. 1** and **2**) indicated that furan concentrations were more strongly influenced by roasting temperature than by roasting time. The lowest measured concentration (18.24 ± 0.61 μg kg^{-1}) was obtained at 127 °C for 25 min, whereas the highest concentration (52.12 ± 0.22 μg kg^{-1}) was observed at 212 °C for 25 min, representing an approximately 2.9-fold increase. Furan concentrations increased markedly at temperatures above approximately 170 °C, consistent with the increasing contribution of thermally induced reactions. Similar temperature-dependent trends have been reported in previous investigations of coffee roasting **(Guenther et al., 2010; Alsafra et al., 2023; da Costa et al., 2023)**.

Importantly, the roasting condition associated with the highest furan concentration should not be interpreted as an optimal processing condition from a food safety perspective. Although the experimental results demonstrate that roasting severity affects both furan accumulation and aroma-related volatile composition, the present study did not include sensory evaluation or validated multi-response optimization. Consequently, an optimal balance between sensory quality and contaminant minimization cannot be established from these results.

Furan can be generated through several thermal pathways, including carbohydrate degradation, Maillard reactions, ascorbic acid decomposition, and lipid oxidation. Reactive carbonyl intermediates formed during sugar degradation are considered important precursors of furan formation **(Becalski and Seaman, 2005; Yaylayan, 2006)**. Nevertheless, the observed response to roasting severity was not strictly linear. For example, prolonged roasting at 170 °C produced furan concentrations comparable to those obtained after shorter roasting at 200 °C. This observation may reflect competing formation and volatilization processes, although their individual contributions were not directly measured. Similar considerations regarding furan volatility during thermal processing have been discussed by **Zoller et al. (2007)**.

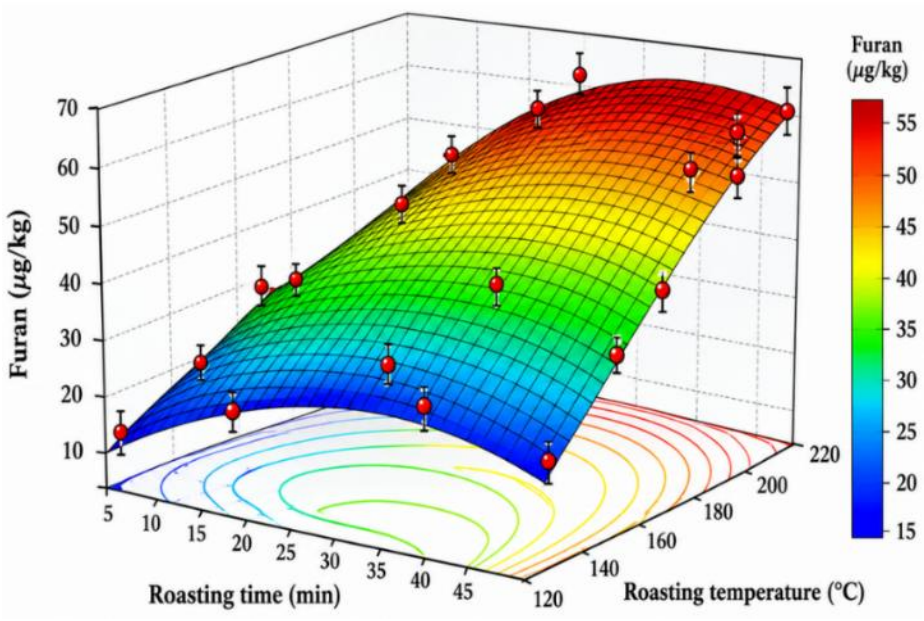


Figure 1. Response surface plot showing the combined effects of roasting temperature and roasting time on furan formation in Dibek coffee.

The overall increase in furan concentration with roasting severity is consistent with previous reports describing the effects of roasting conditions on furan and related compounds in coffee **(Arisseto et al., 2011; Zhu et al., 2022; Engelhardt et al., 2023)**. However, the behavior of different process contaminants may vary with roasting intensity. For example, **Kung et al. (2024)** reported contrasting responses of acrylamide and furanic compounds to increasing roasting severity. These observations highlight the importance of evaluating individual processing contaminants rather than assuming that all thermally generated compounds follow similar trends.

Among the identified furan derivatives, 3-furanmethanol generally increased with roasting temperature, whereas 2-furanmethanol was mainly detected under low and intermediate roasting conditions and was not detected under the most intensive conditions. These differences may be associated with compound-specific formation, transformation, or volatilization processes. Previous studies have also described changes in furfuryl alcohol derivatives during coffee roasting **(Baggenstoss et al., 2008)**.

The concentration of 5-(hydroxymethyl)-2-furancarboxaldehyde (HMF) also varied with roasting conditions. HMF can be generated through hexose dehydration, caramelization, and Maillard reactions and may undergo further degradation during prolonged thermal treatment **(Farag et al., 2020; Guo et al., 2021; Martins et al., 2022; Shen et al., 2011)**. The relatively

limited variation observed in the present study may therefore reflect competing formation and degradation processes.

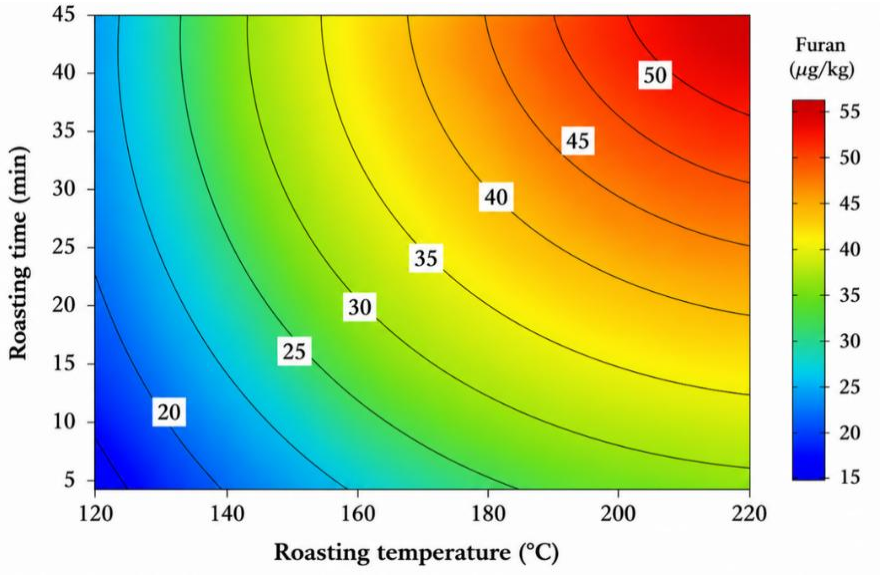


Figure 2. Contour plot illustrating the distribution of furan concentration as affected by roasting temperature and roasting time.

Several furanone compounds also increased with roasting severity. In particular, 2(3H)-furanone derivatives and Furaneol-related compounds have been associated with sweet, caramel-like, and roasted aroma characteristics **(Blank et al., 1996; Cerny, 2008; Schwab, 2013; Haag et al., 2021)**. Benzofuran derivatives also increased at higher roasting temperatures, potentially reflecting thermal transformations of phenolic precursors **(Moon et al., 2010; Bork et al., 2024).** These observations suggest that individual furan-related compounds respond differently to roasting conditions and should not be interpreted as chemically or toxicologically equivalent to furan itself.

Overall, the findings indicate that roasting temperature is the principal experimental factor associated with furan accumulation in Dibek coffee, while roasting duration has a smaller but significant influence. The RSM analysis provides a useful empirical description of these effects within the studied conditions, subject to the limitation indicated by the significant lack-of-fit. The simultaneous changes observed in furan concentrations and aroma-related volatile compounds emphasize the need for future studies combining contaminant measurements, sensory evaluation, and multi-response optimization to identify roasting conditions that appropriately consider both chemical composition and food safety.

3.2. Evolution of Volatile Aroma Compounds During Coffee Roasting

GC–MS analysis identified 47 volatile compounds belonging to furans and their derivatives, pyrazines, phenolic compounds, aldehydes, ketones, alcohols, and fatty acid derivatives. Detailed information on compound identification, retention times, experimental and literature retention indices (RI), aroma descriptors, and semi-quantitative concentrations is provided in Supplementary **Tables S1** and **S2**. Roasting conditions markedly influenced both the composition and abundance of these compounds, with increasing roasting severity promoting thermally induced reactions such as the Maillard reaction, Strecker degradation, caramelization, and lipid oxidation. Consequently, medium- and high-temperature roasting generated a more complex volatile profile, consistent with previous findings for Turkish coffee **(Dadalı, 2022) (Fig. 3)**.

Pyrazines were among the most characteristic aroma compounds and increased markedly between 150 and 212 °C. Methyl pyrazine, 2,5-dimethyl pyrazine, ethyl pyrazine, and 2-ethyl-5-methyl pyrazine all showed temperature-dependent increases, consistent with the progression of Maillard reactions during roasting. For example, the concentration of 2-ethyl-5-methyl pyrazine increased from 0.02 ± 0.01 µg IS eq./kg at 150 °C to 0.29 ± 0.01 µg IS eq./kg at 212 °C. These compounds are well known contributors to roasted, nutty, and cocoa-like aroma notes in coffee **(Buffo and Cardelli-Freire, 2004)**.

Furan and several furan derivatives exhibited marked changes across roasting conditions. In agreement with the RSM results, furan concentration increased from 18.24 ± 0.61 to 52.12 ± 0.22 µg kg^{-1} as roasting severity increased, while 3-furanmethanol, benzofuran, and furanone derivatives showed similar temperature-dependent trends. Although certain furan derivatives are associated with caramel-like and roasted aroma characteristics, furan itself was evaluated primarily as an undesirable process contaminant in the present study. The increasing abundance of 3-furanmethanol is consistent with roasting-induced chemical transformations reported in the literature, but does not independently establish the underlying reaction pathways **(Yaylayan, 2006).**

Among the phenolic compounds, hydroquinone, 1,2-benzenediol, and 2-methoxy-4-vinylphenol exhibited temperature-dependent increases. In particular, 2-methoxy-4-vinylphenol reached 1.46 ± 0.01 µg IS eq./kg at 212 °C and is recognized as an important contributor to the smoky, spicy, and woody aroma of roasted coffee **(Wang et al., 2018)**. Its formation is mainly associated with the thermal degradation of chlorogenic acids and feruloylquinic acid derivatives, whereas the increases in hydroquinone and 1,2-benzenediol are attributed to the decomposition of other phenolic precursors during roasting **(Farah et al., 2006; Dorfner et al., 2003; Moon et al., 2010)**.

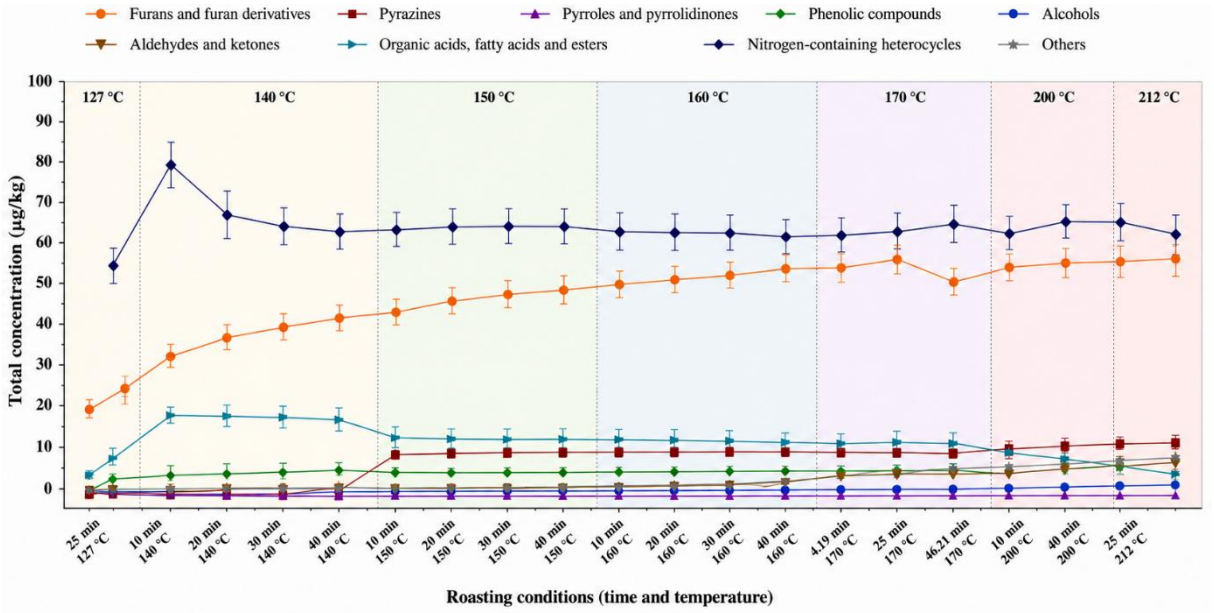


Figure 3. Changes in the Total Concentrations of Chemical Classes During Dibek Coffee Roasting Under Different Temperature–Time Conditions (μg kg^{-1} ± SD)

The evolution of volatile compound classes during roasting is summarized in **Fig. 3.** Furans and their derivatives constituted the dominant chemical class and exhibited an approximately threefold increase with roasting severity. Pyrazines and phenolic compounds also increased progressively, consistent with Maillard-associated transformations and thermal degradation of phenolic precursors. In contrast, aldehydes and ketones were more abundant at low and medium roasting temperatures and declined under severe roasting conditions because of further thermal conversion or degradation **(Moon et al., 2009; Poisson et al., 2017)**.

Aldehydes and ketones, including heptanal, 2-octanone, and 1-octen-3-one, were predominantly detected at low and intermediate roasting temperatures. These compounds originate mainly from lipid oxidation and contribute fruity, green, mushroom-like, and fatty aroma notes **(Dippong et al., 2022)**. Their subsequent decline at higher roasting temperatures suggests participation in secondary thermal reactions or degradation processes **(Moon et al., 2009; Poisson et al., 2017)**. Alcohols and fatty acid derivatives also changed during roasting. Phenylethyl alcohol increased gradually with roasting severity, whereas fatty acids such as palmitic acid reached their highest concentrations during intermediate roasting stages before declining at higher temperatures, consistent with their role as precursors of lipid-derived volatile compounds.

A comparison of representative volatile compounds further illustrates the magnitude and direction of roasting-induced changes in Dibek coffee. For example, the concentration of 2-

ethyl-5-methylpyrazine increased from 0.02 ± 0.01 µg IS eq./kg at 150 °C to 0.29 ± 0.01 µg IS eq./kg at 212 °C, corresponding to an approximately 14.5-fold increase. Similarly, 2-methoxy-4-vinylphenol reached 1.46 ± 0.01 µg IS eq./kg at 212 °C, indicating the greater abundance of selected phenolic compounds under intensive roasting conditions. Among furan-related compounds, 3-furanmethanol generally increased with roasting temperature, whereas 2-furanmethanol was mainly detected under mild and intermediate roasting conditions and was not detected under the most severe conditions. In contrast, several lipid-derived aldehydes and ketones, particularly heptanal, 2-octanone, and 1-octen-3-one, were more abundant under mild or intermediate roasting conditions and declined with increasing roasting severity. These contrasting responses demonstrate that roasting does not uniformly increase all volatile compounds but instead produces compound-specific changes in the chemical profile. The complete semi-quantitative results are provided in Supplementary **Tables S1** and **S2**.

The observed differences in volatile compound profiles suggest that roasting temperature and duration influence the relative contributions of several competing chemical pathways. The increasing abundance of pyrazines under more intensive roasting conditions is consistent with the progression of Maillard reactions and Strecker degradation **(Buffo and Cardelli-Freire, 2004; Baggenstoss et al., 2008)**, whereas changes in furan derivatives may reflect carbohydrate degradation, secondary thermal reactions, and compound-specific volatilization **(Alsafra et al., 2023)**. Similarly, the increasing concentrations of selected phenolic compounds may be associated with the thermal decomposition of chlorogenic acids and related phenolic precursors **(Moon et al., 2010)**. In contrast, the lower abundance of several aldehydes and ketones at higher roasting temperatures may result from their further transformation, degradation, or volatilization **(Poisson et al., 2017; Kumazawa and Masuda, 2003)**. Importantly, the simultaneous increase in furan and certain aroma-related volatile compounds indicates that more intensive roasting conditions can influence both aroma-associated chemical constituents and undesirable processing contaminants. However, these associations should not be interpreted as direct evidence of common formation mechanisms, and the measured volatile concentrations alone cannot establish the sensory quality of the resulting coffee. Overall, these findings highlight the complexity of roasting-induced chemical transformations and the importance of considering individual volatile compound classes when evaluating roasting conditions in Dibek coffee.

3.3. Multivariate Analysis of Furan Formation and Volatile Aroma Profile

To obtain a comprehensive understanding of the relationships between roasting conditions, furan formation, and volatile aroma compounds, multivariate statistical analyses were performed. Pearson correlation analysis was used to evaluate pairwise associations among roasting variables and volatile compounds, whereas principal component analysis (PCA) and hierarchical cluster analysis (HCA) were applied to characterize the distribution of roasting treatments and identify groups of chemically related compounds. These complementary approaches have been widely employed to investigate the evolution of coffee aroma and to discriminate roasting conditions based on volatile composition **(Ruosi et al., 2012; Zakidou et al., 2021; Lapčíková et al., 2023)**. The integration of correlation analysis with multivariate pattern recognition provides complementary information on associations between roasting conditions, furan concentrations, and volatile compound profiles. However, these statistical relationships do not directly establish the underlying chemical reaction pathways, because the relevant precursors, reaction intermediates, and reaction kinetics were not experimentally investigated. The multivariate analyses were performed using the volatile compounds summarized in Supplementary **Tables S1** and **S2**.

3.3.1. Pearson Correlation Analysis

Pearson correlation analysis (**Fig. 4**) was performed to evaluate the relationships between roasting conditions, furan formation, and volatile aroma compounds in Dibek coffee. The correlation matrix revealed distinct groups of positively and negatively correlated compounds, reflecting similarities in their responses to roasting conditions.

A strong positive correlation ($r \approx 0.70$–0.95) was observed among furan and several furan-derived compounds, including 2-furanmethanol, furfural, 5-hydroxymethylfurfural (5-HMF), 2-furanone, and furaneol acetate. These compounds are recognized as typical products of carbohydrate degradation, caramelization, and Maillard reactions **(Van Lancker et al., 2011; Liu et al., 2022; Zakidou et al., 2021)**. Similar relationships have been reported for roasted coffee, where furfuryl alcohol, methylfurfural derivatives, and related furanic compounds constitute one of the dominant volatile groups **(Amanpour and Selli, 2015; Poyraz et al., 2016; Lapčíková et al., 2023)**.

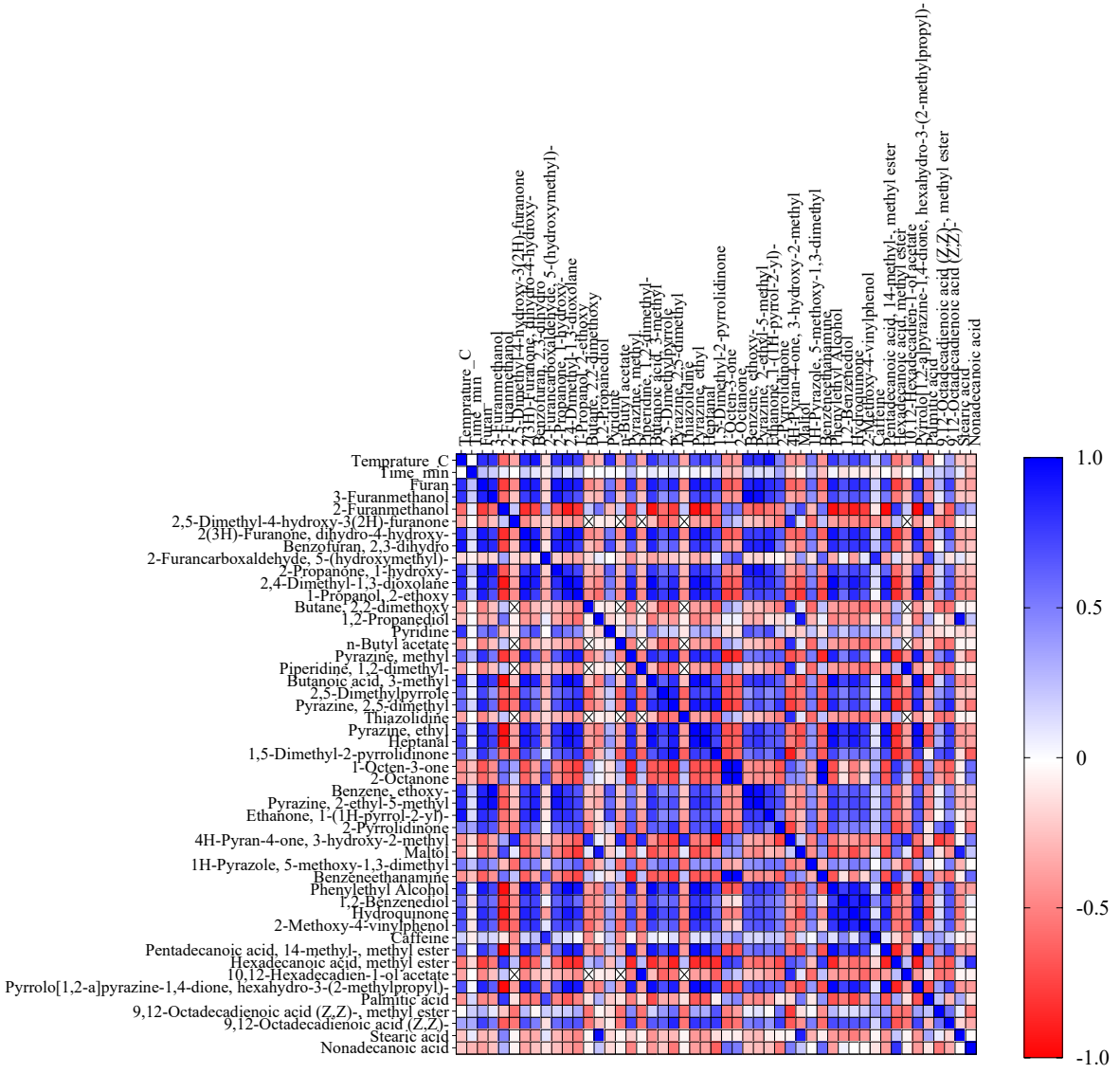


Figure 4. Pearson correlation heatmap showing the relationships between volatile aroma compounds and process parameters in dibek coffee roasted under different temperature–time combinations. Blue and red colors indicate positive and negative correlations, respectively. The symbol "×" denotes statistically non-significant correlations ($p \geq 0.05$).

Pyrazine derivatives, including methylpyrazine, dimethylpyrazines, ethylpyrazine, and 2-ethyl-5-methylpyrazine, formed another strongly correlated cluster ($r \approx 0.80$–1.00). These compounds originate mainly from Strecker degradation and advanced Maillard reactions involving amino acids and reducing sugars **(Yaylayan, 2003; Buffo and Cardelli-Freire, 2004)**. Their close association with furan supports previous observations that increasing roasting severity simultaneously promotes the formation of both pyrazines and furanic

compounds **(Parr et al., 2020; Yu et al., 2021; Spreng et al., 2021; Choe et al., 2023; Wang et al., 2025)**.

Nitrogen-containing heterocyclic compounds, including pyridine, pyrrole derivatives, diketopiperazines, and pyrrolidinones, also exhibited positive correlations with the pyrazine–furan cluster. Their distribution reflects the contribution of trigonelline degradation and cyclization reactions to coffee aroma formation and further illustrates the close interaction among the major thermal reaction pathways during roasting **(Liu et al., 2018; Spreng et al., 2021; Rusinek et al., 2023)**.

Phenolic compounds, particularly hydroquinone, 1,2-benzenediol, and 2-methoxy-4-vinylphenol, showed positive correlations with roasting intensity, consistent with their formation through thermal degradation of chlorogenic acids and related hydroxycinnamic acid derivatives **(Dorfner et al., 2003; Moon et al., 2010; Wang et al., 2018)**.

In contrast, several lipid-derived compounds, including heptanal, 1-octen-3-one, 2-octanone, selected fatty acid derivatives, showed negative correlations with the furan–pyrazine cluster. Their decline under severe roasting conditions is consistent with further oxidation, thermal degradation, or conversion into secondary aroma compounds **(Whitfield and Mottram, 1992; Baggenstoss et al., 2008; Grebenteuch et al., 2021; Alsafra et al., 2023; Zhang et al., 2025)**.

Overall, Pearson correlation analysis revealed associations between furan concentrations and selected aroma-related volatile compounds across the investigated roasting conditions. These relationships are consistent with the influence of roasting severity on several chemical responses, but do not establish shared reaction mechanisms or demonstrate an optimal balance between sensory quality and food safety.

3.3.2. Principal Component Analysis

Principal component analysis (PCA) was performed to evaluate the influence of roasting conditions on the distribution of furan-related volatile compounds in Dibek coffee. As shown in **Fig. 5**, the first two principal components explained 70.08% of the total variance, with PC1 and PC2 accounting for 55.25% and 14.83%, respectively. The high cumulative variance indicates that the PCA model adequately summarized the variability among the roasting treatments. The separation of samples primarily along the PC1 axis demonstrates that roasting severity was the major factor associated with differences in the measured profiles of furan-related volatile compounds. Similar observations have been reported for roasted coffee, where the first principal component is closely associated with roasting degree and the accumulation

of Maillard-derived volatiles **(Ruosi et al., 2012; Zakidou et al., 2021; Lapčíková et al., 2023)**.

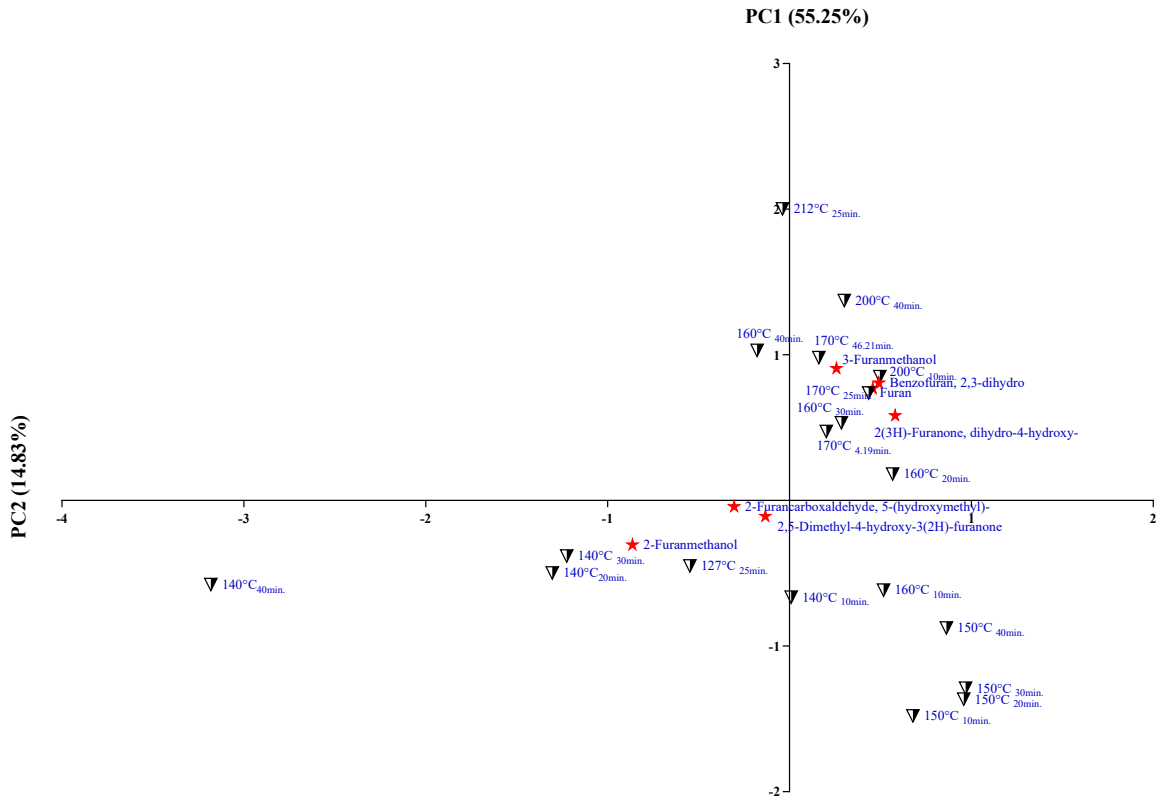


Figure 5. PCA biplot illustrating the relationships between roasting conditions and furan-related compounds in dibek coffee.

As illustrated in **Fig. 5**, furan, 3-furanmethanol, benzofuran (2,3-dihydro), and 2(3H)-furanone, dihydro-4-hydroxy- were located on the positive side of PC1, indicating their close association with high-intensity roasting conditions. These compounds are recognized as products of carbohydrate degradation, caramelization, and Maillard reactions occurring during coffee roasting **(Bressanello et al., 2016)**.

The distribution of the roasting treatments further supports this interpretation. Samples roasted at 170 °C for 46.21 min, 200 °C for 10 min, 200 °C for 40 min, and 212 °C for 25 min were positioned on the positive side of PC1 and showed close associations with furan, 3-furanmethanol, and benzofuran derivatives. This distribution is consistent with the greater abundance of selected furan-related compounds under more intensive roasting conditions; it does not independently establish the responsible reaction pathways. Similar findings have been reported by **Alsafra et al. (2023)**, who highlighted the critical role of reactive carbonyl

intermediates in furan and alkylfuran formation during coffee roasting. Likewise, **Limacher et al. (2008)** demonstrated that furan may originate both from intact sugar molecules and from recombination reactions involving low-molecular-weight carbonyl fragments generated during thermal processing.

The increase in furan concentration observed in the present study, from 18.24 ± 0.61 μg kg^{-1} to 52.12 ± 0.22 μg kg^{-1} across the roasting treatments, further supports the PCA findings. This trend is consistent with previous reports demonstrating a positive relationship between roasting severity and furan formation in coffee **(Guenther et al., 2010; Bragotto et al., 2011; Lachenmeier et al., 2018)**. Similar relationships between roasting intensity and furan-derived compounds have also been reported by **Ruosi et al. (2012)**, **Zakidou et al. (2021)**, and **Zhu et al. (2022)**.

In contrast, samples roasted at 140 °C for 20, 30, and particularly 40 min were positioned on the negative side of PC1 and were associated with 2-furanmethanol. This distribution suggests that milder roasting conditions favor the formation or retention of early-stage furan derivatives. Previous studies have reported that furfuryl alcohol and related compounds are abundant in lightly roasted coffee products and may subsequently undergo further transformation during prolonged thermal treatment **(Moon & Shibamoto, 2009; Swasti & Murkovic, 2012)**.

Along the PC2 axis, 2-furancarboxaldehyde, 5-(hydroxymethyl)- (5-HMF) and 2,5-dimethyl-4-hydroxy-3(2H)-furanone were partially separated from the other furan-related compounds. This pattern suggests that the formation of these compounds is influenced not only by roasting temperature but also by roasting duration and the extent of carbohydrate degradation. **Murkovic and Bornik (2007)** reported that 5-HMF is rapidly generated during roasting but may subsequently degrade during prolonged heating. Similarly, **Liu et al. (2018)** observed significant increases in furaneol and HMF formation at elevated roasting temperatures, while **Poisson et al. (2019)** associated the formation of hydroxylated furanones with advanced sugar degradation pathways.

Overall, PCA differentiated the roasting treatments according to the measured furan-related volatile profiles. More intensive roasting conditions were associated with higher abundances of furan, 3-furanmethanol, and selected benzofuran derivatives, whereas milder treatments were associated with a greater relative contribution of 2-furanmethanol. These findings demonstrate the usefulness of PCA for visualizing differences among roasting treatments but should not be interpreted as direct evidence of specific chemical reaction mechanisms or sensory quality.

3.3.3. Hierarchical Cluster Analysis

Hierarchical cluster analysis (HCA) combined with a clustered heatmap was performed to evaluate the distribution of volatile compounds under different roasting conditions (**Fig. 6**). The dendrogram clearly separated the samples into three major clusters corresponding to low- (127–150 °C), intermediate- (160–170 °C) and high-intensity (200–212 °C) roasting conditions, indicating that roasting severity was the primary factor governing changes in volatile composition. Similar clustering patterns associated with roasting intensity have previously been reported for roasted coffee **(Baggenstoss et al., 2008; Ruosi et al., 2012)**.

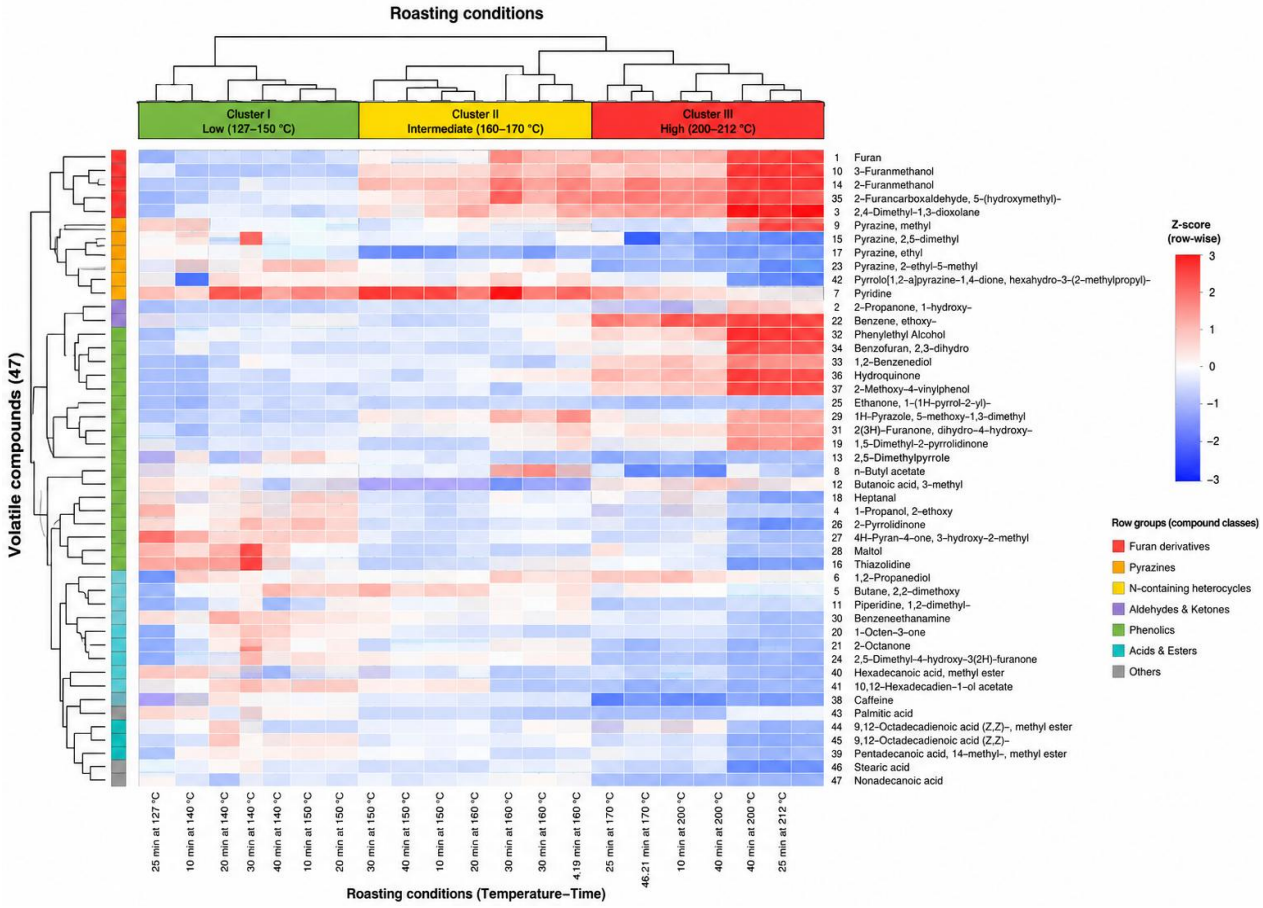


Figure 6. Hierarchical clustered heatmap (AHC) of volatile compounds under different roasting conditions.

Cluster I (127–150 °C) was characterized by relatively low abundances of furan derivatives, pyrazines, and phenolic compounds, together with comparatively higher levels of lipid-derived volatiles, consistent with less extensive thermal transformation under mild roasting conditions **(Moon et al., 2009; Zakidou et al., 2021)**. Cluster II (160–170 °C)

represented a transitional stage, where pyrazines, phenolic compounds, and nitrogen-containing heterocycles became more abundant, indicating changes in the aroma-related chemical profile. Similar observations have been reported for intermediate roasting conditions, where aroma generation is enhanced while thermal degradation remains relatively limited (Baggenstoss et al., 2008).

Cluster III (200–212 °C) was strongly associated with furan, 3-furanmethanol, benzofuran, hydroquinone, 2-methoxy-4-vinylphenol, and several pyrazine derivatives, consistent with greater contributions from compounds commonly associated with thermal processing. Similar trends have been reported for highly roasted coffee, where increasing roasting severity promotes the accumulation of both furanic and pyrazine compounds (**Buffo & Cardelli-Freire, 2004; Spreng et al., 2021; Zakidou et al., 2021)**.

The hierarchical clustering patterns indicated that several volatile compounds exhibited similar responses across the investigated roasting conditions. However, these groupings reflect similarities in their measured abundance profiles and do not independently establish common formation pathways.

Overall, the HCA results were broadly consistent with the Pearson correlation analysis and PCA, supporting an association between roasting severity and differences in furan concentrations and volatile composition.

The present study has several limitations. Because Dibek-style grinding was performed after roasting and no conventional-grinding control was included, the specific contribution of the grinding procedure could not be determined. Furthermore, the precursors and reaction intermediates involved in the proposed thermal pathways were not measured, limiting direct mechanistic interpretation. The 47 identified volatile compounds represent the compounds detected under the selected analytical conditions rather than an exhaustive characterization of coffee aroma. As sensory evaluation, GC–olfactometry, and odor activity value analysis were not performed, the results cannot establish sensory quality or consumer preference. Finally, the assessment of processing contaminants was restricted to furan, without determination of other relevant compounds such as acrylamide.

4. Conclusion

This study demonstrated that roasting temperature was the dominant factor influencing furan formation and volatile composition in Dibek coffee. Furan concentrations increased from 18.24 ± 0.61 to 52.12 ± 0.22 μg kg^{-1} across the investigated roasting conditions. A total of 47 volatile compounds were identified, with more intensive roasting generally promoting

pyrazines, furan derivatives, and selected phenolic compounds, whereas several aldehydes and ketones were more abundant under milder conditions. Correlation analysis, PCA, and HCA revealed distinct associations between roasting intensity, furan concentrations, and volatile profiles. Although the RSM model explained a substantial proportion of the observed variation (R^2 = 0.952), its significant lack-of-fit warrants caution when making predictions. Overall, the findings demonstrate that roasting conditions affect both aroma-related chemical composition and furan accumulation, providing a basis for further studies aimed at developing roasting strategies that consider volatile composition and process contaminant formation.

Ethical approval

This study did not involve human participants or animals; therefore, ethical approval was not required.

Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Declaration on the Use of Generative AI in Scientific Writing

During the preparation of this work, the corresponding author used ChatGPT (OpenAI) to improve language and readability. After using this tool, the author reviewed and edited the content as needed and takes full responsibility for the publication's content.

Author Contributions

Ali Gundogdu (A.G.): Conceptualization, Methodology, Investigation, Data curation, Visualization, Writing - original draft, Writing - review & editing, Project administration.
Cemalettin Baltaci (C.B.): Conceptualization, Methodology, Investigation, Supervision, Formal analysis, Writing - review & editing.
Merve Seyda Karatas (M.S.K.): Methodology, Investigation, Writing - review & editing.
Arda Akdoğan (A.A.): Methodology, Investigation, Formal analysis, Data curation.
Firat Yilmaz (F.Y.): Methodology, Investigation, Formal analysis, Data curation, Visualization.
Erdogan Yildiz (E.Y.): Methodology, Investigation, Formal analysis, Data curation.

Data availability

Data will be made available on reasonable request.

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