Sánchez-García, JM.; Muñoz-Pina, S.; García Hernández, J.; Heredia Gutiérrez, AB.; Andrés Grau, AM. (2024). Volatile profile of quinoa and lentil flour under fungal fermentation and drying. Food Chemistry. 430. https://doi.org/10.1016/j.foodchem.2023.137082

VOLATILE PROFILE OF QUINOA AND LENTIL FLOUR UNDER FUNGAL FERMENTATION AND DRYING

Janaina Sánchez-García[1], Sara Muñoz-Pina[1*], Jorge García-Hernández[2], Ana Heredia[1], Ana Andrés[1]

[1] *Instituto Universitario de Ingeniería de Alimentos para el Desarrollo (IUIAD-UPV). Universitat Politècnica de València. Camino de Vera s/n, 46022, Valencia, Spain.*

[2] *Centro Avanzado de Microbiología de Alimentos (CAMA), Universitat Politècnica de València. Camino de Vera s/n, 46022, Valencia, Spain.*

**Corresponding author (e-mail: samuopi@upvnet.upv.es)*

ABSTRACT

Solid-state fermentation reportedly improves the nutritional and sensory properties of legumes and pseudocereals. This study examined changes in the volatile profile using HS-SPME-GC-MS of two varieties of lentil and quinoa flour fermented with *Pleurotus ostreatus* and dried using hot-air drying and lyophilisation. Fermentation significantly increased the volatile profile. Pardina lentil flour showed a 570% increase in its volatile profile, and 10 compounds were created. In white quinoa, the total area rose from 96 to 4500, and 30 compounds were created. Compounds such as 1-octen-3-ol, benzaldehyde, 3-octanone and hexanal were generated during fermentation, providing a sweet, grassy, cocoa flavour. Hot-air drying led to decrease of over 40% in total peak area. Dried fermented flour retained higher levels of compounds that provide a sweet, cocoa aroma. Air-drying temperature had no significant influence on the volatile profile. This a allows the inclusion of these flours in a wide variety of food products.

Keywords: Volatile compounds; quinoa; lentil; flour; fermentation; *Pleurotus ostreatus*

1. Introduction

Global concern for environmental sustainability and food security, coupled with a focus on healthy eating and low costs, is driving the search for new plant-based high-protein foods. Legumes and pseudocereals play a prominent role in ensuring a balanced human diet worldwide. They offer large quantities of macro- and micro-nutrients. They also have several environmental benefits. For instance, legume cultivation reduces greenhouse gas emissions, helps fix atmospheric nitrogen in the soil and decreases the carbon footprint (Stagnari et al., 2017).

Legumes are a rich source of protein. Their protein content ranges from 21% to 31% on a dry basis, depending on the species and crop. These proteins are high in essential amino acids such as lysine and leucine. Studies have emphasised the importance of consuming legumes not only because they offer a source of protein but also because they are high in dietary fibre, minerals and polyphenols. Lentils (*Lens culinaris spp*.) are a type of legume cultivated in over 70 countries. They are valued around the world for their nutritional richness in proteins, dietary fibres, complex carbohydrates and essential micronutrients such as iron, zinc and vitamin B complex(Liberal et al., 2023). In addition, lentil seeds have a higher antioxidant capacity than other legumes due to the presence of certain phenolic compounds (Grela et al., 2017).

Another group of crops that offer large quantities of macro- and micro-nutrients is pseudocereals. They are resilient crops capable of withstanding salinity and extreme temperatures, and they can be easily grown with limited resources (Rodríguez et al., 2020). Quinoa seeds *(Chenopodium Willd*) offer an alternative gluten-free protein source with nutritional value similar to that of cow's milk (Repo-Carrasco et al., 2006). The amino acid profile of quinoa includes all essential amino acids, with high quantities of lysine. Quinoa is also rich in various vitamins and minerals, with a higher protein content than traditional grains (Repo-Carrasco et al., 2006).

Despite all their nutritional benefits, legumes and pseudocereals also contain antinutrients such as protease inhibitors, phytates, tannins and saponins. These antinutrients can affect the nutritional quality of these foods. Certain processing methods such as heat treatments, germination and fermentation appear to reduce these antinutrients and increase the bioaccessibility of bioactive compounds (Thakur et al., 2021). One such processing method is solid-state fermentation (SSF). It involves the microbial fermentation of a substrate without water, resulting in greater productivity than other forms of fermentation. Many microorganisms are susceptible to fermentation. The fungal kingdom offers certain advantages over other microorganisms. These advantages include a high protein content (20% to 30% in dry matter) and protein quality. Fungi also serve as a source of dietary fibre and have a high vitamin B content and low fat content.

During the fermentation process, fungi play a crucial role in the formation of different volatile compounds, primarily through the hydrolysis of large molecules such as lipids and proteins into fatty acids and amino acids. This process provides precursors for the formation of a variety of volatile organic compounds (VOCs), including acids, aldehydes, ketones, alcohols, esters and hydrocarbons (Zhong et al., 2022). Both fermentation and drying methods tend to affect the volatile profile of legumes, cereals, and other foods, leading to variations and the generation of new compounds. These variations can have a positive effect on the creation of new ingredients, with an improved volatile profile. Studies have shown that legumes fermented by *Lactobacillus* have more flavour, potentially reducing their beany flavour while forming aldehydes, alcohols, acids and sulphur compounds through further biotransformation (Yi et al., 2021). However, processes such as hot-air drying and freeze drying can affect VOCs, which are necessary to provide stable fermented ingredients such as flour. For instance, hot-air drying has been observed to decrease aldehydes, alcohols and esters in volatile compounds of coffee beans (Zhang et al., 2022). Different drying methods can affect the final volatile profile in different ways (Rajkumar et al., 2017).

Despite the existence of studies of the effect of bacteria- and fungi-based fermentation on the volatile profile of fermented substrates, no studies have examined the influence of *Pleurotus ostreatus* fermentation on the volatile profile of quinoa and lentils. The hypothesis tested in the present study is that the volatile profile changes after fermentation and drying of quinoa and lentil flour. This process thereby modifies the final aroma, which is one of the main sensory considerations when choosing new food products. Thus, the aim of this study is to analyse the impact of SSF with *Pleurotus ostreatus* and drying (air-drying at 50 °C, 60 °C and 70 °C and lyophilisation) on the volatile profile of two varieties of lentil and quinoa flour. The findings will be useful for developing new products based on high-protein flour.

2. Materials and methods

2.1. Materials

Hacendado® brand lentils (*Lens culinaris*) of the Pardina and Castellana varieties were purchased from local shops in Valencia, Spain. Hacendado® and Nut&me brand quinoa (*Chenopodium quinoa Wild*) of white and black varieties, respectively, was also purchased from local shops in Valencia, Spain. The *Pleurotus ostreatus* strain was obtained from the Spanish Type Culture Collection (CECT20311). Sodium chloride (NaCl) was obtained from Sigma-Aldrich Co. (St. Louis, MO, USA). Glucose and mycopeptone were also obtained from Sigma-Aldrich Co. (St. Louis, MO, USA). Malt extract and agar-agar were obtained from Scharlau (Barcelona, Spain).

2.2. Fungal solid-state fermentation

Starter culture preparation

A starter culture was prepared by hydrating 10 g of lentil and quinoa flour to 65% moisture. Sterilisation was performed using an autoclave (Vertical Stand Autoclave 4002136, JP Selecta™, Barcelona, Spain; for 20 min at 121°C). Then, 1 mL of *Pleurotus ostreatus* culture was added and kept for 14 days at 28 °C in a digital incubator (digital oven 2001249, JP Selecta™, Barcelona,

Spain). *Pleurotus ostreatus* mycelium was previously grown in a culture broth prepared with 2% glucose, 2% malt extract and 0.1% mycopeptone. It was incubated for 14 days at 28 °C in a digital incubator (JP Selecta™ digital oven 4002136, Barcelona, Spain).

Fermentation process

The SSF process was conducted following the method described by Sánchez-García et al. (2023). In total, 35 g of lentils and quinoa at 65% moisture were placed in each glass jar and sterilised in an autoclave (Vertical Stand Autoclave 4002136, JP Selecta™, Barcelona, Spain) for 20 min at 121°C. To achieve a moisture content of 65%, a preliminary material balance calculation was performed. The samples were then mixed with water and left for 20 minutes. Next, the moisture content was checked and autoclaved. *Pleurotus ostreatus* was then inoculated from the starter culture by adding a 1/8 portion. The jars were incubated at 28 °C for 14 days in a digital oven (2001249, JP Selecta™, Barcelona, Spain).

2.3. Drying and milling of fermented grains and seeds

To prepare the different flours, 500 g of fermented quinoa and lentils were hot-air dried at three different temperatures (50, 60 and 70 °C) in a convective dryer (Pol-Eko-Aparatura, CLW 750 TOP+, Wodzisław Śląski, Poland) following the method described by Sánchez-García et al. (2023). As a control, freeze drying was performed using a freeze dryer (Lyoquest-55, Telstar, Terrassa, Spain), as described by Sánchez-García et al. (2023). After drying, the samples were milled into flour using a food processor at 15-second intervals for 1 min at 10,000 rpm (Thermomix® TM6-1, Vorwerk, Wuppertal, Germany). Multiple glass jars were inoculated to produce an adequate amount of fermented substrate to conduct the experiments. The fermented substrates were combined to create a uniform sample for the subsequent drying process and analysis.

2.4. HS-SPME-GC-MS volatile compounds

The volatile compounds in unfermented, fermented and fermented-dried quinoa and lentil flours were determined using headspace-solid phase microextraction (HS-SPME) and were

analysed by gas chromatography/mass spectrometry (GC/MS) following the method described by Escriche et al. (2022). Briefly, 2.5 g of sample and 7.5 mL of 20% w/v sodium chloride were added to a 20 mL glass vial with a screw cap and a PTFE-silicone septum. The mixture was homogenised using a vortex mixer. The sample was then heated on a stirring heating platform at 50 °C and 250 rpm for 30 min. Volatile compounds were trapped using a divinylbenzene/carboxene/polydimethylsiloxane fibre (DVB/CAR/PDMS 50/30 µm). Volatile compounds were analysed using a gas chromatograph (Intuvo 9000, Agilent Technologies, Palo Alto, CA, USA) coupled with a triple quadrupole detector (7000 Series GC/TQ, Agilent Technologies, , Palo Alto, CA, USA) that was fitted with an electron ionisation source at 70 eV. A capillary column (DB WAX, 30 m × 0.25 mm × 0.25 µm, Agilent J&W, Santa Clara, CA, USA) with helium as the carrier gas at a constant flow rate of 1 mL/min was used. The MassHunter Workstation software (Unknown analysis) was used for the data analysis and identification of volatile compounds. The mass spectra of each compound were analysed using the NIST spectral library (NIST 17, National Institute of Standards and Technology). The procedure used a coincidence factor of ≥ 80% and the linear retention indices (LRI). The results are presented as peak area/100,000 for ease of reading. The total amount of volatile compounds was estimated by summing all areas of the chromatogram.

2.5. Statistical analysis

The analytical data were analysed using one-way analysis of variance (ANOVA). The least significant difference (LSD) Fisher test was performed to identify homogeneous groups among different drying temperatures. The R programming language (version 4.2.2) was used for analysis. The confidence level was set to 95 % (p-value < 0.05). All experiments were conducted in triplicate. The data are reported as the mean ± standard deviation.

3. Results and discussion

3.1. Changes in volatile profile due to fungal solid-state fermentation

The fermentation process *per se* strongly influences the flavour and aroma of the final product. Fungal SSF with *Pleurotus ostreatus* causes different changes depending on the characteristics of the initial substrates and the cultivar. Hence, there may be significant differences in composition, structure, seed coat to cotyledon ratio and seed size (Espinosa-Páez et al., 2017). The use of HS-SPME-GC-MS showed the generation of sufficient amounts of volatile compounds during fermentation in lentils and quinoa (Tables 1 to 4). In general, none of the unfermented flour types had a varied volatile profile before fermentation. More volatile compounds were found for white quinoa (Table 1) than for black quinoa (Table 2). Unfermented black quinoa had nine volatile compounds, constituting 32 ± 9 of the total peak area. These volatile compounds consisted of three aldehydes, one alcohol, one ketone, two heterocyclic compounds, one phenolic compound and one other compound. Unfermented white quinoa had a significantly superior aroma, with a sum of 96 ± 2 in the total peak area. Yang et al. (2021) also observed and correlated these differences between different colour quinoas. In unfermented white quinoa, the concentration and variety of alcohols was higher than in black quinoa. The highest compound was 1-Hexanol (Song et al., 2021; Yang et al., 2021). In unfermented white quinoa (UFWQ), 2-pentylfuran, 1-octen-3-ol, and benzaldehyde were the individual substances that contributed the most to its aroma, in addition to hexanol. The substance 1-Hexanol is correlated with a grassy/green odour, whereas 2-pentylfuran and benzaldehyde were found to give out strong nutty aromas (Yang et al., 2021; Zhang et al., 2019).

On the other hand, unfermented lentils had a greater volatile profile than that of black quinoa and almost double that of white quinoa (Tables 3 and 4). There were no statistically significant differences between lentil varieties. Nonetheless, the volatile profiles of Pardina and Castellana lentils differed both in concentration and compounds. Unfermented Pardina lentils (UFPL) (Table 3) had nine compounds, mainly belonging to alcohol and aldehyde chemical groups. Castellana lentils (Table 4) also contained heterocyclic compounds such as furfural and furfuryl

methylamphetamine. In both lentil varieties, 1-hexanol and hexanal were highly present, but in different ratios. In unfermented Pardina lentils (UFPL), the aldehyde hexanal contributed most to the aroma. Its peak area was almost four times lower in unfermented Castellana lentils. The alcohol 1-hexanol was the most present in unfermented Castellana lentils. Although there are no studies of the volatile profile of these lentil varieties, both 1-hexanol and hexanal have been found in red and green lentil flour, with different results in each case (Paucean et al., 2018). Hexanal provided a green, grassy, leafy odour. Furfural, the heterocycle compound, also played an important role in the odour of the unfermented Castellana lentils (UFCL), providing a soil/roasted odour that was absent in unfermented Pardina lentils (UFPL) (Paraskevopoulou et al., 2012).

Regarding the impact of fungal fermentation, the volatile profiles changed notably during fermentation, with *Pleurotus ostreatus* significantly raising the flavour. Fungi are highly polymorphic in their ability to produce a unique profile of VOCs. Each species emits a profile that changes qualitatively and quantitatively. Profiles vary according to the genotype of strains and species. In addition, VOC profiles are influenced by the physical environment, as well as factors such as the age of the fungal colony, the availability of water, the type of substrate, temperature and the presence of interacting species. Each mushroom species has a characteristic mixture of volatile flavour components that may include additional aliphatic, terpenoid, aromatic and sulphur-containing compounds (Inamdar et al., 2020). During the process of fermentation and fungal growth, complex biochemical changes take place to create the characteristic aroma. Recent data have shown that fermentation can modify the metabolism of flavour-related compounds (Zhang et al., 2021).

The Pardina lentil was the least affected by fermentation, despite increasing the total peak area by 570% with respect to the unfermented Pardina lentil. On the contrary, fermented white quinoa (FWQ) was the most affected by fermentation, raising the total area from 96 to 4500. Four common compounds were observed in the volatile profile of the fermented lentils and

quinoa. These compounds were also the main contributors to the aroma of the fermented flour. They were 1-octen-3-ol, benzaldehyde, hexanal and 3-methoxybenzaldehyde. The latter two compounds were not found in Pardina lentils. It is well known that the dominant aroma associated with mushrooms is due to a mixture of aliphatic, oxygenated and 8-carbon compounds. They all function as host location cues, especially 1-octen-3-ol, which is related to the odour of mushrooms (Xu et al., 2019; Yang et al., 2019). Indeed, Aisala et al. (2019) found that compounds containing 8-carbons are responsible for mushroom-like odour, being 1-octen-3-ol and 1-octen-3-one the most important. Other compounds such as 1-octanol, 3-octanol, octenal, 2-octenal, 3-octanona and 3-octen-2-one are also typically found in mushrooms. Therefore, the fungal metabolism could be expected to be responsible for generating these compounds during fermentation in the four flour types included in this study.

Benzaldehyde has also been linked to *Pleurotus ostreatus*, giving off sweet and fruity notes (Beltran-Garcia et al., 1997). Besides the sweet aroma, this aldehyde is found in cocoa beans and is linked to the aroma of cocoa (Escobar et al., 2021; Mohamadi Alasti et al., 2019). Unsurprisingly, benzaldehyde is the second most found compound in white and black quinoa flour, since when one smells the flour, the sweet and cocoa aroma is evoked. Fermentation with *Pleurotus ostreatus* transformed the initially odourless unfermented lentil and quinoa samples into sweet-cocoa smelling flour, generating a pleasant and appetizing flavour that does not typically exist in legumes and pseudocereal flour.

In addition to these major compounds, a cascade of reactions was triggered during fermentation. These reactions increased the complexity of the volatile profile in both lentil and quinoa flour. In fermented Castellana lentils (FCL), six alcohols, 14 aldehydes, two ketones, four heterocyclic compounds, one phenolic compound and three esters were formed, enriching their aroma. Nonanal provided citrus notes, benzeneacetaldehyde provided grassy and flowery aromas, and 2-Ethyl-5-methylpyrazine provided a nutty, roasted and chocolaty flavour (Aisala et al., 2019; FAO, 2023; Qian et al., 2019). The compound 1-hexanol was prevalent in unfermented

Castellana lentils. This compound decreased significantly with benzyl alcohol during fermentation.

Fermented Pardina lentils presented only four alcohols, seven aldehydes, three ketones, one heterocyclic compound and one phenolic compound after fermentation. Ketones mainly provided fresh, herbaceous, woody and fruity flavours (Xu et al., 2019). Other volatile components such as 1-hepten-3-one provided an earthy, green odour (Ebert et al., 2022).

Finally, in quinoa seeds the number of compounds increased significantly after fermentation. The white quinoa profile increased from 13 to 41 compounds, whereas the black quinoa profile rose from 9 to 26. In both cases, aldehydes were the most affected chemical group. The aroma of fermented black quinoa (FBQ) was also characterised by other compounds such as 2-heptanal, nonanal and furfural. Despite being found in lower concentrations, these compounds also provided the FBQ with a fruity, sweet, cooked-bean aroma (Hao et al., 2023; Sharan et al., 2022). In fermented white quinoa, other compounds such as 1-heptanol and 2-methyl-1-butanol also formed. The concentration of the alcohol 1-heptanol multiplied 40 times, contributing to a sweeter final aroma after fermentation (Wu et al., 2021). Compounds such as decanal increased this sweet aroma. After fermentation, furan compounds such as 2-Pentylfuran, ethyl 2-(5-methyl-5-vinyltetrahydrofuran-2-yl) propan-2-yl carbonate, 2-propylfuran, 2-nButyl-furan, 3-penthylfuran, and 2(3H)-Furanone, dihydro-5-pentyl- increased/formed. The generation of furan compounds in the volatile profile resulted from sterilisation prior to fermentation rather than the fungal metabolism itself (Maga & Katz, 2009). Finally, esters were also generated after fermentation in fermented white quinoa. They were responsible for fruity odours (Ouellette & Rawn, 2014). In short, the aroma of white quinoa was the most affected by SSF with *Pleurotus ostreatus,* generating a new, more complex, sweeter aroma than that of unfermented white quinoa.

3.2. Impact of dehydration on the volatile profile of fermented quinoa and lentil flour

After fermentation, the samples were submitted to hot-air drying at three temperatures (50 °C, 60 °C and 70 °C), as well as lyophilisation. The impact of drying on the volatile profile seemed to depend on the substrate. The results appear in Tables 1 to 4.

Dried fermented white quinoa flour (Table 1) decreased in total volatile volume from 4500 to less than 2500 total peak area. This decrease means that the total volatile compounds fell by 50%. There were no significant differences between the results for different hot-air drying temperatures and drying methods. However, the profile differed slightly between white quinoa flour. Some compounds, such as 4-Ethylcyclohexanol, 2-hydroxy-benzaldehyde and 2-undecanone, disappeared completely after drying. Meanwhile, the presence of others decreased or increased significantly. The amount of hexanal and 2-pentylfuran decreased by more than 50% after lyophilisation. In contrast, the amount of 3-Octanol and 1-Octen-3-ol increased significantly. This combination resulted in lyophilised fermented white quinoa (FWQ-L) with a less nutty aroma but a stronger mushroom odour than hot-air dried fermented white quinoa. Similar results are reported in the literature for the comparison between hot-air drying and freeze drying. Rajkumar et al. (2017) reported that some compounds were formed after freeze drying and/or hot-air drying of cabbage, whereas other compounds did not withstand drying and disappeared. Four main routes are involved in the formation of volatile flavour compounds during drying. These routes include Maillard reactions, long-chain compound degradation and lipid oxidation and degradation (Deng et al., 2015; Yang et al., 2016). The loss of volatile flavour compounds is more common with increased temperature at the same moisture content due to thermal degradation, volatilisation and other chemical reactions (Ge et al., 2020).

In the case of fermented black quinoa flour (Table 2), the sum of volatile compounds in the hot-air dried samples decreased by approximately 65% with respect to fermented black quinoa flour. There were no significant differences between fermented black quinoa dried at 50 °C, 60 °C and 70 °C. However, FBQ-L did not change significantly (1960 ± 150) from fermented black quinoa

285 (2310 ± 200). Almost all compounds that increased (hexanol, 3-Octanol and 1-Octen-3-ol) and
286 decreased (hexanal) in FWQ-L also decreased in lyophilised fermented black quinoa. However,
287 other compounds, such as 3-methoxybenzaldehyde increased significantly in FBQ-L. Thus, the
288 concentration of volatiles was equal to that found in fermented black quinoa. The results imply
289 that hot-air drying significantly reduced all volatile compounds, except for 2-pentylfuran, which
290 increased. In addition, another three compounds (ethyl octanoate, beta-bisabolene and 2,3-
291 dihydro-1H-indene-4-carbaldehyde) were generated with drying, regardless of temperature. As
292 noted earlier, the formation of furan compounds such as 2-pentylfuran increased with
293 temperature. Ge et al. (2020) studied the volatile flavour in peppers dried at different
294 temperatures, finding that some furan compounds increased with temperature under Strecker
295 degradation. They also found that the compound ethyl octanoate increased significantly ($p <$
296 0.05) due to drying.
297 There were also changes in fermented lentil flour following drying. The total sum of volatiles in
298 Pardina lentil flour (Table 3) decreased the least after drying, falling by only around 30%.
299 However, an interesting effect occurred at 60 °C in fermented Pardina lentils, where the total
300 sum of the areas decreased by 70%. This finding is mainly due to a decrease in the aldehyde
301 benzaldehyde area from 405 to 129. Meanwhile, an increase in this compound was found in FPL
302 dried at 50 °C, 70°C, and lyophilised. According to previous studies, this compound commonly
303 increases after drying due to the degradation of benzoic acid into benzaldehyde (Pei et al., 2016).
304 However, this compound decreased or maintained its presence in all other hot-air dried flour.
305 This volatile compound was the most prevalent in Pardina lentil flour, giving it a nutty smell.
306 Therefore, FPL-60 flavour would be milder in this hue than its equivalents.
307 The total peak area of fermented Castellana lentils dried at the three different temperatures
308 (FCL-50, FCL-60, and FCL-70) decreased by 70% with respect to fermented Castellana lentils.
309 Even though fermentation enriched the volatile profile in Castellana lentils to a greater extent
310 than in Pardina lentils, hot-air drying led to a loss of aroma because of the drastic reduction of

311 relevant compounds such as 1-octen-3-ol, hexanal, benzaldehyde and 3-methoxybenzaldehyde.
312 However, lyophilised fermented Castellana lentil (FCL-L) significantly improved in terms of the
313 concentration of these compounds. This greater concentration intensified the flavour of
314 fermented Castellana lentil flour by 118%. Alcohols and aldehydes were the most affected
315 chemical groups, gradually diminishing as air temperature increased. Similar results have been
316 reported for hot-air dried peppers (Ge et al., 2020). Given these changes, lyophilised fermented
317 Castellana lentils had a green, grassy, leafy odour with nut and mushroom aromas. In the case
318 of fermented Castellana lentils dried at 50 °C, 60 °C and 70 °C, the aroma had sweet, burnt, and
319 baked notes due to the pyrazines and 3-pentylfuran generated by Strecker degradation and
320 Maillard reactions in hot-air drying (Fischer et al., 2017).
321 In sum, lyophilisation was much better in terms of maintenance of volatile compounds in the
322 samples of black quinoa and Castellana lentils. In black quinoa, there was no loss of volatiles. In
323 Castellana lentils, the total peak area increased by 14%. However, for white quinoa, there were
324 no differences between types of drying or temperatures of hot-air drying. All reduced total peak
325 area by 50%. In the case of Pardina lentils, drying at 60 °C provided lower results. In contrast,
326 hot-air drying at 50 °C and 70 °C and freeze drying did not lead to significant differences. These
327 processes affected the sample in the same way. Thus, even though hot-air drying led to a loss of
328 the aroma generated during fermentation, the sweet, fruity, cocoa smell with hints of
329 mushrooms and cooked substrates that was observed in the undried fermented samples was
330 preserved, given the high concentrations of benzaldehyde, hexanal, nonanal, furfural and 1-
331 octen-3-ol.
332

3.3. Principal component analysis of the volatile profile of different flours

Principal component analysis (PCA) was performed to visualise the contribution of the volatile compounds to the flavour profile of each flour. This analysis highlighted differences in the generation of volatiles during SSF and the drying of samples. Figure 1 shows the biplot for each substrate (Castellana lentils, Pardina lentils, white quinoa and black quinoa). The sum of the first two principal components (PC1 and PC2) explained 74.49%, 75.02%, 80.74% and 83.53% of the total variance of Pardina lentils, Castellana lentils, white quinoa and black quinoa, respectively. A consistent pattern in sample clustering was observed for all substrates. PC1 distinguished unfermented samples (negative axis) from fermented and lyophilised samples (positive axis), except for white quinoa. Lyophilised fermented white quinoa was closer to UWQ. PC2 distinguished hot-air dried samples (positive axis) from unfermented and fermented samples (negative axis). In the case of lyophilised flour, PC2 grouped these samples together with hot-air dried samples for Castellana lentils and black quinoa. However, it distinguished between the samples for Pardina lentils and white quinoa. Therefore, freeze drying had different effects on the volatile profile depending on the substrate. The values of the variables defining the PC1 and PC2 equations were standardised by subtracting the means and dividing by the standard deviations of the volatile compounds. More strongly positive or negative values meant a greater contribution to explaining variability in the data. The volatile compounds that contributed the most to discriminating Pardina lentil samples (Figure 1A) in PC1 were the alcohols 2-ethyl-1-hexanol, 1-hexanol and 1-Heptanol and the aldehyde hexanal. In PC1, the concentrations of the heterocyclic compound 3-ethyl-2,5-dimethylpyrazine, phenylethyl alcohol and the aldehydes 3-chloro-4-methoxybenzaldehyde, 3,5,5-trimethyl-1-hexanal and 1-Hepten-3-one distinguished hot-air dried samples from the other samples. For Castellana lentils (Figure 1B), differences in concentrations of some alcohols (3-octanol, 1-octanol and cyclooctyl alcohol), the ester methyl 4-methoxybenzoate and some aldehydes (Hexanal, 4-Ethylbenzaldehyde and 2-Phenylpropenal) distinguished unfermented flour from fermented and lyophilised flour (PC1). Hot-air dried

359 fermented Castellana flour was characterised by higher concentrations of acetophenone,
360 benzeneacetaldehyde, furans and pyrazines. The aroma of unfermented flour was characterised
361 by the presence of higher concentrations of some alcohols (1-hexanol, 2-ethyl-1-hexanol, 1-
362 nonanol and benzyl alcohol) and furfural (PC2). Briefly, white quinoa flour had the most complex
363 profile. Fermentation gave a new flavour to the flour, as reflected by the location of
364 unfermented white quinoa with respect to fermented white quinoa (PC1) and the volatile
365 compounds near the samples. White quinoa hot-air dried samples at three temperatures (50,
366 60 and 70 °C) (FWQ-50, FWQ-60, FWQ-70) differed from UWQ and FWQ (PC2). In contrast, the
367 fermented lyophilised samples had similarities with unfermented flour. A similar trend was
368 observed for black quinoa. Unfermented black quinoa was characterised by naphthalene and 4-
369 Isothiocyanate-1butene. In contrast, high concentrations of 3-octanone, Hexanal, 1-octen-3-ol
370 and benzaldehyde defined FBQ. Finally, PC2 distinguished hot-air dried samples based on 2,3-
371 dihydro-1H-indene-4-carbaldehyde, furfural and ethyl octanoate from the other samples.
372 These findings suggest that freeze drying is the optimal method for preserving desirable odours
373 in the types of flour under study. However, despite its effectiveness for certain ingredients such
374 as black quinoa and Pardina lentils, freeze drying is rarely used in the industry due to cost.
375 Instead, hot-air drying is more common. This method provides comparable results to freeze
376 drying for white quinoa and Castellana lentils.
377 Therefore, it is crucial to evaluate the impact of hot-air drying on sensory attributes such as
378 odour when developing new products. Freeze drying should be used as a control. All
379 temperature variations tested in this study had similar effects on the samples. Considering only
380 the influence on volatile compounds, higher temperatures would be preferred because they
381 enable faster drying and reduce energy consumption. However, further studies on other
382 physicochemical characteristics are necessary before recommending specific temperature
383 ranges. The main limitation of this study is that these results cannot be extrapolated to other

384 substrates. A possible escalation would require fine-tuning and readjustments. Larger-scale
385 trials should be carried out to check possible changes in the volatile profile.
386
387 **4. Conclusions**
388 Solid-state fermentation (SSF) with *Pleurotus ostreatus* enhances the volatile profile of lentils
389 and quinoa. Unfermented white and black quinoa had the least aroma, with a total peak area of
390 96 and 32, respectively. In contrast, unfermented lentils had a stronger, green, grassy, leafy
391 odour, with a total peak area of more than 160. After fungal fermentation, the volatile profiles
392 gained in complexity and intensity. Pardina lentils were the least affected by fermentation, with
393 a 570% increase in total peak area and the generation of 10 compounds. White quinoa aroma
394 was the most affected by fermentation. Total area rose from 96 to 4500, and 30 compounds
395 were created. Even though the volatile profile varied among samples, 8-carbon volatile
396 compounds were found in all fermented samples due to fungal fermentation. Benzaldehyde,
397 hexanal and 3-methoxybenzaldehyde were formed after fermentation, providing sweet, green,
398 cocoa aromas to the fermented lentils and quinoa. Hot-air drying significantly reduced the total
399 aromatic compounds by up to 40% in total peak area for fermented black quinoa flour. No
400 significant differences were found between different drying temperatures.

401 Dried fermented flour retained higher levels of the key compounds that provide a sweet, cocoa
402 aroma. Lyophilisation preserved the volatile compounds generated in fermentation to a greater
403 extent than hot-air drying in black quinoa and Castellana lentils (more than 150% in the total
404 peak area). In conclusion, fermented lentil and quinoa flours offer a richer and more intense
405 flavour than unfermented lentil and quinoa flours. This finding presents a new opportunity for
406 the inclusion of these flours in a wide variety of food products.

407 **Acknowledgements**

This research was conducted under project PID2019-107723RB-C22, funded by the Ministry of Science and Innovation MCIN/AEI/10.1309/501100011033. Sara Muñoz Pina was also a beneficiary of a post-doctoral grant from the Universitat Politècnica de València (PAID-10-21).

References

Aisala, H., Sola, J., Hopia, A., Linderborg, K. M., & Sandell, M. (2019). Odor-contributing volatile compounds of wild edible Nordic mushrooms analyzed with HS–SPME–GC–MS and HS–SPME–GC–O/FID. *Food Chemistry*, *283*, 566–578. https://doi.org/10.1016/J.FOODCHEM.2019.01.053

Beltran-Garcia, M. J., Estarron-Espinosa, M., & Ogura, T. (1997). Volatile Compounds Secreted by the Oyster Mushroom (Pleurotus ostreatus) and Their Antibacterial Activities. *Journal of Agricultural and Food Chemistry*, *45*(10), 4049–4052. https://doi.org/10.1021/JF960876I

Deng, Y., Luo, Y., Wang, Y., & Zhao, Y. (2015). Effect of different drying methods on the myosin structure, amino acid composition, protein digestibility and volatile profile of squid fillets. *Food Chemistry*, *171*, 168–176. https://doi.org/10.1016/J.FOODCHEM.2014.09.002

Ebert, S., Michel, W., Nedele, A. K., Baune, M. C., Terjung, N., Zhang, Y., Gibis, M., & Weiss, J. (2022). Influence of protein extraction and texturization on odor-active compounds of pea proteins. *Journal of the Science of Food and Agriculture*, *102*(3), 1021–1029. https://doi.org/10.1002/JSFA.11437

Escobar, S., Santander, M., Zuluaga, M., Chacón, I., Rodríguez, J., & Vaillant, F. (2021). Fine cocoa beans production: Tracking aroma precursors through a comprehensive analysis of flavor attributes formation. *Food Chemistry*, *365*, 130627. https://doi.org/10.1016/J.FOODCHEM.2021.130627

Escriche, I., Juan-Borrás, M., Visquert, M., Asensio-Grau, A., & Valiente, J. M. (2022). Volatile profile of Spanish raw citrus honey: The best strategy for its correct labeling. *Journal of Food Processing and Preservation*, *46*(6). https://doi.org/10.1111/JFPP.16200

Espinosa-Páez, E., Alanis-Guzmán, M. G., Hernández-Luna, C. E., Báez-González, J. G., Amaya-Guerra, C. A., & Andrés-Grau, A. M. (2017). Increasing antioxidant activity and protein digestibility in Phaseolus vulgaris and Avena sativa by fermentation with the Pleurotus ostreatus Fungus. *Molecules*, *22*(12), 2275. https://doi.org/10.3390/MOLECULES22122275

FAO. (2023). *Online Edition: "Specifications for Flavourings."* https://www.fao.org/food/food-safety-quality/scientific-advice/jecfa/jecfa-flav/details/en/c/829/

Fischer, M., Wohlfahrt, S., Varga, J., Matuschek, G., Saraji-Bozorgzad, M. R., Walte, A., Denner, T., & Zimmermann, R. (2017). Evolution of Volatile Flavor Compounds During Roasting of Nut Seeds by Thermogravimetry Coupled to Fast-Cycling Optical Heating Gas Chromatography-Mass Spectrometry with Electron and Photoionization. *Food Analytical Methods*, *10*(1), 49–62. https://doi.org/10.1007/S12161-016-0549-8/FIGURES/9

Ge, S., Chen, Y., Ding, S., Zhou, H., Jiang, L., Yi, Y., Deng, F., & Wang, R. (2020). Changes in volatile flavor compounds of peppers during hot air drying process based on headspace-gas chromatography-ion mobility spectrometry (HS-GC-IMS). *Journal of the Science of Food and Agriculture*, *100*(7), 3087–3098. https://doi.org/10.1002/JSFA.10341

Grela, E. R., Kiczorowska, B., Samolińska, W., Matras, J., Kiczorowski, P., Rybiński, W., & Hanczakowska, E. (2017). Chemical composition of leguminous seeds: part I—content of basic nutrients, amino acids, phytochemical compounds, and antioxidant activity. *European Food Research and Technology*, *243*(8), 1385–1395. https://doi.org/10.1007/S00217-017-2849-7/TABLES/5

Hao, Z., Zhang, X., Peng, X., Shi, X., Wang, R., & Guo, S. (2023). Identification of the key off-flavor odorants for undesirable spoiled odor in thermally sterilized fermented soymilk. *Food Research International*, *164*, 112407. https://doi.org/10.1016/J.FOODRES.2022.112407

Inamdar, A. A., Morath, S., & Bennett, J. W. (2020). Fungal Volatile Organic Compounds: More Than Just a Funky Smell? In *Annual Review of Microbiology* (Vol. 74, pp. 101–116). Annual Reviews. https://doi.org/10.1146/annurev-micro-012420-080428

Liberal, Â., Almeida, D., Fernandes, Â., Pereira, C., Ferreira, I. C. F. R., Vivar-Quintana, A. M., & Barros, L. (2023). Nutritional, chemical and antioxidant evaluation of Armuña lentil (Lens culinaris spp): Influence of season and soil. *Food Chemistry*, *411*, 135491. https://doi.org/10.1016/J.FOODCHEM.2023.135491

Maga, J. A., & Katz, I. (2009). Furans in foods. *Http://Dx.Doi.Org/10.1080/10408397909527268*, *11*(4), 355–400. https://doi.org/10.1080/10408397909527268

Mohamadi Alasti, F., Asefi, N., Maleki, R., & SeiiedlouHeris, S. S. (2019). Investigating the flavor compounds in the cocoa powder production process. *Food Science & Nutrition*, *7*(12), 3892–3901. https://doi.org/10.1002/FSN3.1244

Ouellette, R. J., & Rawn, J. D. (2014). Carboxylic Acid Derivatives. *Organic Chemistry*, 699–745. https://doi.org/10.1016/B978-0-12-800780-8.00021-8

Paraskevopoulou, A., Chrysanthou, A., & Koutidou, M. (2012). Characterisation of volatile compounds of lupin protein isolate-enriched wheat flour bread. *Food Research International*, *48*(2), 568–577. https://doi.org/10.1016/J.FOODRES.2012.05.028

Paucean, A., Moldovan, O. P., Mureșan, V., Socaci, S. A., Dulf, F. V., Alexa, E., Man, S. M., Mureșan, A. E., & Muste, S. (2018). Folic acid, minerals, amino-acids, fatty acids and volatile compounds of green and red lentils. Folic acid content optimization in wheat-lentils composite flours. *Chemistry Central Journal*, *12*(1), 1–9. https://doi.org/10.1186/S13065-018-0456-8/TABLES/4

Pei, F., Yang, W., Ma, N., Fang, Y., Zhao, L., An, X., Xin, Z., & Hu, Q. (2016). Effect of the two drying approaches on the volatile profiles of button mushroom (Agaricus bisporus) by headspace GC–MS and electronic nose. *LWT - Food Science and Technology*, *72*, 343–350. https://doi.org/10.1016/J.LWT.2016.05.004

Qian, C. Y., Quan, W. X., Xiang, Z. M., & Li, C. C. (2019). Characterization of Volatile Compounds in Four Different Rhododendron Flowers by GC×GC-QTOFMS. *Molecules 2019, Vol. 24, Page 3327*, *24*(18), 3327. https://doi.org/10.3390/MOLECULES24183327

Rajkumar, G., Shanmugam, S., Galvâo, M. de S., Dutra Sandes, R. D., Leite Neta, M. T. S., Narain, N., & Mujumdar, A. S. (2017). Comparative evaluation of physical properties and volatiles profile of cabbages subjected to hot air and freeze drying. *LWT*, *80*, 501–509. https://doi.org/10.1016/J.LWT.2017.03.020

Repo-Carrasco, R., Espinoza, C., & Jacobsen, S. E. (2006). Nutritional Value and Use of the Andean Crops Quinoa (Chenopodium quinoa) and Kañiwa (Chenopodium pallidicaule). *Http://Dx.Doi.Org/10.1081/FRI-120018884*, *19*(1–2), 179–189. https://doi.org/10.1081/FRI-120018884

Rodríguez, J. P., Rahman, H., Thushar, S., & Singh, R. K. (2020). Healthy and resilient cereals and pseudo-cereals for marginal agriculture: molecular advances for improving nutrient bioavailability. *Frontiers in Genetics*, *11*, 49. https://doi.org/10.3389/fgene.2020.00049

Sánchez-García, J., Muñoz-Pina, S., García-Hernández, J., Heredia, A., & Andrés, A. (2023). Impact of Air-Drying Temperature on Antioxidant Properties and ACE-Inhibiting Activity of Fungal Fermented Lentil Flour. *Foods 2023, Vol. 12, Page 999*, *12*(5), 999. https://doi.org/10.3390/FOODS12050999

Sharan, S., Zanghelini, G., Pernin, A., Descharles, N., Zotzel, J., Bonerz, D., Aschoff, J., Maillard, M. N., & Saint-Eve, A. (2022). Flavor of fava bean (Vicia faba L.) ingredients: Effect of processing and application conditions on odor-perception and headspace volatile chemistry. *Food Research International*, *159*, 111582. https://doi.org/10.1016/J.FOODRES.2022.111582

Song, J., Shao, Y., Yan, Y., Li, X., Peng, J., & Guo, L. (2021). Characterization of volatile profiles of three colored quinoas based on GC-IMS and PCA. *LWT*, *146*, 111292. https://doi.org/10.1016/J.LWT.2021.111292

Stagnari, F., Maggio, A., Galieni, A., & Pisante, M. (2017). Multiple benefits of legumes for agriculture sustainability: an overview. *Chemical and Biological Technologies in Agriculture*, *4*(2), 1–13. https://doi.org/10.1186/s40538-016-0085-1

Thakur, P., Kumar, K., & Dhaliwal, H. S. (2021). Nutritional facts, bio-active components and processing aspects of pseudocereals: A comprehensive review. *Food Bioscience*, *42*, 101170. https://doi.org/10.1016/J.FBIO.2021.101170

Wu, S., Yang, J., Dong, H., Liu, Q., Li, X., Zeng, X., & Bai, W. (2021). Key aroma compounds of Chinese dry-cured Spanish mackerel (Scomberomorus niphonius) and their potential metabolic mechanisms. *Food Chemistry*, *342*, 128381. https://doi.org/10.1016/J.FOODCHEM.2020.128381

Xu, M., Jin, Z., Lan, Y., Rao, J., & Chen, B. (2019). HS-SPME-GC-MS/olfactometry combined with chemometrics to assess the impact of germination on flavor attributes of chickpea, lentil, and yellow pea flours. *Food Chemistry*, *280*, 83–95. https://doi.org/10.1016/J.FOODCHEM.2018.12.048

Xu, X., Xu, R., Jia, Q., Feng, T., Huang, Q., Ho, C. T., & Song, S. (2019). Identification of dihydro-β-ionone as a key aroma compound in addition to C8 ketones and alcohols in Volvariella

volvacea mushroom. *Food Chemistry*, *293*, 333–339. https://doi.org/10.1016/J.FOODCHEM.2019.05.004

Yang, N., Liu, C., Liu, X., Degn, T. K., Munchow, M., & Fisk, I. (2016). Determination of volatile marker compounds of common coffee roast defects. *Food Chemistry*, *211*, 206–214. https://doi.org/10.1016/J.FOODCHEM.2016.04.124

Yang, W., Cadwallader, K. R., Liu, Y., Huang, M., & Sun, B. (2019). Characterization of typical potent odorants in raw and cooked Toona sinensis (A. Juss.) M. Roem. by instrumental-sensory analysis techniques. *Food Chemistry*, *282*, 153–163. https://doi.org/10.1016/J.FOODCHEM.2018.12.112

Yang, X., Zhu, K., Guo, H., Geng, Y., Lv, W., Wang, S., Guo, Y., Qin, P., & Ren, G. (2021). Characterization of volatile compounds in differently coloured Chenopodium quinoa seeds before and after cooking by headspace-gas chromatography-ion mobility spectrometry. *Food Chemistry*, *348*, 129086. https://doi.org/10.1016/J.FOODCHEM.2021.129086

Yi, C., Li, Y., Zhu, H., Liu, Y., & Quan, K. (2021). Effect of Lactobacillus plantarum fermentation on the volatile flavors of mung beans. *LWT*, *146*, 111434. https://doi.org/10.1016/J.LWT.2021.111434

Zhang, C., Guo, J., Zhang, Z., Tian, S., Liu, Z., & Shen, C. (2021). Biochemical components and fungal community dynamics during the flowering process of Moringa-Fu brick tea, a novel microbially fermented blended tea. *LWT*, *140*, 110822. https://doi.org/10.1016/j.lwt.2020.110822

Zhang, K., Cheng, J., Hong, Q., Dong, W., Chen, X., Wu, G., & Zhang, Z. (2022). Identification of changes in the volatile compounds of robusta coffee beans during drying based on HS-SPME/GC-MS and E-nose analyses with the aid of chemometrics. *LWT*, *161*, 113317. https://doi.org/10.1016/J.LWT.2022.113317

Zhang, Y., Zhang, S., Fan, W., Duan, M., Han, Y., & Li, H. (2019). Identification of volatile compounds and odour activity values in quinoa porridge by gas chromatography–mass spectrometry. *Journal of the Science of Food and Agriculture*, *99*(8), 3957–3966. https://doi.org/10.1002/JSFA.9621

Zhong, A., Chen, W., Hu, L., Wu, Z., Xiao, Y., Li, K., Li, Z., Wang, Y., & Wang, C. (2022). Characterisation of key volatile compounds in fermented sour meat after fungi growth inhibition. *LWT*, *165*, 113662. https://doi.org/10.1016/j.lwt.2022.113662

564 **Table captions**

565 **Table 1.** Volatile profile of unfermented white quinoa flour (UFWQ), fermented white quinoa
566 (FWQ) dried at 50, 60, and 70 °C (FWQ-50, FWQ-60, FWQ-70) and lyophilised fermented white
567 quinoa (FWQ-L).

568 **Table 2.** Volatile profile of unfermented black quinoa flour (UFBQ), fermented black quinoa
569 (FBQ) dried at 50, 60, and 70 °C (FBQ-50, FBQ-60, FBQ-70) and lyophilised fermented black
570 quinoa (FBQ-L).

571 **Table 3.** Volatile profile of unfermented Pardina lentil flour (UFPL), fermented Pardina lentil
572 (FPL) dried at 50, 60, and 70 °C (FPL-50, FPL-60, FPL-70) and lyophilised fermented Pardina lentil
573 (FPL-L).

574 **Table 4.** Volatile profile of unfermented Castellana lentil flour (UFCL), fermented Castellana
575 lentil (FCL) dried at 50, 60, and 70 °C (FCL-50, FCL-60, FCL-70) and lyophilised fermented
576 Castellana lentil (FCL-L).

577 **Figure captions**

578 **Figure 1.** Biplot based on principal component analysis (PCA) of the different volatile
579 compounds found in white quinoa (A), black quinoa (B), Pardina lentil (C) and Castellana lentil
580 (D) before and after of fermentation and drying.

581 **TABLE 1.**

NAME	RT	LRI	UFWQ	FWQ	FWQ-50	FWQ-60	FWQ-70	FWQ-L
Free acids								
2-Methylpentanoic acid anhydride	22.13	1655	-	24 ± 4	-	-	-	-
Alcohols								
2-Methyl-1-butanol	10.85	1207	-	31 ± 5^{a}	7.2 ± 0.4^{b}	11 ± 2^{b}	10.0 ± 1.2^{b}	38 ± 4^{a}
1-Hexanol	14.80	1280	77.11 ± 0.73^{b}	36.0 ± 0.8^{e}	54 ± 2^{c}	48.4 ± 1.4^{d}	47.8 ± 0.9^{d}	91 ± 2^{a}
3-Octanol	15.85	1299	-	28 ± 8^{b}	15 ± 2^{b}	-	-	140 ± 4^{a}
1-Octen-3-ol	17.25	1452	2.19 ± 0.01^{e}	196 ± 2^{b}	119 ± 5^{dc}	115 ± 3^{d}	132.9 ± 0.4^{c}	395 ± 14^{a}
1-Heptanol	17.41	1459	1.56 ± 0.01^{e}	42 ± 5^{a}	9 ± 0.5^{d}	7.3 ± 0.2^{d}	21 ± 11^{b}	11 ± 3^{c}
2-Methyl-6-hepten-1-ol	17.60	1466	1.75 ± 0.14	-	-	-	-	-
4-Ethylcyclohexanol	19.50	1543	-	33.55 ± 0.08	-	-	-	-
1-Octanol	19.90	1560	-	51.8 ± 2.5^{a}	9.2 ± 0.3^{c}	8.53 ± 0.16^{c}	8.6 ± 0.4^{c}	22 ± 3^{b}
Benzyl alcohol	26.80	1871	0.61 ± 0.07^{d}	3.23 ± 0.06^{c}	3.6 ± 0.3^{c}	5.0 ± 0.3^{b}	14.5 ± 0.4^{a}	-
Phenylethyl Alcohol	27.50	1905	0.5 ± 0.2^{d}	3.2 ± 0.4^{a}	1.4 ± 0.3^{c}	3.37 ± 0.04^{a}	3.5 ± 0.3^{a}	2.15 ± 0.01^{b}
Aldehydes								
Hexanal	7.22	1169	-	1400 ± 18^{a}	840 ± 60^{b}	832 ± 92^{b}	741 ± 2^{b}	570 ± 47^{c}
Octanal	12.70	1240	-	99.26 ± 1.12^{a}	74 ± 7^{b}	78.61 ± 1.08^{b}	81 ± 2^{b}	21 ± 3^{c}
2-Heptenal	13.70	1259	-	38 ± 6^{a}	-	-	-	27 ± 5^{a}
Nonanal	15.60	1294	-	123 ± 6^{a}	97 ± 9^{b}	115 ± 4^{a}	129 ± 6^{a}	26.4 ± 0.2^{c}
5-Ethylcyclopent-1-enecarboxaldehyde	16.15	1408	-	45.98 ± 0.08^{a}	27 ± 2^{c}	27.6 ± 0.5^{cb}	29.05 ± 0.01^{b}	11.7 ± 0.9^{d}
2-Octenal	16.50	1422	-	156 ± 9^{a}	36 ± 0.3^{c}	34 ± 11^{c}	35.7 ± 1.5^{c}	65 ± 9^{b}
Decanal	18.30	1494	-	17.55 ± 0.18^{a}	14 ± 3^{a}	16 ± 2^{a}	17.6 ± 1.2^{a}	4.6 ± 1.2^{b}
Benzaldehyde	18.80	1514	1.91 ± 0.39^{c}	1221 ± 113^{c}	309 ± 10^{b}	295 ± 7^{b}	308 ± 29^{b}	405 ± 17^{b}
5-methyl-2-furancarboxaldehyde	20.05	1566	-	13.63 ± 0.35^{a}	4.6 ± 0.3^{c}	9.50 ± 1.14^{b}	12 ± 2^{ab}	-
Benzeneacetaldehyde	21.60	1632	-	3 ± 2^{c}	15 ± 3^{b}	25.1 ± 1.8^{a}	25.2 ± 0.3^{a}	6.43 ± 0.07^{c}
2-Decenal	21.73	1638	-	15 ± 2^{a}	-	4.83 ± 1.14^{b}	5.9 ± 0.4^{b}	4.3 ± 0.3^{b}
2-Butyl-2-octenal	22.30	1663	-	12.76 ± 2.8^{a}	6.8 ± 0.4^{b}	6.59 ± 0.04^{b}	6 ± 2^{bc}	1.8 ± 0.4^{c}
2-hydroxy-benzaldehyde	22.40	1667	-	2.9 ± 0.3	-	-	-	-
3-Methoxybenzaldehyde	29.65	>2000	-	345 ± 24^{a}	190 ± 26^{b}	235 ± 2^{b}	231 ± 5^{b}	318 ± 23^{a}
3-Chloro-4-methoxybenzaldehyde	32.78	>2000	-	4.5 ± 0.44^{b}	2.59 ± 0.13^{c}	3.0 ± 0.2^{c}	3.3 ± 0.4^{c}	8.9 ± 0.4^{a}
Ketones								
3-Octen-2-one	16.02	1403	-	65.11 ± 1.19^{a}	26 ± 4^{b}	28.8 ± 1.1^{b}	29.2 ± 0.6^{b}	11.3 ± 1.8^{c}
Methyl-1-cyclopenten-1-yl)-ethanone	20.60	1589	0.28 ± 0.07^{d}	6.32 ± 0.18^{c}	14 ± 2^{b}	18.4 ± 0.3^{a}	20.1 ± 0.7^{a}	1.1 ± 0.1^{d}
2-Undecanone	20.75	1595	-	11 ± 3	-	-	-	-
Acetophenone	21.80	1641	0.36 ± 0.01^{c}	2.39 ± 0.01^{b}	2.93 ± 0.04^{a}	2.7 ± 0.3^{a}	2.5 ± 0.4^{a}	1.2 ± 0.2^{a}
5-(Hydroxymethyl)dihydrofuran-2(3H)-one	23.00	1693	-	7.9 ± 0.2	-	-	-	-
Heterocyclic compounds								
2-Pentylfuran	11.25	1214	6.69 ± 0.31^{e}	123 ± 14^{c}	153 ± 7^{b}	230 ± 13^{a}	244 ± 6^{a}	97 ± 12^{d}
Ethyl 2-(5-methyl-5-vinyltetrahydrofuran-2-yl)propan-2-yl carbonate	17.01	1261	1.54 ± 0.01^{b}	22.34 ± 0.09^{a}	-	-	-	-
2-Propylfuran	18.13	1487	-	4.36 ± 0.05	-	-	-	-
Pyrrole	18.70	1510	1.58 ± 0.18	-	-	-	-	-
2-n-Butyl furan	20.46	1583	-	10.4 ± 0.7^{a}	-	-	-	7.1 ± 1.3^{a}
3-Phenylfuran	26.22	1844	-	42 ± 14^{a}	1.8 ± 0.2^{b}	2.9 ± 0.2^{b}	3.6 ± 0.3^{b}	21 ± 10^{b}
2(3H)-Furanone, dihydro-5-pentyl-	29.78	>2000	-	2.3 ± 0.5^{c}	12 ± 2^{ab}	14.78 ± 0.10^{a}	10.28 ± 0.07^{b}	9.5 ± 0.5^{b}
Phenolic compounds								
Phenol	29.34	1998	0.37 ± 0.02^{d}	2.1 ± 0.2^{c}	3.74 ± 0.01^{b}	4.6 ± 0.2^{a}	4.3 ± 0.5^{ab}	-
Esters								
Ethyl hexanoate	11.46	1218	-	200.06 ± 0.04^{a}	116 ± 2^{c}	140.4 ± 1.7^{bc}	122 ± 10^{c}	175 ± 30^{ab}
Ethyl octanoate	16.75	1432	-	58 ± 8^{a}	36 ± 2^{b}	57 ± 8^{a}	53 ± 2^{a}	35 ± 12^{b}
Ethyl benzoate	22.22	1660	-	-	2.7 ± 0.3^{b}	1.87 ± 0.10^{a}	2.09 ± 0.09^{b}	6.4 ± 0.5^{a}
Methyl 4-methoxybenzoate	30.96	>2000	-	7.6 ± 0.9^{a}	2.5 ± 0.6^{c}	4.80 ± 0.13^{b}	5.4 ± 0.2^{b}	5.5 ± 0.7^{b}
ether								
2-Propylphenol, methyl ether	26.07	1836	-	3.42 ± 0.16^{b}	1.9 ± 0.5^{c}	3.12 ± 0.01^{b}	4.2 ± 0.4^{a}	1.03 ± 0.04^{d}
Total			96 ± 2^{c}	4500 ± 250^{a}	2200 ± 145^{b}	2389 ± 155^{b}	2360 ± 88^{b}	2550 ± 200^{b}

582 The data represent the peak area/100,000. [abc] Lowercase letters indicate significant differences at the 95% ($p < 0.05$) significance level between

583 flours in the same row. RT: retention time. LRI: linear retention indices.

584

TABLE 2.

VOLATILE COMPOUND	RT	LRI	UFBQ	FBQ	FBQ-50	FBQ-60	FBQ-70	FBQ-L
Alcohols								
1-Pentanol	12.21	1232	-	32 ± 3^{a}	18.8 ± 1.8^{bc}	13 ± 4^{c}	-	25 ± 3^{ab}
1-Hexanol	14.80	1280	3.6 ± 0.1^{e}	12.6 ± 1.2^{d}	14.1 ± 0.5^{c}	11.88 ± 0.04^{d}	15.843 ± 0.009^{b}	29 ± 0.3^{a}
3-Octanol	15.85	1299	-	69 ± 17^{b}	25 ± 4^{c}	0.9 ± 0.2^{c}	-	165 ± 3^{a}
1-Octen-3-ol	17.25	1452	-	323 ± 25^{a}	50.5 ± 0.3^{a}	61.6 ± 1.5^{c}	62.6 ± 1.6^{c}	177 ± 3^{b}
1-Heptanol	17.41	1459	-	8.08 ± 0.02	-	-	-	-
1-Octanol	19.90	1560	-	14.1 ± 0.9^{a}	3.98 ± 0.03^{c}	1.9 ± 0.3^{d}	1.5 ± 0.8^{d}	7.8 ± 0.8^{b}
Aldehydes								
Hexanal	7.22	1169	-	724 ± 38^{a}	310 ± 3^{c}	279 ± 8^{c}	311 ± 31^{c}	490 ± 27^{b}
Octanal	12.70	1240	-	24.8 ± 1.5^{c}	40.1 ± 1.5^{b}	24.7 ± 1.0^{c}	40 ± 13^{b}	66 ± 3^{a}
2-Heptenal	13.70	1259	-	18.2 ± 0.8	-	-	-	-
Nonanal	15.60	1294	-	23 ± 4^{c}	30 ± 2^{b}	29.8 ± 0.6^{b}	38 ± 3^{a}	29 ± 3^{b}
5-Ethylcyclopent-1-enecarboxaldehyde	16.15	1408	-	17.1 ± 0.4^{ab}	13.2 ± 0.3^{d}	14.5 ± 1.0^{cd}	16 ± 0.06^{bc}	19 ± 2^{a}
2-Octenal, (E)-	16.50	1422	-	71 ± 3	-	-	-	-
Benzaldehyde	18.80	1514	3.9 ± 0.6^{c}	437 ± 22^{a}	130 ± 3^{b}	194 ± 23^{b}	187 ± 6.9^{b}	406 ± 77^{a}
5-methyl-2-furancarboxaldehyde	20.05	1566	-	16.1 ± 0.8^{a}	12 ± 3^{b}	11.0 ± 0.2^{b}	10.7 ± 0.3^{b}	17.2 ± 1.3^{a}
Benzeneacetaldehyde	21.60	1632	5 ± 5^{d}	13.7 ± 1.2^{c}	28 ± 2^{a}	25.7 ± 0.2^{ab}	29.7 ± 1.2^{a}	20.4 ± 0.6^{b}
2,3-dihydro-1H-indene-4-carbaldehyde	27.80	1921	-	-	0.658 ± 0.002^{a}	0.41 ± 0.02^{b}	0.34 ± 0.02^{c}	-
3-Methoxybenzaldehyde	29.65	>2000	2.3 ± 0.7^{d}	112 ± 6^{b}	90 ± 4^{c}	98 ± 4^{bc}	86 ± 4^{c}	202 ± 16^{a}
3-Chloro-4-methoxybenzaldehyde	32.78	>2000	-	2.7 ± 0.2^{b}	1.7 ± 0.2^{c}	1.94 ± 0.06^{c}	2.1 ± 0.3^{c}	3.4 ± 0.3^{a}
Ketones								
3-Octanone	11.85	1225	-	238 ± 63^{a}	-	-	-	122 ± 4^{a}
1-Hepten-3-one	13.25	1251	-	13 ± 3	-	-	-	-
	14.00	1264	-	10.4 ± 0.3	-	-	-	-
3-Octen-2-one	16.02	1403	-	34 ± 3^{c}	38.6 ± 1.2^{b}	31.9 ± 0.3^{c}	31 ± 0.3^{c}	44 ± 3^{a}
Acetophenone	21.80	1641	0.42 ± 0.1^{d}	1.4 ± 0.2^{c}	3.5 ± 0.4^{a}	2.8 ± 0.5^{b}	2.31 ± 0.04^{b}	2.37 ± 0.09^{b}
Heterocyclic compounds								
2-Pentylfuran	11.25	1214	-	75 ± 4^{c}	248 ± 17^{a}	223 ± 4^{a}	165 ± 32^{b}	93 ± 4^{c}
Furfural	17.39	1458	3.4 ± 0.2^{d}	7.5 ± 0.4^{ab}	8.81 ± 0.02^{a}	6.6 ± 0.9^{abc}	4.9 ± 1.7^{bcd}	4.2 ± 1.2^{cd}
Naphthalene	23.70	1725	2.9 ± 0.9	-	-	-	-	-
Phenolic compounds								
Phenol	29.34	1998	0.83 ± 0.02^{b}	1.14 ± 0.08^{b}	3.4 ± 0.4^{a}	3.2 ± 0.2^{a}	3.1 ± 0.4^{a}	1.2 ± 0.1^{b}
Esters								
Ethyl octanoate	16.75	1432	-	-	7.8 ± 0.4^{a}	2.3 ± 0.3^{c}	5.0 ± 0.2^{b}	-
Methyl 4-methoxybenzoate	30.96	>2000	-	1.06 ± 0.06^{a}	0.99 ± 0.09^{a}	0.9 ± 0.2^{a}	0.86 ± 0.15^{a}	1.5 ± 0.2^{a}
Others								
4-Isothiocyanate-1-butene	17.15	1448	7.6 ± 1.1	-	-	-	-	-
.beta.-Bisabolene	23.55	1719	-	-	2.9 ± 0.2^{a}	2.2 ± 0.2^{b}	2.3 ± 0.1^{b}	-
Total			32 ± 9^{c}	2310 ± 200^{a}	1080 ± 45^{b}	1040 ± 50^{b}	1020 ± 97^{b}	1970 ± 150^{a}

The data represent the peak area/100,000. ^{abc} Lowercase letters indicate significant differences at the 95% ($p < 0.05$) significance level between flours in the same row. RT: retention time. LRI: linear retention indices.

TABLE 3.

NAME	RT	LRI	UFPL	FPL	FPL-50	FPL-60	FPL-70	FPL-L
Alcohols								
1-Hexanol	14.80	1207	29 ± 2^{a}	-	1.9 ± 0.6^{c}	-	-	6 ± 2^{b}
3-Octanol	15.85	1299	-	98 ± 14^{a}	31 ± 3^{c}	35.6 ± 1.8^{bc}	49 ± 12^{c}	59 ± 4^{b}
1-Octen-3-ol	17.25	1452	-	353 ± 27^{a}	83 ± 2^{c}	102 ± 5^{c}	108 ± 21^{c}	207 ± 8^{b}
1-Heptanol	17.41	1459	2.5 ± 0.4	-	-	-	-	-
2-Ethyl-1-hexanol	18.25	1492	1.4 ± 0.1	-	-	-	-	-
Benzyl alcohol	26.80	1871	1.51 ± 0.02^{a}	2.14 ± 0.17^{a}	1.01 ± 0.03^{a}	-	-	2.1 ± 0.4^{a}
Phenylethyl Alcohol	27.50	1905	0.9 ± 0.1^{a}	0.47 ± 0.06^{b}		-	-	0.5 ± 0.1^{ab}
Aldehydes								
Hexanal	7.22	1169	132 ± 11^{a}	10 ± 5^{b}	17 ± 3^{b}	14 ± 2^{b}	15 ± 2^{b}	-
3,5,5-trimethyl-1-hexanal	18.10	1485	-	27 ± 1^{a}	-	-	-	22 ± 3^{a}
Benzaldehyde	18.80	1514	6.9 ± 0.6^{d}	405 ± 6^{b}	583 ± 8^{a}	129 ± 6^{c}	444 ± 47^{b}	473 ± 50^{b}
Benzeneacetaldehyde	21.60	1632	1.5 ± 0.3^{c}	39 ± 3^{a}	24.7 ± 1.5^{b}	25 ± 3^{b}	36 ± 4^{b}	29 ± 2^{b}
2,3-dihydro-1H-indene-4-carbaldehyde	27.80	1921	-	0.62 ± 0.04	-	-	-	-
3-Methoxybenzaldehyde	29.65	>2000	-	2.7 ± 0.2^{b}	1.7 ± 0.1^{b}	1.1 ± 0.1^{b}	1.8 ± 0.2^{b}	8.0 ± 1.5^{a}
3-Chloro-4-methoxybenzaldehyde	32.78	>2000	-	3.5 ± 0.3^{a}	4.4 ± 0.3^{a}	3.13 ± 0.06^{a}	4.6 ± 0.3^{a}	3.1 ± 1.4^{a}
Ketones								
3-Octanone	11.85	1225	-	172 ± 28^{a}	20 ± 7^{b}	34.1 ± 0.9^{b}	16 ± 6^{b}	43 ± 5^{b}
1-Hepten-3-one	13.25	1251	-	18 ± 1^{a}	-	-	-	11.3 ± 0.2^{b}
Acetophenone	21.80	1641	-	4.1 ± 0.8^{b}	2.5 ± 0.2^{b}	3.3 ± 0.2^{b}	3.28 ± 0.02^{b}	3 ± 1^{a}
Heterocyclic compounds								
3-Ethyl-2,5-dimethylpyrazine	17.06	1445	-	-	3.20 ± 0.06^{c}	8.7 ± 0.7^{b}	5.5 ± 0.8^{a}	-
3-Phenylfuran	26.22	1844	-	0.99 ± 0.03^{b}	0.85 ± 0.08^{b}	1.29 ± 0.06^{a}	1.54 ± 0.07^{a}	0.79 ± 0.09^{b}
Phenolic compounds								
Phenol	29.34	1998	0.44 ± 0.06^{d}	0.85 ± 0.01^{ab}	0.73 ± 0.02^{bc}	0.6 ± 0.1^{cd}	1.05 ± 0.15^{a}	0.684 ± 0.001^{ab}
Total			199 ± 18^{d}	1137 ± 87^{a}	780 ± 28^{b}	362 ± 18^{c}	685 ± 93^{b}	880 ± 36^{b}

The data represent the peak area/100,000. ^{abc} Lowercase letters indicate significant differences at the 95% ($p < 0.05$) significance level between flours in the same row. RT: retention time. LRI: linear retention indices.

TABLE 4.

NAME	RT	LRI	UFCL	FCL	FCL-50	FCL-60	FCL-70	FCL-L
Alcohols								
1-Hexanol	14.80	1280	70.3 ± 0.4^{a}	28 ± 2^{b}	8.6 ± 0.5^{d}	5.6 ± 0.4^{e}	5.4 ± 0.5^{e}	17.7 ± 0.2^{c}
3-Octanol	15.85	1299	-	99 ± 15^{a}	-	-	-	14 ± 5^{b}
1-Octen-3-ol	17.25	1452	3.1 ± 0.3^{d}	302 ± 27^{b}	30 ± 2^{c}	45.4 ± 0.6^{c}	37.5 ± 1.5^{c}	460.6 ± 1.4^{a}
2-Ethyl-1-hexanol	18.25	1492	1.6 ± 0.2	-	-	-	-	-
1-Octanol	19.90	1560	-	6.8 ± 0.2^{a}	1.8 ± 0.2^{c}	-	-	3.9 ± 0.2^{b}
Cyclooctyl alcohol	21.25	1617	-	7.3 ± 0.6^{a}	-	-	-	4.24 ± 0.08^{b}
1-Nonanol	22.25	1661	1.2 ± 0.1	-	-	-	-	-
Benzyl alcohol	26.80	1871	4.7 ± 0.7^{a}	0.57 ± 0.02^{d}	0.9 ± 0.2^{cd}	1.5 ± 0.1^{c}	3.8 ± 0.2^{b}	1.6 ± 0.2^{c}
Aldehydes								
Hexanal	7.22	1169	38 ± 3^{b}	131 ± 11^{a}	48 ± 3^{b}	16 ± 3^{c}	-	45.9 ± 0.2^{b}
Nonanal	15.60	1294	-	10.7 ± 0.8^{a}	5.1 ± 0.2^{b}	-	-	8.9 ± 0.8^{a}
5-Ethylcyclopent-1-enecarboxaldehyde	16.15	1408	-	11.5 ± 0.6^{a}	4.1 ± 0.2^{b}	-	-	-
2-Octenal	16.50	1422	-	16 ± 2^{a}	17.8 ± 0.8^{a}	-	-	-
Decanal	18.30	1494	-	6.9 ± 1.4	-	-	-	-
Benzaldehyde	18.80	1514	4.5 ± 0.2^{e}	250 ± 19^{b}	91 ± 0.7^{d}	118 ± 2^{d}	149.1 ± 0.7^{c}	530 ± 22^{a}
2-Nonenal, (E)-	19.20	1531	-	3.8 ± 0.3	-	-	-	-
5-methyl-2-furancarboxaldehyde	20.05	1566	4.5 ± 1.6^{ab}	5.4 ± 0.2^{a}	4.1 ± 0.1^{abc}	3.4 ± 0.2^{bc}	3.81 ± 0.02^{abc}	2.4 ± 0.8^{c}
Benzeneacetaldehyde	21.60	1632	-	27 ± 2^{b}	45.7 ± 1.6^{a}	49.5 ± 0.4^{a}	46.8 ± 1.9^{a}	26.9 ± 0.4^{b}
3-Ethylbenzaldehyde	23.00	1694	-	1.39 ± 0.06^{a}	0.78 ± 0.07^{b}	-	-	0.95 ± 0.01^{b}
2-Phenylpropenal	25.10	1790	-	2.02 ± 0.09	-	-	-	-
Benzeneacetaldehyde, .alpha.-ethylidene-	27.80	1921	-	0.61 ± 0.01^{c}	1.9 ± 0.2^{a}	1.24 ± 0.01^{b}	1.2 ± 0.2^{b}	0.963 ± 0.002^{b}
3-Methoxybenzaldehyde	29.65	>2000	-	698 ± 20^{b}	145 ± 10^{d}	260 ± 11^{c}	260 ± 6^{c}	838 ± 19^{a}
3-Chloro-4-methoxybenzaldehyde	32.78	>2000	-	28.3 ± 0.4^{b}	5.7 ± 0.5^{e}	15.9 ± 0.9^{d}	17.6 ± 0.8^{c}	31.7 ± 0.03^{a}
Ketones								
1-Hepten-3-one	13.25	1251	-	14 ± 3^{a}	-	-	-	13 ± 2^{a}
Acetophenone	21.80	1641	-	1.7 ± 0.5^{b}	5.5 ± 0.5^{a}	5.9 ± 0.3^{a}	5.6 ± 0.3^{a}	1.5 ± 0.2^{b}
Heterocyclic compounds								
Furfurylmethylamphetamine	11.00	1210	2.1 ± 0.2^{c}	9 ± 2^{b}	-	-	-	24.8 ± 0.5^{a}
2-Pentylfuran	11.25	1214	-	-	87.3 ± 0.02^{a}	20 ± 5^{b}	14 ± 4^{b}	-
2,6-Dimethylpyrazine	14.02	1265	-	-	16.4 ± 1.9^{a}	8.9 ± 0.4^{b}	13.6 ± 0.6^{a}	-
3-Ethyl-2,5-dimethylpyrazine	17.06	1445	-	-	5.3 ± 0.4^{a}	3.8 ± 0.2^{b}	4.988 ± 0.001^{a}	-
Furfural	17.39	1458	27 ± 9^{a}	21 ± 4^{ab}	13 ± 3^{bc}	11.5 ± 1.7^{bc}	11.6 ± 0.02^{bc}	10 ± 2^{c}
2-Ethyl-5-methylpyrazine	18.10	1486	-	16 ± 0.4^{a}	4.4 ± 0.7^{c}	6.1 ± 0.2^{b}	6.9 ± 0.3^{b}	-
2-n-Butyl furan	20.46	1583	-	1.11 ± 0.05	-	-	-	-
3-Phenylfuran	26.22	1844	-	-	2.14 ± 0.09^{ab}	1.8 ± 0.2^{b}	2.6 ± 0.3^{a}	0.63 ± 0.08^{c}
Phenolic compounds								
Phenol	29.34	1998	1.2 ± 0.2^{cd}	1.00 ± 0.01^{d}	2.9 ± 0.1^{c}	2.22 ± 0.14^{b}	1.4 ± 0.1^{c}	2.8 ± 0.3^{a}
Esters								
Ethyl 4-(ethyloxy)-2-oxobut-3-enoate	13.85	1262	-	2.5 ± 0.3^{a}	2.1 ± 0.2^{a}	-	-	-
Methyl benzoate	21.17	1614	-	1.03 ± 0.07	-	-	-	-
Methyl 4-methoxybenzoate	30.96	>2000	-	2.38 ± 0.01^{a}	-	-	-	2.3 ± 0.2^{a}
Others								
Methane-d, trichloro-	5.58	1154	-	28 ± 5^{a}	-	-	-	-
Total			160 ± 15^{d}	1730 ± 120^{b}	554 ± 28^{c}	585 ± 27^{c}	592 ± 18^{c}	2052 ± 56^{a}

The data represent the peak area/100,000. ^{abc} Lowercase letters indicate significant differences at the 95% ($p < 0.05$) significance level between flours in the same row. RT: retention time. LRI: linear retention indices.

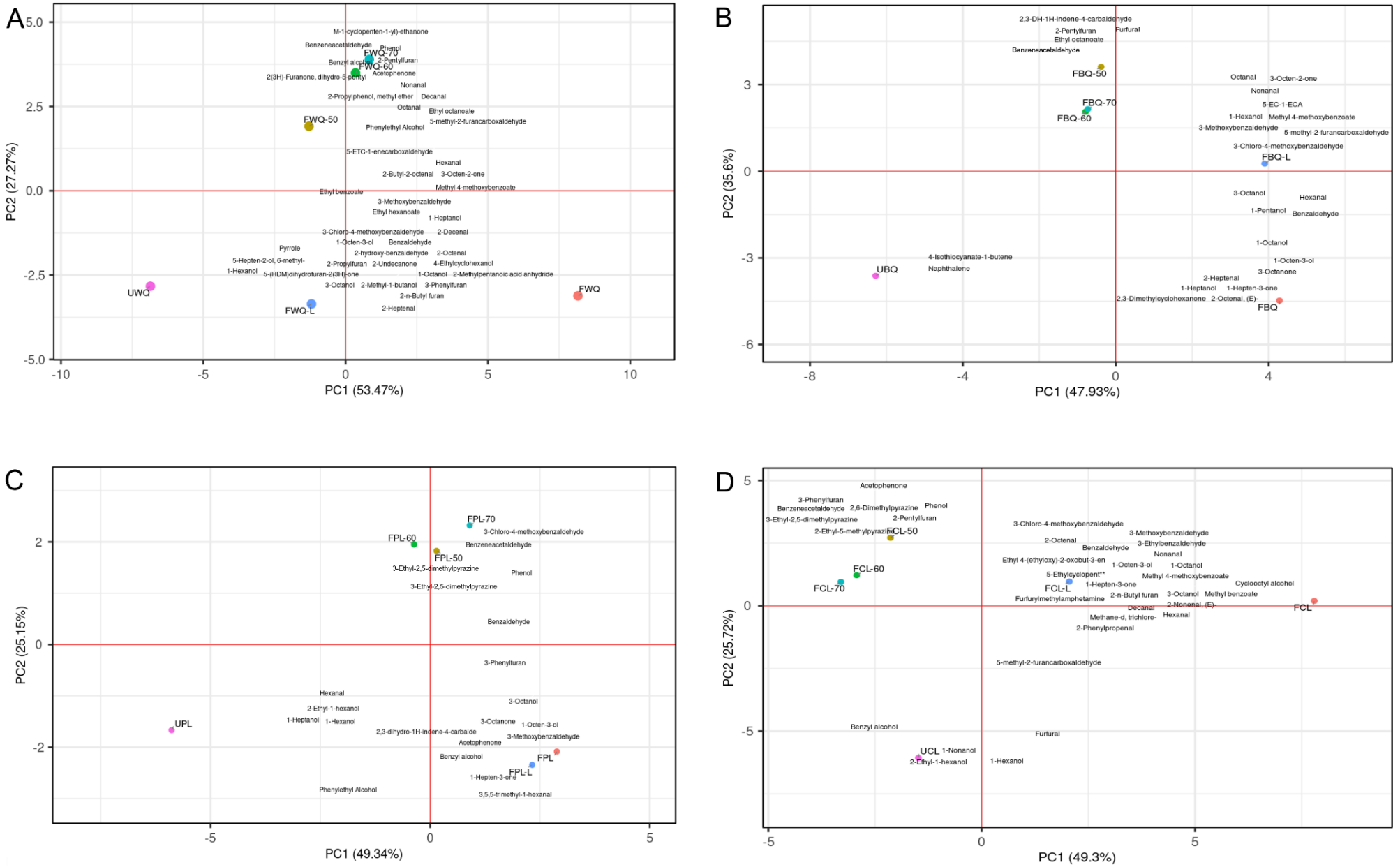
A
PC2 (27.27%)
PC1 (53.47%)
M-1-cyclopenten-1-yl)-ethanone
Benzeneacetaldehyde
Phenol
FWQ-70
2-Pentylfuran
Benzyl alcohol
FWQ-60
2(3H)-Furanone, dihydro-5-pentyl-
Acetophenone
Nonanal
2-Propylphenol, methyl ether
Decanal
Octanal
Ethyl octanoate
FWQ-50
5-methyl-2-furancarboxaldehyde
Phenylethyl Alcohol
5-ETC-1-enecarboxaldehyde
Hexanal
2-Butyl-2-octenal
3-Octen-2-one
Ethyl benzoate
Methyl 4-methoxybenzoate
3-Methoxybenzaldehyde
Ethyl hexanoate
1-Heptanol
3-Chloro-4-methoxybenzaldehyde
2-Decenal
1-Octen-3-ol
Benzaldehyde
Pyrrole
2-hydroxy-benzaldehyde
2-Octenal
5-Hepten-2-ol, 6-methyl-
2-Propylfuran
2-Undecanone
4-Ethylcyclohexanol
1-Hexanol
5-(HDM)dihydrofuran-2(3H)-one
1-Octanol
2-Methylpentanoic acid anhydride
3-Octanol
2-Methyl-1-butanol
3-Phenylfuran
UWQ
2-n-Butyl furan
FWQ
FWQ-L
2-Heptenal
B
PC2 (35.6%)
PC1 (47.93%)
2,3-DH-1H-indene-4-carbaldehyde
2-Pentylfuran
Furfural
Ethyl octanoate
Benzeneacetaldehyde
FBQ-50
Octanal
3-Octen-2-one
Nonanal
FBQ-70
5-EC-1-ECA
FBQ-60
1-Hexanol
Methyl 4-methoxybenzoate
3-Methoxybenzaldehyde
5-methyl-2-furancarboxaldehyde
3-Chloro-4-methoxybenzaldehyde
FBQ-L
3-Octanol
Hexanal
1-Pentanol
Benzaldehyde
1-Octanol
4-Isothiocyanate-1-butene
1-Octen-3-ol
Naphthalene
UBQ
3-Octanone
2-Heptenal
1-Heptanol
1-Hepten-3-one
2,3-Dimethylcyclohexanone
2-Octenal, (E)-
FBQ
C
PC2 (25.15%)
PC1 (49.34%)
FPL-70
3-Chloro-4-methoxybenzaldehyde
FPL-60
Benzeneacetaldehyde
FPL-50
3-Ethyl-2,5-dimethylpyrazine
Phenol
3-Ethyl-2,5-dimethylpyrazine
Benzaldehyde
3-Phenylfuran
Hexanal
3-Octanol
2-Ethyl-1-hexanol
UPL
1-Heptanol
1-Hexanol
3-Octanone
1-Octen-3-ol
2,3-dihydro-1H-indene-4-carbalde
3-Methoxybenzaldehyde
Acetophenone
Benzyl alcohol
FPL
FPL-L
1-Hepten-3-one
Phenylethyl Alcohol
3,5,5-trimethyl-1-hexanal
D
PC2 (25.72%)
PC1 (49.3%)
Acetophenone
3-Phenylfuran
Benzeneacetaldehyde
2,6-Dimethylpyrazine
Phenol
3-Ethyl-2,5-dimethylpyrazine
2-Pentylfuran
2-Ethyl-5-methylpyrazine
FCL-50
3-Chloro-4-methoxybenzaldehyde
3-Methoxybenzaldehyde
2-Octenal
3-Ethylbenzaldehyde
Benzaldehyde
Nonanal
Ethyl 4-(ethyloxy)-2-oxobut-3-en
1-Octen-3-ol
1-Octanol
FCL-60
5-Ethylcyclopent**
Methyl 4-methoxybenzoate
FCL-70
1-Hepten-3-one
FCL-L
Cyclooctyl alcohol
2-n-Butyl furan
3-Octanol
Methyl benzoate
Furfurylmethylamphetamine
Decanal
2-Nonenal, (E)-
FCL
Methane-d, trichloro-
Hexanal
2-Phenylpropenal
5-methyl-2-furancarboxaldehyde
Benzyl alcohol
Furfural
UCL
1-Nonanol
2-Ethyl-1-hexanol
1-Hexanol

FIGURE 1.

598