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Accelerated storage of ground coffee: Merging of analytical techniques to assess sensitivity to oxygen and moisture exposure

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ABSTRACT

This study investigates the effectiveness of analytical techniques—electronic nose (e-nose), headspace (HS) analysis by solid phase micro extraction coupled with gas chromatography–mass spectrometry (HS-SPME-GC–MS), and Fourier-transform near-infrared (FT-NIR) spectroscopy—in detecting quality changes in ground coffee exposed to accelerated conditions, including elevated oxygen levels, relative humidity, and temperature. The goal is to assess how these techniques could be complementary to sensory analysis to provide a comprehensive evaluation of coffee aging and quality deterioration. Sensory analysis revealed a decline in attributes such as body, odor intensity, aftertaste, and aroma intensity over time and at higher temperatures, while oxidative notes and astringency increased. Bitterness and acidity were also intensified under accelerated aging. HS-SPME-GC–MS analysis showed a reduction in volatile compounds during storage, with high temperatures accelerating this loss. *E*-nose data reflected a rapid decline in sensor signals for aromatic and sulphur-containing compounds, indicating significant aroma degradation. FT-NIR analysis identified variations in moisture, carbohydrate, and lipid content between fresh and aged samples. Principal component analysis (PCA) of combined sensory and instrumental data revealed that fresh samples were associated with higher volatile levels and more favourable sensory characteristics, whereas aged samples exhibited greater bitterness, acidity, and oxidation. Mid-level data fusion confirmed consistent trends across techniques, enhancing the interpretation of quality changes. These findings underscore the complementarity of instrumental and sensory methods to monitor the aging process of ground coffee and ensure product quality over time.

1. Introduction

Coffee is one of the most widely consumed beverages globally, appreciated for its rich and peculiar aroma. In 2022 counted over 10 million tons (International Coffee Organization, 2024), and its market value was estimated at USD 41.80 billion (Custom Market Insight, 2024). Due to its significant economic value, preserving coffee's unique and highly prized flavour throughout the entire value chain is crucial, with particular emphasis on the post-roasting operations. One of the most detrimental defects affecting coffee aroma -and consequently quality- is staling, an overall deterioration primarily driven by lipid oxidation, volatilization and hydrolysis over time. This process becomes especially critical during secondary shelf life, resulting in rancid and unpleasant aroma (Anese, Manzocco, & Nicoli, 2006; Toci, Neto, Torres, & Farah, 2013). Numerous studies have investigated the staling of ground coffee, aiming to identify the main factors that affect this phenomenon, such as temperature, humidity, and oxygen exposure (Anese et al., 2006; Benković & Tušek, 2018; Smrke, Adam, Mühlemann, Lantz, & Yeretzian, 2022). Temperature and moisture are particularly influential during secondary shelf life (Orfanou, Dermesonlouoglou, & Taoukis, 2019), as they can enhance the oxidative degradation caused by oxygen.

Sensory analysis is widely employed as a key tool for identifying defects resulting from coffee oxidation, particularly in the evaluation of both primary (Barrera-López et al., 2022; Guerra, Lagazio, Manzocco, Barnabà, & Cappuccio, 2008; Rosillo, Ríos, Huatangari, & Lalangui,

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2023) and secondary shelf-life (Anese et al., 2006; Benković & Tušek, 2018; Makri, Tsimogiannis, Dermesonluoglu, & Taoukisa, 2011; Smrke et al., 2022). Although sensory techniques are considered highly reliable, the coffee industry increasingly requires faster and more objective analytical methods to support decision-making processes, especially when validating new packaging solutions or monitoring product quality under accelerated or extreme conditions.

Given that staling also leads to changes in the aromatic profile, numerous studies have been conducted to identify chemical markers capable of monitoring the aging process by correlating them with sensory degradation. Among the available techniques, gas chromatography coupled with mass spectrometry (GC–MS), typically applied through headspace sampling, is the most widely used. This method is a powerful tool for the identification and quantification of volatile organic compounds (VOCs) in coffee. By examining the VOC fraction, it is possible to monitor changes in aroma compounds and gain valuable insights into processing dynamics (Galarza & Figueroa, 2022; Ruosi et al., 2012) and product quality (Bressanello et al., 2017). Moreover, GC–MS can be employed to identify specific markers associated with oxidized coffee (Strocchi et al., 2022) and to investigate the effects of storage conditions on both aromatic and sensory profiles (Bröhan, Huybrighs, Wouters, & Van der Bruggen, 2009; Caporaso, Whitworth, Cui, & Fisk, 2018; Cincotta, Tripodi, Merlino, Verzera, & Condurso, 2020; Cotter & Hopfer, 2018; Mahmud, Shellie, & Keast, 2020; Sun et al., 2020). The use of HS-GC–MS in conjunction with electronic sensing methods has become common practice in the study of aroma profiles for identifying oxidative reactions in coffee products (Cui et al., 2020; Dong et al., 2019). *E*-nose (electronic nose) devices consist of arrays of chemical sensors capable of detecting classes of volatile compounds and are widely used in food sciences and shelf-life studies (Benedetti et al., 2005; Grassi et al., 2023; Limbo, Sinelli, Torri, & Riva, 2009; Torri, Sinelli, & Limbo, 2010). Although this technique has already been applied to detect oxidation in various food products (Cosio, Ballabio, Benedetti, & Gigliotti, 2007; Mildner-Szkudlarz, Zawirska-Wojtasiak, Korczak, & Jeleń, 2007), to date no studies have reported its application in assessing staling effects in coffee.

With growing interest over time, NIR spectroscopy has been recognized as a rapid and non-destructive technique, useful for evaluating the quality and shelf-life of food products (Grassi et al., 2023; Limbo et al., 2009; Torri et al., 2010). Specifically, the use of NIR spectroscopy in coffee quality assessment enables real-time monitoring of molecular changes, allowing the evaluation of parameters such as moisture content (Alessandrini, Romani, Pinnavaia, & Rosa, 2008; Tugnolo et al., 2021), antioxidant compounds (Páscoa, Magalhães, & Lopes, 2013; Shan, Suzuki, Suhandy, Ogawa, & Kondo, 2014) and the effects of storage conditions and shelf-life stability (Lázaro, Ferreira, Gomes Neto, & Ferreira, 2022). However, to date, no studies have reported the use of Fourier Transform (FT)-NIR spectroscopy to investigate the staling of ground coffee under stressful storage conditions-namely high oxygen levels, elevated relative humidity, and increased temperatures.

The analytical techniques (i.e., HS-SPME-GC–MS, e-nose, and NIR spectroscopy) are typically employed individually to investigate specific aspects of food quality. Only a limited number of studies have explored the complementary use for a more comprehensive assessment of food degradation processes (Borràs et al., 2015). In the field of coffee research, most efforts have focused on combining sensory analysis with aroma profiling to assess freshness and characterize the aromatic attributes of oxidized coffee (Calligaris, Manzocco, Anese, & Nicoli, 2016; Manzocco, Calligaris, Anese, & Nicoli, 2016; Strocchi et al., 2022; Yeretzian, Blank, & Wyser, 2017).

In this context, the aim of this study was to evaluate the potential of sensory analysis, HS-SPME-GC–MS, e-nose, and FT-NIR spectroscopy-used individually and in combination- for characterizing the changes occurring in ground coffee under stressful environmental conditions (i.e., high levels of oxygen, relative humidity, and storage temperature). In particular, an innovative approach is proposed that integrates the outputs of the three instrumental techniques through mid-level data fusion, a strategy increasingly recognized for its effectiveness in food quality and shelf-life assessment (Borràs et al., 2015; Smolinska, Engel, Szymanska, Buydens, & Blanchet, 2019). This approach involves the extraction of relevant features from each analytical data block separately, followed by the concatenation of these features into a single matrix for further processing (Azcarate, Ríos-Reina, Amigo, & Goicoechea, 2021). Mid-level data fusion offers several advantages, including data dimensionality reduction, noise filtering, and improved result interpretation (Borràs et al., 2015). Its effectiveness has been demonstrated in diverse areas of food analysis and shelf-life control (Biancolillo, Boqué, Cocchi, & Marini, 2019; Malegori et al., 2020; Silvestri et al., 2014), particularly for integrating data from e-sensing, spectroscopy, and mass spectrometric techniques (Calvini & Pigani, 2022; Li, Xiong, & Min, 2019; Massaro et al., 2021).

2. Materials and methods

2.1. Experimental plan and sample preparation

A single lot of roasted and ground coffee (100 % *Coffea Arabica*) for moka preparation was used, kindly supplied by Lavazza Group (Lavazza, Turin, Italy). The coffee used for this test was a 100 % Arabic coffee with a light roasting which emphasizes the acidity and sweetness of the blend, giving rise to floral notes. In details, 80 vacuum-sealed soft-packs (250 g each) in aluminum barrier material (PP/Alu/PE) were opened on the first day of the experiment and stored in air (O_2, 20.9 %) at 25 °C, 37 °C, and 45 °C with a 55 ± 5 % Relative Humidity (RH%) and 45 °C with a 45 ± 5 % Relative Humidity (RH%) for a maximum of 42 days, into temperature and relative humidity controlled incubators incubators (FOC 225I, Velp Scientifica, Italy). Every two weeks, for the samples stored at 25 °C, or one week, for the samples stored at higher temperatures, three opened packages were analysed, collecting 50 g of coffee on the upper part of each pack. Ground coffee in equal amounts from each of the three packages was pooled and used for triplicate analyses. Moisture content (MC), water activity (a_w), HS-SPME-GC–MS, electronic nose (e-nose), and FT-NIR analyses were carried out on each coffee powder sample, while sensory analysis was performed on the brewed coffee, prepared as described in §1.2.

2.2. Sensory analysis

For the sensory evaluation, a Quantitative Descriptive Analysis (QDA) was conducted by 8 trained sensory panelists (International Organization for Standardization, 2021). For the brewed coffee preparation, 17 g of powder coffee (pooled as described in §1.1) from each experimental condition were used along with 160 mL of mineral water (Levissima, Sanpellegrino S.p.A., Nestlè S.A., Switzerland) were placed into a 3 cup-size Italian moka pot. The brewed coffee samples were served immediately to the panel for the sensory analysis, and eight attributes (i.e., Odor Intensity, Oxidation, Aroma Intensity, Acidity, Bitterness, Astringency, Body, and Aftertaste) (Table 1) were evaluated using an anchored 11-point rating scale from “none” (0) to “very high” (10), with 1.0-point increments. The “oxidation” attribute was defined as the intensity of the smell/aroma associated with rancid notes, walnut oil, peanut shells, old, dried fruit, or “old” coffee, perceived by the olfactory system either directly or indirectly. When barely perceptible, it was rated 1.5 on a 0–10 scale; when clearly perceptible, it was rated 3 on the same scale. Two sessions for each storage condition were conducted.

2.3. Moisture content and water activity

Coffee MC (expressed as g_{H20}/100 g of product) was determined by a gravimetric method drying the samples (4.00 ± 0.5 g) in a thermostatic oven (Thermostatic Oven M710, Fratelli Galli, Milan, Italy) at 105 ± 1 °C until constant weight. Water activity (a_w) at 25 °C was determined

Table 1
Sensory attributes and definition for the coffee lexicon.

Sensory attribute	Definition
Odor Intensity	*Intensity of the odors and aromas of the brewed coffee perceived orthonasally.*
Oxidation	*A sweet but unlovely flavour and aroma of roasted coffee which reflects the oxidization of many of the pleasant volatiles and the loss of others (* Buffo & Cardelli-Freire, 2004*)*
Aroma Intensity	*Intensity of the odors and aromas of the brewed coffee perceived retronasally.*
Acidity	A key taste sensation linked to organic acids like malic or citric linked to the basic taste sensation of sourness
Bitterness	Distinctive taste that reminds bitter ingredients like hops in beer.
Astringency	Tactile sensation often characterized by a coarse, dry feeling in the mouth akin to the effects of tannins in red wines
Body	Viscosity of the of liquid on the palate, especially as felt between the tongue and palate
Aftertaste	The persistency of pleasant flavors and aromas after coffee is swallowed; a short or an unpleasant persistency is not appreciated (Lingle, 2011)

using a water activity meter (LabMaster, Novasina, Lachen, Switzerland) after calibration with standard saturated salt solutions (LabMaster, Novasina, Lachen, Switzerland) at a_w values of 0.06–0.11-0.33-0.53-0.75-0.97 and 0.97. Both the analyses were performed in triplicate.

2.4. Volatile profile of ground coffee by solid phase-microextraction (SPME) and GC–MS

The volatile profile of ground coffee was analysed using headspace solid-phase microextraction coupled with gas chromatography–mass spectrometry (HS-SPME-GC–MS) to identify volatile organic compounds (VOCs).

For each sampling point and storage temperature, 2 g of ground coffee (pooled as described in § 1.1) were placed into 20 mL glass vials sealed with magnetic screw caps and silicone/PTFE septa (VWR International, USA). An internal standard (20 μL of n-toluene, 1 mg/mL; Sigma-Aldrich, USA) was added to each vial, to check the quality of the fibers. At time zero, the coffee was sampled before packaging and analysed in triplicate. At each subsequent sampling point, for each temperature, three replicates—each corresponding to an independent sample—were prepared and analysed under each storage condition.

Samples were incubated at 45 °C for 5 min, followed by VOC extraction from the headspace at the same temperature for 25 min using a pre-conditioned Divinylbenzene/Carboxen/polydimethylsiloxane (CAR/PDMS/DVB) fiber (50/30 μm × 1 cm, 23 Ga; Supelco, Bellefonte, PA, USA). Volatile compounds were thermally desorbed in the GC injector for 5 min in split mode (injection temperature: 250 °C; split flow: 10 mL/min; split ratio: 10) by MultiPurpose Sampler (Gerstel GmbH & Co.KG, Germany). To evaluate carryover and peaks from the fiber, blank samples were run on a regular basis. HS-SPME analysis was performed using a GC system (TRACE 1300, Thermo Fisher Scientific, Waltham, USA) equipped with a mass spectrometer (TSQ 8000 EVO, Thermo Fisher Scientific, Waltham, USA).

Chromatographic separation was achieved using a WAX MS capillary column (MEGA, Italy), 30 m in length, 0.25 mm in internal diameter, and with a film thickness of 0.25 μm. The GC oven temperature program was as follows: initial hold at 40 °C for 5 min; increase to 180 °C, at 3 °C/min; then to 240 °C, at 8 °C/min, with a final hold of 2 min. The carrier gas was helium at a constant flow of 1 mL/min. The MS ion source was maintained at 200 °C, the GC–MS interface at 240 °C. Mass spectra were acquired using electron ionization (EI) at 70 eV in the *m/z* range of 40–350, by collecting data at a rate of 1 scan s^{-1}.

The identification of MS fragmentation patterns was performed either by comparison with those of pure compounds or using the National Institute of Standards and Technology (NIST) Mass Spectral Search 2.2 Library. Mass spectral Match Factor (similarity >900) was also used to decide whether a peak was correctly identified or not. Compounds were identified by comparing the retention times of the chromatographic peaks with those of authentic compounds analysed under the same conditions when available. Additionally, linear retention indices (LRIs) were determined by analysing a homologous series of n-alkanes (C8–C20, 40 mg/L each; Sigma-Aldrich) under identical chromatographic conditions with a 20-min extraction time. The LRIs were then validated against literature values for confirmation. Volatile compound measurements from each headspace of coffee extracts were carried out by peak area (expressed in arbitrary units) of each recognized compound and by peak area normalization to the GC–MS total peak area (expressed in percentage).

The repeatability of the method and the consistent performance of HS-SPME were assessed by monitoring the peak area of the internal standard (IS). The coefficient of *v*ariation (CV) of the IS remained ≤8.8 % throughout the study. Retention time (RT) stability was evaluated using n-alkanes as references, with the CV of their RT values being ≤0.01 %.

2.5. E-nose

E-nose analysis was performed using a commercial e-nose (PEN3; Win Muster Airsense Analytics Inc., Schwerin, Germany) combined with the HTA2010 autosampler. The e-nose was composed of 10 metal oxide semi-conductor (MOS) chemical sensors whose selectivity is reported in Buratti et al. (2015). Aliquots of 0.50 g (±0.05 g) were collected from the ground coffee sampled at each storage time and placed in 20 mL airtight *v*ials. After 1 h at room temperature and 5 min at 30 ± 1 °C, the measurement started. The volatile compounds were pumped over the sensor surfaces at a flow rate of 300 mL/min for 60 s; the sensor signals were taken after 50 s of sampling time. After sample analysis, the sensors were purged for 300 s with filtered air and then, before the next sample injection, the sensor baselines were re-established for 5 s.

2.6. FT-NIR spectroscopy

For FT-NIR spectroscopy analysis, a MPA spectrometer (Bruker Optics, Milan, Italy) equipped with an integrating sphere was used. Aliquots of 1 g (±0.5 g) were collected from the ground coffee sampled at each storage time and put in 10 mL glass vials. Spectra were acquired with 8 cm^{-1} resolution, 32 scans for both background and samples, in a wavenumber range of 9000–3800 cm^{-1}. OPUS software (*v*. 7.5 Bruker Optics, Milan, Italy) was used to control the instrument and collect the spectra.

2.7. Statistical data analysis

Analysis of Variance (ANOVA) was performed using SPSS Win 12.0 version 28 statistical software (SPSS Inc. IBM Corp., Chicago, IL) on MC, a_w, and QDA results considering time or temperature as factors. Before ANOVA, the homogeneity of variance using Levene's Test was evaluated. For dataset with homogeneous variance, ANOVA was followed by the Tukey's HSD post-hoc test ($p \leq 0.05$). In case of inhomogeneous variance, the Welch ANOVA was used, followed by the Games Howell post-hoc test ($p \leq 0.05$).

Principal Component Analysis (PCA) was performed to highlight sample similarity and the relationship between the variables by using the Origin Pro software (v. 2021b, OriginLab Corp., Northampton, MA) on individual datasets from HS-SPME-GC–MS, FT-NIR, and QDA results. In addition, PCA was performed after data merging of sensory data with the results obtained from each technique (i.e., e-nose/sensory, HS-SPME-GC–MS/sensory, and FT-NIR/sensory) with appropriate data pretreatment applied to each dataset. Finally, using a middle level data fusion approach, a global PCA was conducted combining the PC1 and PC2 scores obtained from the PCA of each dataset.

3. Results and discussion

3.1. Sensory analysis of brewed coffee

Mean values of the sensory attributes considered in QDA of brewed coffee samples are reported in Table 2. As the sample set presented inhomogeneity of variance, the Welch ANOVA followed by the Games Howell post-hoc test ($p \leq 0.05$) was applied.

At each temperature, storage time resulted always significant ($p \leq 0.05$) in affecting all the attributes. Odor intensity, aroma intensity, body, and aftertaste decreased over time, as already reported by Orfanou et al. (2019), while oxidation, acidity, bitterness, and astringency increased. For samples aged under accelerated conditions, panelists used the words "aggressive" and "unpleasant" to describe aftertaste. Thus, in these cases (i.e., storage temperatures of 37 and 45 °C), the sensory evaluations stopped at 21 days due to high scores of negative and unpleasant attributes, including oxidation and bitterness.

At the same storage time (i.e., 14 or 21 days), increasing the temperatures significantly ($p \leq 0.05$) influenced all sensory attributes except for the body. *Specifically, odor intensity, aroma intensity, and aftertaste decreased, while oxidation, acidity, bitterness, and astringency increased.*

The distribution of the samples based on storage temperature and time is illustrated in the PCA score plot derived from the sensory dataset (Fig. 1a). Samples are distributed along PC1 according to both the storage temperature and duration. As expected, samples stored at higher temperatures are characterized by elevated values of oxidation, acidity, and astringency attributes (Fig. 1b). In contrast, samples at time 0 exhibit higher values for body, odor intensity, and aftertaste, which progressively decline during storage, especially under accelerated conditions (i.e., 37 °C and 45 °C). Fresh samples (time 0) and those stored for up to 14 days at 25 °C are primarily grouped at PC1 values above 1, clearly separated from all the other samples. This distribution is consistent with their low oxidation sensory score —an attribute also employed by coffee producers to define the end of shelf-life, using a sensory threshold of 3–4 on a 11-points scale. Specifically, the "oxidation" attribute is described by panelists as a "rancid flavour" when a score of 4 is reached. As shown in Table 2, the sensory oxidation threshold was already exceeded within the first week under accelerated conditions, while at 25 °C it was surpassed between the second and the third week of conditioning.

The literature does not provide a single explanation for the sensory perception of oxidation; rather, it is considered a multifactorial phenomenon. According to Borém et al. (2019), the perception of off-flavors and unpleasant aromas is mainly due to aldehydes such as hexanal, formed through lipid oxidation and hydrolysis of triacylglycerols. Other authors (Anese et al., 2006; Marin, Požrl, Zlatić, & Plestenjak, 2008; Strocchi et al., 2022) attribute oxidation perception as the general loss of volatiles responsible for the characteristic coffee aroma. Moreover, Kreuml, Majchrzak, Ploederl, and Koenig (2013) reported that the chlorogenic acids degradation may directly contribute to increased oxidation and astringency. Bitterness and astringency have been associated with phenolic acids which can oxidize over time and generate bitter compounds (Drewnowski, 2001). These effects may also result from the hydrolysis of chlorogenic acid lactones, (Lin, Tello, Simons, & Peterson, 2022; Ribeiro, Ferreira, & Salva, 2011) particularly under high moisture conditions (Toci et al., 2013). The increasing trend in bitterness at 25 °C aligns with the findings of Ross, Pecka, and Weller (2006) and may be linked to the loss of volatile compounds that otherwise mask bitterness (Leino, Kaitaranta, & Kallio, 1992). Interestingly, under accelerated storage conditions, bitterness showed a slight decline over time. This observation partially contradicts previous literature and suggests that the rising presence of oxidation-related volatiles may obscure the perception of bitterness. In our study, we observed that high levels of oxidation (above 3 on a 0–10 scale) tend to disrupt the overall balance of sensory attributes, significantly altering the product's profile. Consequently, a reduction in bitterness may be a consequence of advanced oxidation, which hinders the full appreciation of other sensory characteristics. Nevertheless, further studies should be performed for confirmation. At room temperature, acidity decreased significantly during storage, whereas at elevated temperatures it increased. Manzocco et al. (2016) proposed that this rise in acidity may result from the hydrolysis of quinic acid esters and lactones.

3.2. Volatile profile of ground coffee during storage

Studying the composition and evolution of the volatile profile is essential for assessing the freshness and quality of ground coffee. In this study, changes in volatile organic compounds in ground coffee subjected to elevated oxygen (i.e. 20.9 %) and moisture levels were analysed using HS-SPME-GC–MS. A total of 77 compounds were identified and are listed in Table S1, along with their calculated LRI values, aroma descriptions, and, for many compounds, their relative odor thresholds in water, as reported in various relevant studies (Chen, Wu, Wang, Sun, & Chen, 2025; Van Gemert, 2011; Yuan et al., 2025; Zhai et al., 2022; Zhou et al., 2025).

The total peak area of the identified volatile compounds is shown in Fig. 2, alongside the aroma intensity scores obtained through sensory analysis up to QDA completion (i.e., until day 21 for the tests carried out at 37 and 45 °C). Consistent with the observed decline in aroma intensity, a general decrease in the total volatile fraction was detected over the storage period, particularly at elevated storage temperatures. This trend aligns with findings from previous studies (Anese et al., 2006; Buffo & Cardelli-Freire, 2004; Leino et al., 1992; Marin et al., 2008; Ribeiro et al., 2011). At all tested temperatures, a significant reduction ($p \leq 0.05$) in the total volatile compounds' area was observed after 35 days of storage. In general, this reduction is primarily attributed to the volatilization of the compounds following package opening, a process that is further accelerated at higher temperatures.

Table 2
Mean and standard deviation values of the sensory attributes evaluated on brewed coffee samples (0–10 scale). Superscript lowercase letters refer to differences (Games-Howell test; $p \leq 0.05$) between samples collected at different storage time at the same temperature; superscript uppercase letters refer to differences (Games Howell test; $p \leq 0.05$) between samples collected at the same storage time (14 and 21 days) for the different temperatures.

Temperature	Days	Odor Intensity	Oxidation	Aroma Intensity	Acidity	Bitterness	Astringency	Body	Aftertaste
25 °C	0	6.8 ± 0.9^{a}	0.1 ± 0.3^{a}	6.8 ± 1.6^{a}	3.3 ± 0.4^{a}	0.5 ± 0.5^{a}	0.1 ± 0.3^{a}	6.6 ± 1.4^{a}	6.8 ± 0.9^{a}
	14	5.6 ± 1.7^{bA}	2.6 ± 0.6^{bA}	$5.8 \pm 1.5^{ab\ A}$	3.1 ± 0.7^{abA}	1.6 ± 0.6^{bA}	$0.4 \pm 0.5^{a\ A}$	5.7 ± 1.8^{abA}	5.8 ± 0.91^{abA}
	21	5.6 ± 0.5^{bcA}	3.8 ± 1.1^{cA}	$5.6 \pm 1.6^{ab\ A}$	2.5 ± 0.5^{bcA}	2.6 ± 1.0^{cA}	$1.1 \pm 0.6^{b\ A}$	5.6 ± 0.9^{abA}	5.8 ± 1.0^{abA}
	28	5.5 ± 0.8^{bc}	4.5 ± 0.6^{cd}	5.5 ± 1.2^{ab}	2.1 ± 0.4^{cd}	3.0 ± 0.6^{cd}	1.5 ± 0.5^{b}	5.5 ± 0.9^{ab}	5.8 ± 1.5^{ab}
	35	5.0 ± 1.1^{bc}	5.5 ± 1.4^{de}	5.1 ± 0.9^{b}	1.5 ± 0.7^{d}	3.5 ± 0.9^{d}	2.5 ± 0.7^{c}	5.0 ± 1.2^{ab}	5.0 ± 1.6^{b}
	42	4.5 ± 0.8^{c}	5.8 ± 1.4^{e}	5.0 ± 1.1^{b}	1.8 ± 0.8^{d}	3.5 ± 0.9^{d}	2.5 ± 0.8^{c}	5.3 ± 1.4^{b}	5.8 ± 1.2^{ab}
37 °C	7	5.0 ± 0.7^{b}	4.6 ± 0.6^{b}	5.1 ± 0.4^{b}	3.0 ± 0.5^{a}	3.3 ± 1.3^{b}	0.8 ± 0.5^{b}	5.0 ± 0.6^{b}	5.0 ± 0.8^{b}
	14	5.1 ± 0.7^{bB}	$5.5 \pm 0.6^{c\ B}$	3.7 ± 0.60^{cB}	3.6 ± 0.6^{abA}	$2.3 \pm 0.6^{c\ B}$	0.6 ± 0.5^{bA}	5.1 ± 0.9^{bA}	$5.1 \pm 1.1^{b\ A}$
	21	5.1 ± 0.9^{bB}	$8.4 \pm 1.3^{d\ B}$	4.1 ± 1.4^{bcB}	4.2 ± 1.8^{bB}	1.9 ± 0.5^{cB}	2.3 ± 0.9^{cB}	4.6 ± 1.3^{bA}	6.0 ± 1.6^{abA}
45 °C	7	4.5 ± 1.2^{b}	7.0 ± 0.5^{b}	4.5 ± 0.7^{b}	4.6 ± 1.1^{b}	4.5 ± 1.0^{b}	1.5 ± 0.5^{b}	4.5 ± 1.2^{b}	5.1 ± 1.1^{b}
	14	3.6 ± 0.5^{cB}	7.9 ± 0.3^{cC}	4.5 ± 0.6^{bB}	4.6 ± 0.9^{bB}	2.6 ± 0.5^{cB}	2.4 ± 0.7^{cB}	4.8 ± 0.7^{bA}	5.1 ± 0.9^{bA}
	21	3.8 ± 0.9^{bcB}	9.1 ± 0.5^{dB}	3.8 ± 0.7^{bB}	5.3 ± 1.1^{bB}	2.6 ± 0.5^{cA}	3.7 ± 0.9^{dC}	4.5 ± 1.6^{bA}	4.5 ± 1.7^{bB}

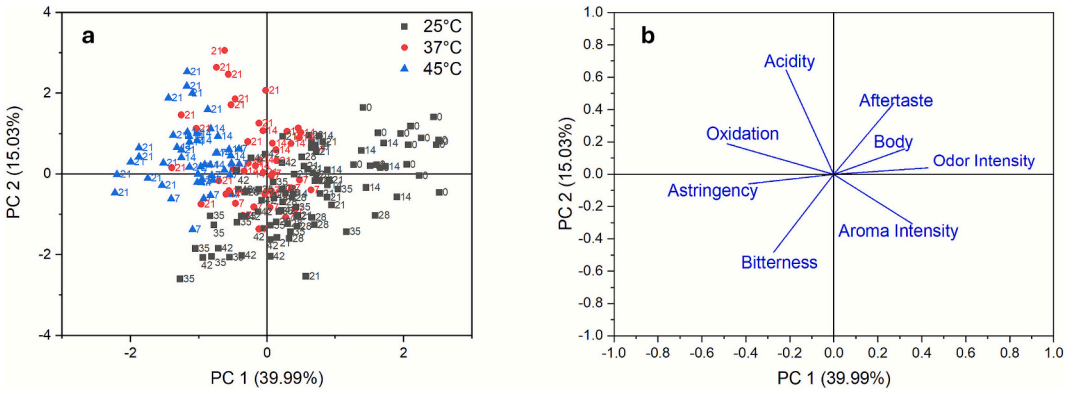

Fig. 1. Principal component analysis score plot (**a**) and loading plot (**b**) for the sensory analysis dataset of brewed coffee prepared from samples stored at different temperatures up to 42 days.

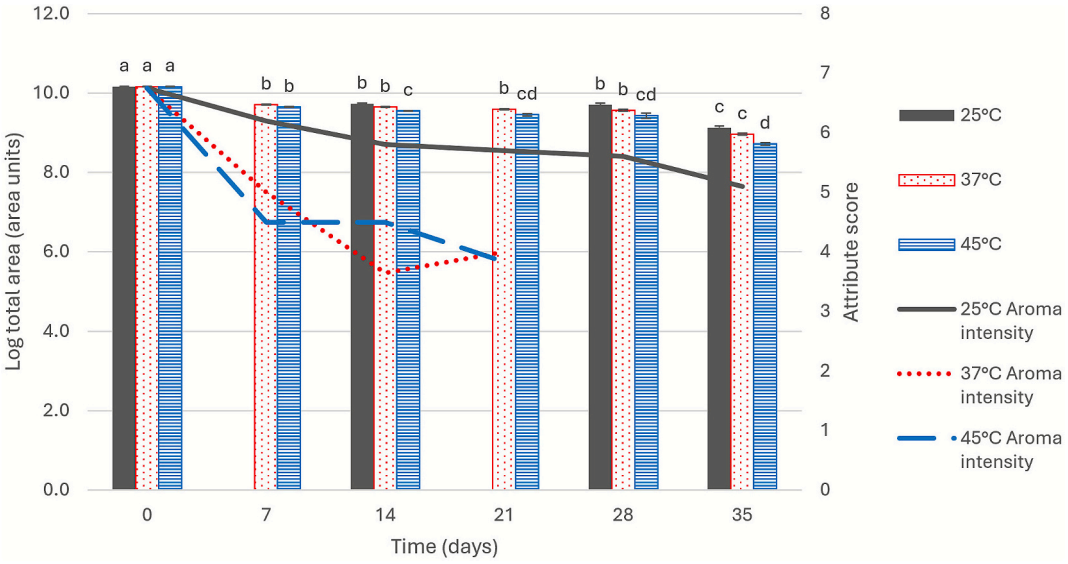

Fig. 2. Volatile organic compounds' total area of ground coffee (bars) and sensory aroma intensity values of brewed coffee (lines), at different storage times and temperatures. Letters refer to differences (F Welsch test; $p \leq 0.05$) between samples collected at different storage time at the same temperature.

The detected compounds can be classified into eight categories based on their chemical properties (Table S1): furans, phenols, pyrazines, pyrroles, ketones and aldehydes, sulphur compounds, alcohols, acids, and cycloalkanes. Variations of the total area of the different compound categories are reported in Table S2.

According to the results of a two-way ANOVA conducted on the HS-SPME-GC–MS results (Table S2), storage time had a statistically significant effect on all classes of volatile compounds. Temperature also showed a significative impact on specific compound classes, namely aldehydes, ketones, acids, phenols, furans, alcohols, pyrazines and pyrroles. Notably, the total content of sulphur-containing compounds decreased significantly ($p < 0.05$, Tukey HSD test) within 28 days of storage at all tested temperatures. Similar findings were reported by Kallio, Leino, Koullias, Kallio, and Kaitaranta (1990), where coffee stored in air-permeable packages at room temperature exhibited a two-thirds reduction in sulphur-containing compounds. As observed for the sulphur compounds, all other classes of volatiles showed a general decline in total peak area over time. Among these, pyrazines and pyrroles-known markers of coffee aroma- are produced during roasting through Maillard reaction involving amino acids and reducing sugars. Their degradation over time is commonly associated with a loss of aroma intensity (Angeloni et al., 2021). However, certain volatile compounds, like aldehydes, ketones and alcohols, are well-documented markers of coffee aging and quality deterioration (Amstalden & Leite, 2001; Anese et al., 2006; Cappuccio, Full, Lonzarich, & Savonitti, 2001; Makri et al., 2011; Marin et al., 2008; Orfanou et al., 2019; Smrke et al., 2022). Their concentrations may increase during storage due to the oxidative degradation of lipids and fatty acids in the presence of oxygen (Frankel, 2005).

To better highlight the changes in the volatile profiles of ground coffee under stressful storage exposure, a PCA was performed using 33 compounds previously identified in the literature as markers of coffee freshness and staling (Calligaris et al., 2016; Manzocco et al., 2016; Strocchi et al., 2022) (Fig. 3). These compounds are highlighted in Table S1 and were autoscaled before PCA. As shown in the score plot of PC1 (77.31 % of explained variance) vs PC2 (16.97 % of explained variance) (Fig. 3a), the samples clustered into three main groups based on storage time. Fresh samples (analysed before oxygen and moisture exposure, t0) were in the quadrant with positive PC1 and PC2 values. This region was primarily associated with higher levels of aldehydes and ketones, as well as key aroma-contributing classes such as pyrazines, furans, and pyrroles. Samples stored for 7, 14, 21, and 28 days exhibited negative PC2 values, and their PC1 scores decreased progressively with increasing storage temperature. Negative values of PC2 could be mainly attributed to alcohol, pyrroles, acids, and phenols -compounds often linked to early-stage staling. The decline in PC1 reflected a reduction in

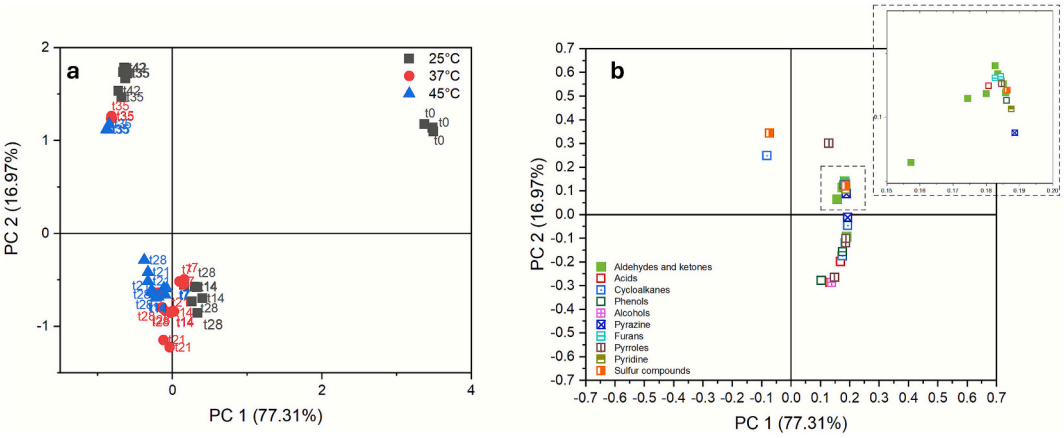

Fig. 3. Principal component analysis score plot (**a**) and loading plot (**b**) for the headspace gas chromatography–mass spectrometry analysis dataset of ground coffee samples stored at different temperatures up to 42 days.

freshness-related volatiles. Samples stored for 35 and 42 days were in the quadrant characterized by negative PC1 and positive PC2 values. Area of the score plot with negative values of PC1 and positive values of PC2. This region was predominantly associated with sulphur-containing compounds and cycloalkanes, both of which are indicative of advanced degradation and off-flavour development. Therefore, the PCA clearly differentiates the evolution of the volatile profile across storage times and conditions, confirming a progressive transition from freshness markers to staling and degradation-related compounds.

Among the compounds included in the PCA, a subset of seven volatile compounds recognized in literature as oxidation markers was further selected to better elucidate the chemical changes occurring during storage (Amstalden & Leite, 2001; Marin et al., 2008; Smrke et al., 2022; Strocchi et al., 2022). Given the overall decrease in the total volatile fraction over time (Fig. 2), the relative concentrations of these selected compounds are expressed as percentage loss over the storage period (Fig. 4). All selected markers exhibited a statistically significant decrease during storage, with the extent of reduction influenced by temperature. In contrast to the findings of Smrke et al. (2022), who reported an increase in 2-butanone and 2- methyl-furan concentrations during storage, this work observed a consistent decline in these compounds, likely due to volatilization following package opening.

Acetaldehyde and 2,3-butanedione also showed a marked reduction during the first two weeks of storage at 37 °C and 25 °C. However, at 45 °C no statistically significant changes were detected, possibly due to an equilibrium between degradation and release rates at higher temperatures. Regarding hexanal, a distinctive trend was observed: an initial decline at all temperatures was followed by a stabilization phase. Unlike the other compounds analysed, hexanal -known as a secondary oxidation product of polyunsaturated fatty acids such as linoleic acid (Amstalden & Leite, 2001; Toci et al., 2013), and a key contributor to rancid flavour (Makri et al., 2011) - demonstrated a divergent behaviour as temperature increased. This may be attributed to a higher rate of formation exceeding its volatilization, as suggested by Błaszkiewicz et al. (2023) and Cascos et al. (2024). Therefore, the persistence of hexanal in stored ground coffee may be associated with the perception of oxidized defects during sensory evaluations of the coffee brew.

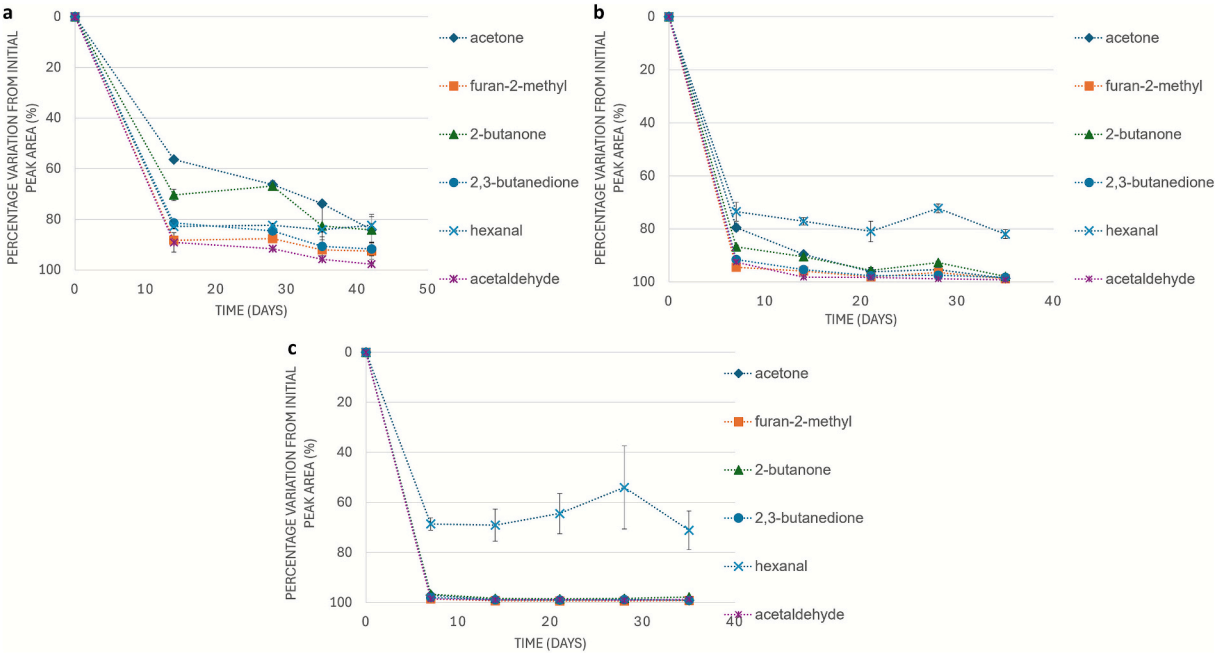

Fig. 4. Variation (%) of peak area from initial value of volatile compounds identified as oxidation and staling markers at different temperatures: 25 °C (**a**), 37 °C (**b**) and 45 °C (**c**).

3.3. E-nose analysis on ground coffee

E-nose was applied to evaluate the evolution of the aromatic profile of ground coffee samples during storage. Since the electronic nose sensors are non-specific or semi-specific (specific for classes of compounds) and the intensity of sensor signals correlates with the volatile component of the sample, in order to depict the aromatic profile of ground coffees, the e-nose sensors were grouped into three categories based on to their known selectivity: WC (W1C, W3C and W5C) specific for alkanes, aromatic compounds, aldehydes and ketones; WW (WW1 and WW2) specific for sulphur-containing compounds; WS (W1S; W2S; W3S; W5S and W6S) with broad range sensitivity (Buratti et al., 2015; Buratti, Benedetti, & Giovanelli, 2017): For each sensor category, the signal intensity was calculated as follows (Fig. 5a, b, c):

$$WC = W1C + W3C + W5C$$

$$WW = W1W + W2W$$

$$WS = W1S + W2S + W3S + W5S + W6S$$

Moreover, to compare the evolution of the aromatic profile of coffee samples stored at the three different temperatures (25, 37, 45 °C), the total signal intensity was calculated summing up the intensity signals of the three sensor categories (WC + WW + WS) (Fig. 5d).

As shown in Fig. 5, at each storage temperature there was a rapid decrease in the intensity signals of WS sensors and WW sensors, showing a significant loss of the aromatic components after a few days of storage. These results are consistent with HS-SPME-GC–MS results indicating a rapid degradation of all classes of volatile compounds with a significant impact on sulphur-containing compounds, pyrazines and pyrroles.

Considering the total signal intensity evolution during storage at the three storage temperatures (Fig. 5d), the decrease of the aromatic profile occurred mainly in the first 14 days of storage. As expected, the decreasing trend up to 35 days of storage was greater at 45 °C.

3.4. FT-NIR spectroscopy on ground coffee

Figure 6a, b shows FT-NIR raw and pre-treated (SNV) spectra of samples stored at 25 °C. The spectra are mainly characterized by absorption bands at 5400–5200 cm^{-1} and 4350–4255 cm^{-1} associated to water content and oily compounds of coffee, respectively. In these regions, fresh samples (t0) seemed to behave differently from the others. However, it is difficult to highlight differences among samples from spectra observation, thus a PCA was applied to SNV-spectra as a fundamental exploratory tool (Fig. 6 c, d).

The PC1 vs PC2 score plot (Fig. 6c), which explains 98.79 % of variance, shows that fresh samples (t00) were well distinguished from the other samples along PC1. With the increase of storage time, samples stored at 25 and 37 °C moved to higher PC1 and PC2 scores, whereas samples stored at 45 °C resulted in increasing values of PC2. Moreover, according to the storage temperatures, a shift of the samples towards the upper left corner of the plot was observed. This aligns with QDA results, as fresh sample are characterized by 0.1 ± 0.3 oxidation scores whereas the sensory attribute rises with storage from 2.6 ± 0.6 after 14 days at 25 °C to 9.1 ± 0.5 after 21 days at 45 °C, time at which the sensory evaluation was stopped. Focusing on the PC1 loading plot (Fig. 6d), two main positive peaks (7000 cm^{-1} and 5200 cm^{-1}) are present, which can be attributed to O—H bonds of water (Grassi et al., 2023). In fact, the water activity of coffee powder increased from 0.22 to 0.47 during storage, and the moisture content rose from 1.08 to 4.93 g/100 g in a few days, reaching the equilibrium with the storage environment hygrometric conditions. Increased moisture uptake alters the physicochemical matrix of roasted coffee, thereby enhancing molecular mobility and reaction kinetics. The trend in water activity was similar to findings reported in other works investigating the secondary shelf-life of coffee (Labuza, 1975; Anese et al., 2006; Makri et al., 2011; Orfanou et al., 2019; Pittia, Nicoli, & Sacchetti, 2007). In the PC2 loading plot (Fig. 7d), the negative peak at 5200 cm^{-1} is also related to water and explains the distribution of the samples stored at 45 °C, for which a slight decrease in water activity was observed, reaching the average value of 0.34. The water loss is linked to the higher vapor tension at 45 °C, also due to a lower RH (45 %) compared to the other storage conditions (55 % UR).

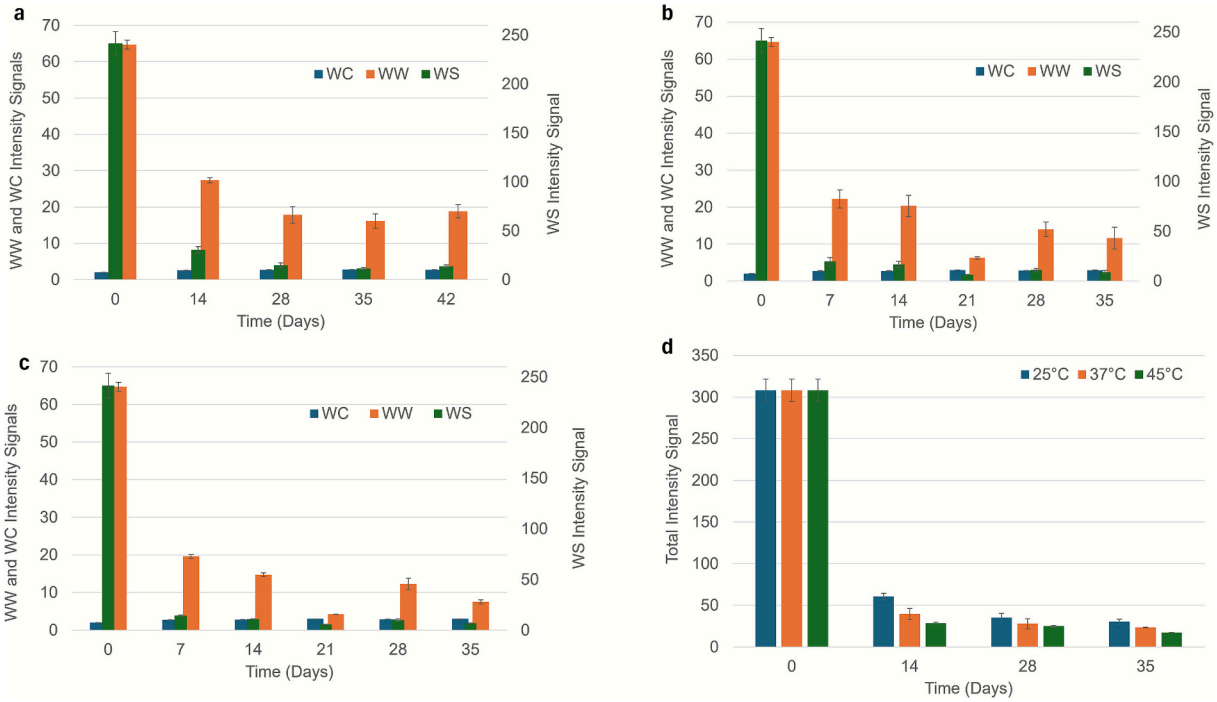

Fig. 5. Signal variation of three e-nose sensor categories at different ground coffee storage temperatures: 25 °C (**a**), 37 °C (**b**) and 45 °C (**c**), total signal variation of e-nose sensors at different ground coffee storage temperatures (**d**).

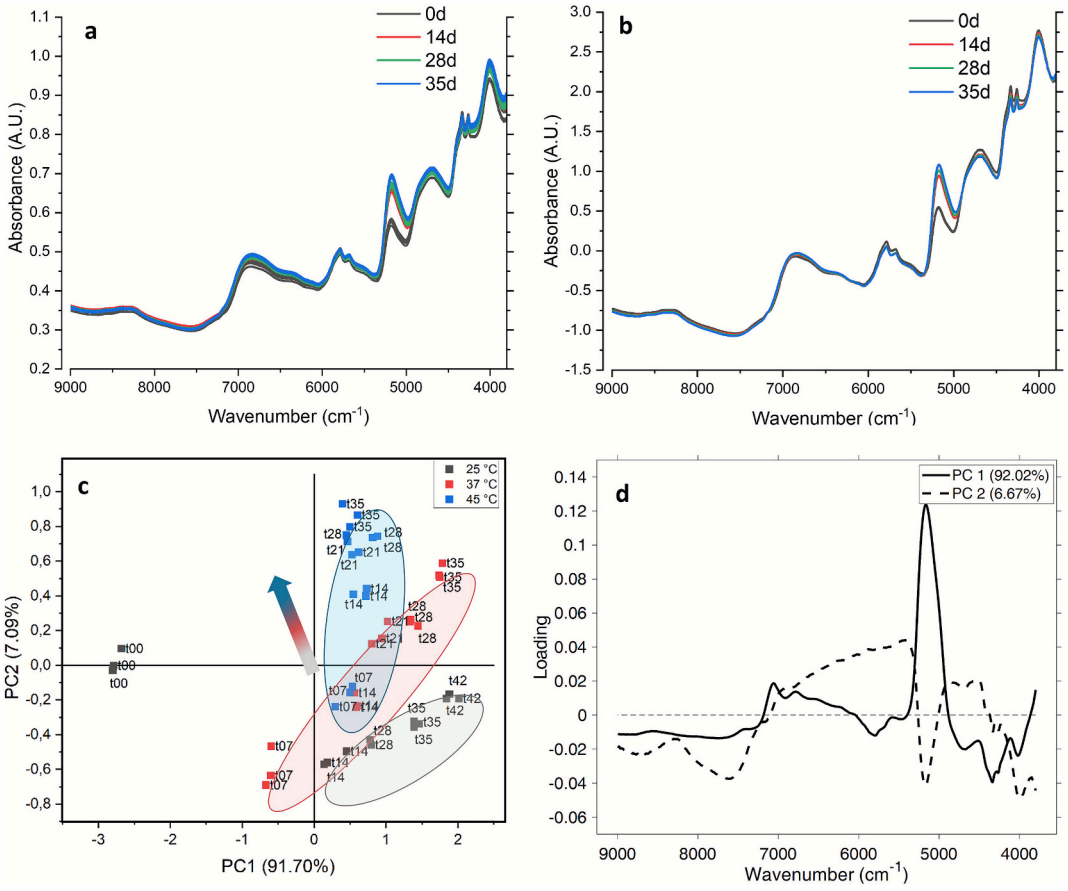

Fig. 6. FT-NIR spectra of ground coffee samples stored at 25 °C coloured according to storage time: raw spectra (**a**); spectra pre-treated with SNV (**b**), Principal component analysis score plot (**c**) and loading plots (**d**) for the SNV-pretreated FT-NIR spectra of ground coffee samples stored at different temperatures up to 42 days.

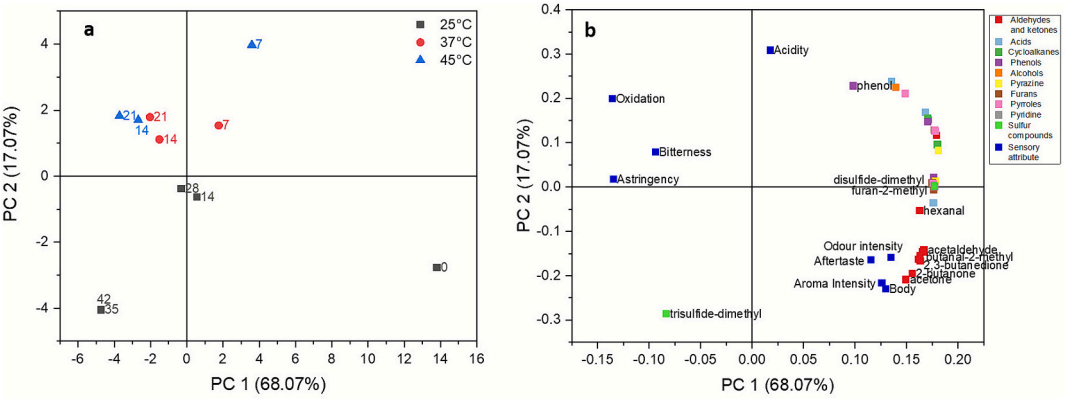

Fig. 7. Principal component analysis score plot (**a**) and loading plots (**b**) of sensory attributes and selected HS-SPME-GC–MS compounds of coffee samples stored at different temperatures up to 42 days.

Furthermore, these changes are manifested also in other regions of the FT-NIR loading plots, indeed that elevated water activity facilitates degradative pathways, such as lipid oxidation and hydrolysis. In the PC1 loading plot, a well-defined negative peak at around 4340 cm^{-1} was also observed, which has been attributed to the stretching of C—H bond (Lázaro et al., 2022; Ribeiro et al., 2011) of fatty acids (Grassi et al., 2023; Lázaro et al., 2022), affected by chemical oxidation and hydrolysis processes during storage (Toci et al., 2013). Moreover, the negative peak at 5700 cm^{-1} (green arrow) is related to CH_2 first overtone of long chain fatty acids (Esteban-Díez, González-Sáiz, & Pizarro, 2004), and it is found in line with the results of Lázaro et al. (2022) where a decrease of signals related to lipid molecules is noticed during the storage of coffee. Thus, the negative loadings at 5700 and 4340 cm^{-1}, characterize samples at time zero before the oxidation of unsaturated fatty acids, thus reflecting the significant ($p < 0.05$, Tukey HSD test) low level of "oxidation" attribute observed in the QDA for these samples compared to stored ones (Table 2). Furthermore, the positive broad band in the PC2 loading plot (7000–5500 cm^{-1}), characterizing aged samples, could be associated with O—H and N—H overtones of chlorogenic acid and amino acids (Ribeiro et al., 2011), which are involved in coffee staling

phenomena and can generate off-flavors (Ribeiro, Augusto, Salva, Thomaziello, and Ferreira (2009), contributing to the panelists' increased perception of the "oxidation" attribute.

3.5. Data fusion

3.5.1. Low-level data fusion: sensory analysis and instrumental techniques

To highlight the relationships between the sensory analysis performed on brewed coffee and the instrumental techniques applied to ground coffee, a low-level data fusion approach was applied. Three datasets combining the values of sensory attributes with the selected HS-SPME-GC–MS compound areas, or the e-nose signals, or the SNV-pretreated FT-NIR spectra were created and subjected to PCA to reveal latent structures that link instrumental signals with sensory perception and to visualize how freshness evolves over storage.

In Fig. 7, the score plot (a) and loading plot (b) of PCA on sensory and selected HS-SPME-GC–MS results are shown in the plane defined by the first two principal components, which explain the 85 % of the total variance. A distribution of samples according to storage time is visible along the PC1, with fresh samples positioned in the positive part of PC1 (Fig. 7A) and characterized by most of the volatile compounds (except for trimethyl disulfide) (Fig. 7B). In fact, as already commented, the total VOC area decreased with storage time. Fresh samples are characterized by higher values of the sensory attributes—odor and aroma intensity, aftertaste, and body—which are closely associated with aldehyde and ketone compounds. Over time, the sensory attributes acidity, bitterness, oxidation and astringency become more prominent; they located at the opposite part of the loading plot with respect to aldehydes and ketones, confirming that the loss in these aromatic compounds make bitterness and oxidation more evident. Furthermore, samples are grouped along PC2 based on storage temperatures (i.e., accelerated conditions vs. room temperature). As noted earlier, trimethyl disulfide is the only compound whose concentration increased with storage time and it is recognized by several authors as an unpleasant flavour for low quality coffee products (Cascos et al., 2024; Cui et al., 2020); actually, in the loading plot this compound is associated with the samples stored for longer times, which were judged with higher values of oxidation, bitterness, and astringency.

Similarly, sensory and e-nose results were merged and submitted to PCA. In Fig. 8, the score plot (a) and loading plot (b) are shown in the plane defined by PC1 and PC2, which explain 73.9 % and 12.7 % of the total variance, respectively. In the score plot (Fig. 8a) coffee samples are distributed along PC1 and PC2 according to storage time and temperature. In the loading plot (Fig. 8b), it can be noticed that WS sensors, with broad-range sensitivity, and WW sensors, sensitive to sulphur-organic compounds, are located at positive values of PC1. These sensors play a key role in discriminating fresh coffee samples (T0 samples), which are characterized by sensory attributes typically associated with coffee freshness (i.e., aroma and odor intensity, body and aftertaste). Stored samples, are characterized by negative PC1 scores (Fig. 8a), related to high intensity of WC sensors, which are sensitive to aromatic and aliphatic compounds, as well as by the sensory attributes -bitterness, astringency, oxidation, and acidity- typically associated with aged samples (Barrera-López et al., 2022; Kreuml et al., 2013).

As for storage temperature, coffee samples stored at 25 °C are grouped in the positive part of PC2 (Fig. 8a) and are characterized by the W3S e-nose sensor and the bitterness attribute. In contrast, coffee samples stored at 37 °C and 45 °C are characterized by increasing negative PC2 scores, associated with highest values of astringency, acidity, and oxidation (Farah, Monteiro, Calado, Franca, & Trugo, 2006; Hii & Borém, 2019).

At last, FT-NIR spectra after SNV pretreatment and sensory results were explored together (Fig. 9). PC1 and PC2 explain 69.8 % and 27.3 % of the total variance, respectively. In the score plot (Fig. 9a), coffee samples are distributed along PC1 according to storage time. The samples analysed before storage (t0) assume a highly positive score, which then decreases with storage time for all the investigated temperatures. This reflects the differences noticed by both PCA on sensory and FT-NIR data independently. Along PC2 it is possible to observe a difference between samples stored at 25 °C and 45 °C. The samples stored at the highest temperature are characterized by positive PC2 scores, whereas samples stored at 25 °C are characterized by negative PC2 scores, as already observed by the two independent PCA. The loading plots are presented separately for PC1 and PC2 to better understand the link among the different variables considered (Fig. 9b and c). The most significant sensory attributes at positive values of PC1 are the odor and aroma intensity, and the body of brewed coffee. Consistently, the FT-NIR regions at 9000–7200 cm^{-1} and 4800–4000 cm^{-1}, corresponding to C—H overtones of fatty acids and N–H/C–H combination bands of amino acids (Munyendo, Njoroge, & Hitzmann, 2021), also exhibited positive PC1 loadings, indicating that these spectral features vary in the same direction as the desirable sensory attributes of fresh coffee. Indeed, lipids contribute to odor intensity and body by acting as carriers for volatile compounds and enhancing brew viscosity, while amino acids serve as precursors in the Maillard reaction and support aroma richness. Thus, higher intensities in these regions are indicative of intact lipid and amino acid profiles in freshly roasted coffee, whereas their degradation during storage is reflected in the loss of odor intensity and body and the emergence of oxidation, bitterness, and astringency. Indeed, samples with the highest negative PC1 scores are instead characterized by higher values of oxidation and astringency, related to the spectral regions 7200–6000 cm^{-1} and 5300–4800 cm^{-1}, associated to water, chlorogenic acid, and organic acids (Munyendo et al., 2021).

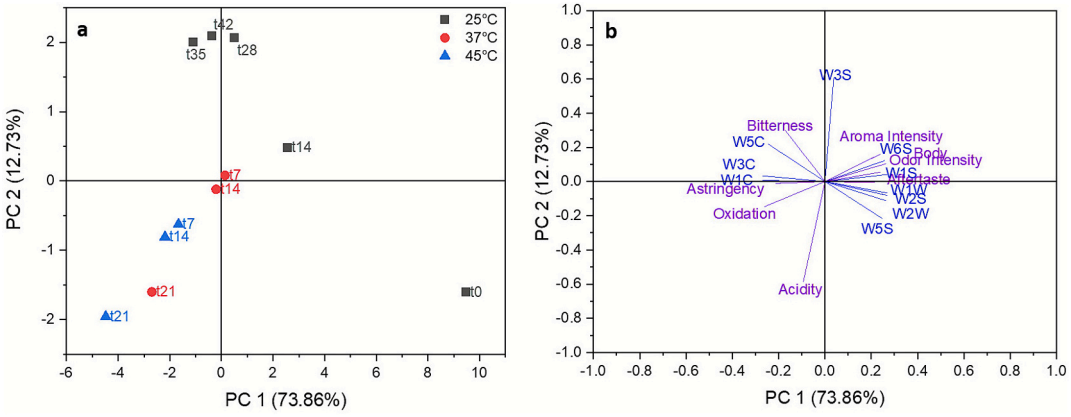

Fig. 8. Principal component analysis score plot (**a**) and loading plots (**b**) of sensory attributes and e-nose data of coffee samples stored at different temperatures up to 42 days.

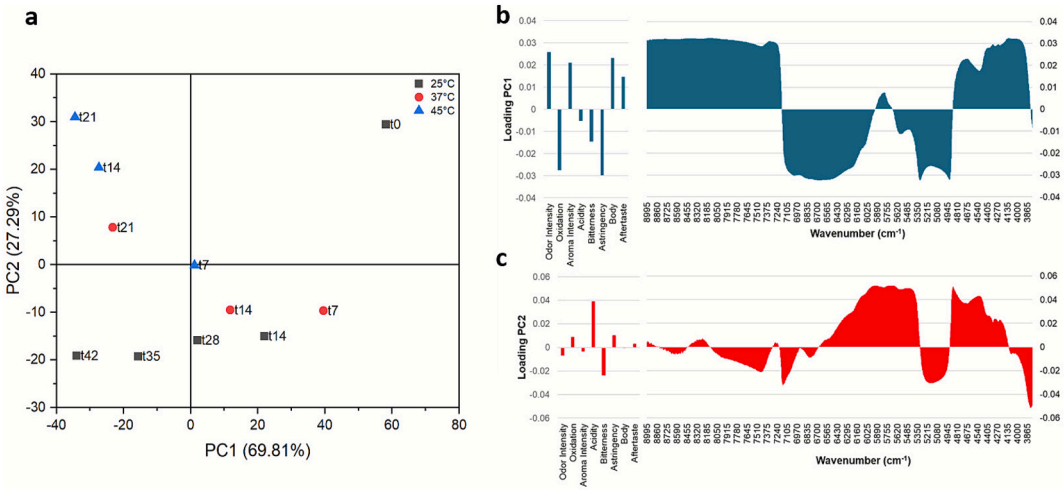

Fig. 9. Principal component analysis score plot (**a**) and loading plots (**b, c**) of sensory attributes and SNV-pretreated FT-NIR spectra of coffee samples stored at different temperatures up to 42 days.

3.6. Mid-level data fusion: instrumental techniques

Mid-level data fusion combines data from all the approaches investigated at an intermediate level, i.e. by considering the scores obtained from the PCA developed for each instrumental analysis. In particular, PC1 and PC2 scores obtained from the PCA applied to e-nose, HS-SPME-GC–MS and FT-NIR data were merged to better understand the quality changes during the secondary shelf life of ground coffee exposed to oxygen and moisture, and to get information concerning the relationships between the different instrumental analyses (Borràs et al., 2015). The mid-level data fusion PCA is presented in Fig. 10. In the resulting score plot (Fig. 10a), the samples are coloured according to the correspondent oxidation values attributed by sensory analysis, while the different storage temperatures are discriminated by different symbols. The score plot confirmed the distributions observed in the PCAs previously analysed. PC1 differentiated samples based on storage time, distinguishing especially fresh samples from the others and highlighting that most of the degradation phenomena occur during the first two week of secondary shelf life of ground coffee. Indeed, fresh samples (coded as 0) assumed high positive PC1 values, and with the increment of storage time samples moved to lower PC1 values. Furthermore, PC2 well described oxidation index changes during storage, with decreasing scores for increasing oxidation levels. As shown by the loading plot (Fig. 10b), the three instrumental techniques are all able to distinguish fresh samples, being their corresponding PC1 located at high positive values of the mid-level PC1. Along PC2, a slightly higher influence of e-nose can be observed, able to distinguish the samples based on oxidation sensory attribute. Importantly, while GC–MS provides detailed chemical fingerprints, it is time-consuming, costly, and not easily adaptable for routine or in-line analyses. In contrast, e-nose and FT-NIR offer rapid, non-destructive, and comparatively less expensive measurements, making them particularly promising for online monitoring of coffee freshness during storage and distribution. Their alignment with the sensory attributes demonstrated in this study highlights their practical potential as efficient alternative tools — whether for screening or monitoring — to be used alongside GC–MS for real-time quality control.

4. Conclusions

In conclusion, this study demonstrates that the quality deterioration of ground coffee under accelerated conditions induced by elevated humidity, oxygen, and temperature—can be effectively monitored through the integration of sensory and instrumental techniques. Sensory analysis revealed that fresh samples are characterized by higher levels of aroma intensity, body, and aftertaste, whereas aged samples exhibit increased bitterness, acidity, and oxidative notes. These sensory changes corresponded closely with analytical findings from HS-SPME-GC–MS and e-nose analyses. HS-SPME-GC–MS identified the depletion of key aroma compounds and the emergence of markers of oxidation, such as trimethyl disulfide and hexanal, which correlated with negative sensory attributes. The e-nose highlighted the role of specific sensors (WC and W3S) in detecting coffee's staling and oxidative degradation. FT-NIR

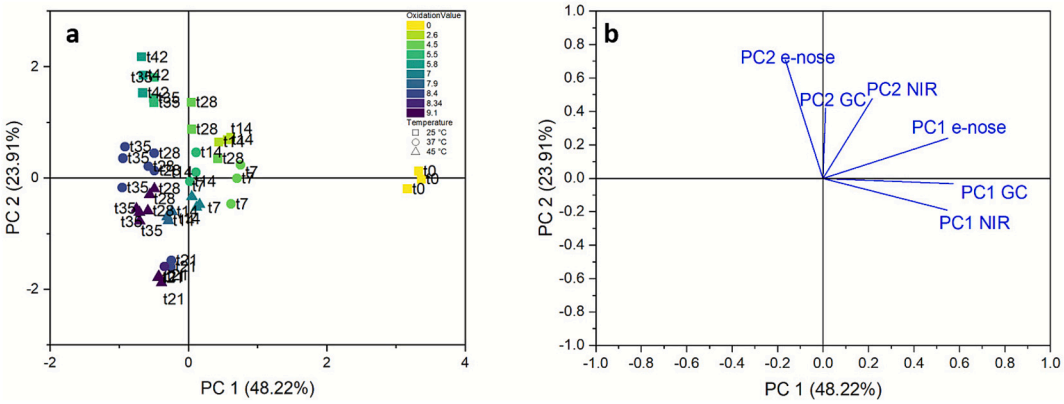

Fig. 10. Principal component analysis score plot (**a**) and loading plot (**b**) obtained from the mid-level fusion of HS-SPME-GC–MS, e-nose, and FT-NIR data collected from ground coffee samples stored at the different temperatures up to 42 days.

analysis further supported these results by distinguishing samples based on oxidation level, temperature, and moisture increase, revealing hydrolysis of fatty acids and formation of alcohols and ketones. Mid-level data fusion confirmed a consistent trend across all the tested analytical techniques, reinforcing the relationship between chemical changes and sensory degradation. Overall, the complementary use of these methods underscores the importance of controlling storage conditions to preserve coffee quality and prevent undesirable sensory transformations. This study was conducted on 100 % Arabic coffee (*Coffea arabica*) with a light roasting under defined storage conditions; thus, the generalizability of the findings to other species (e.g., *Coffea canephora*) or different roast levels remains to be established. In addition, classical lipid oxidation indices (e.g., peroxide value) were not measured, although their known correlation with sensory deterioration highlights their potential as complementary markers. Future studies should therefore aim to validate these results across a wider range of coffee types and include chemical oxidation indices to further develop predictive models of coffee freshness and oxidative stability.

CRediT authorship contribution statement

Federica De Agostini: Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation. **Cristina Alamprese:** Writing – review & editing, Visualization, Methodology, Investigation, Data curation. **Silvia Grassi:** Writing – review & editing, Visualization, Methodology, Investigation, Formal analysis, Data curation. **Susanna Buratti:** Visualization, Methodology, Investigation, Formal analysis, Data curation. **Simona Benedetti:** Visualization, Methodology, Investigation, Formal analysis, Data curation. **Serena Gobbi:** Formal analysis, Data curation. **Vittorio Bassi:** Formal analysis, Data curation. **Chiara Margarone:** Methodology, Investigation, Formal analysis. **Giulia Cusanno:** Methodology, Investigation, Formal analysis. **Daniela Gagliardi:** Methodology, Investigation, Formal analysis. **Sara Limbo:** Writing – review & editing, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization.

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Declaration of competing 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.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.foodres.2025.118025.

Data availability

The authors do not have permission to share data.

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