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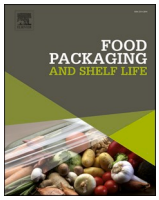

Effects of different coffee storage methods on coffee freshness after opening of packages

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ABSTRACT

The aroma of freshly roasted coffee is one of its most enticing but also ephemeral qualities. Coffee aroma starts deteriorating after roasting, and appropriate packaging and storage are needed to preserve its freshness. After a coffee package has been opened, the protective atmosphere changes, accelerating the loss of freshness and staling of coffee. This study compares four different methods for storing whole roasted beans after opening the package: (i) transferring beans into airtight canister, (ii) closing original package with tape, (iii) closing with clip and (iv) using a package with an integrated screw cap. The aroma was analyzed during storage by gas chromatography mass spectrometry (GC/MS), after grinding. Freshness indices were determined as ratios of GC/MS signal intensities of two selected compounds and used as indicators of storage stability (also called index), to compare different storage approaches. The 2-butanone/2-methylfuran index was found to be best suited to assess coffee staling for such conditions. The screw cap packaging was the best performing storage method. Using a clip, re-closing with tape or transferring beans into a container resulted in a faster loss of freshness. Findings were in line with observed changes in oxygen and carbon dioxide content inside the respective package. While changes of coffee during the primary shelf-life of coffee have been discussed in a series of publications, this study is one of the first that sheds light on how consumer practices can influence the secondary shelf-life of whole bean coffee.

1. Introduction

Coffee is one of the world's most consumed beverages, immediately recognizable by its characteristic aroma and psychoactive effect. The green coffee bean, which is the seed of the coffee fruit, does not exhibit anything resembling the aroma of coffee; the coffee aroma is generated upon roasting (Dorfner et al., 2003; 2004; Gloess et al., 2014; Hertz-Schuenemann et al., 2013; Wieland et al., 2012; Zimmermann et al., 1996). Roasting causes extreme changes in the chemical composition of the bean, as well as its physical and sensory properties. Sugars react with amino acids through Maillard reaction to form a plethora of volatile organic compounds (VOC). In roasted coffee, about 30 compounds are collectively responsible for the characteristic aroma (Blank et al., 1992; Czerny et al., 1999; Holscher et al., 1990; Mayer et al., 2000; Semmelroch & Grosch, 1995). The chemical reactions during roasting also cause physical changes; the coffee beans become brown, dry, brittle, and porous (Fadai et al., 2017).

Roasted coffee is, from a food safety perspective, a shelf-stable product, when stored at moisture content below 5%. Despite this, freshly roasted coffee is unstable and will change in composition after roasting. Chemical and physical changes that underlie quality deterioration can roughly be divided into loss of CO_2 and aroma degradation (Smrke et al., 2018a). Approximately 1% of the weight of freshly roasted coffee is CO_2, gas formed during the roasting process (Baggenstoss et al., 2007; Shimoni & Labuza, 2000; Smrke et al., 2018b; Wang & Lim, 2014), which plays an important role in the formation of crema during espresso brewing. Such a release of gas, also called degassing, occurs for about one month from whole bean coffee (Smrke et al., 2018b). The high amounts of gas released places requirements on coffee packaging. CO_2 needs to be released from the packaging, and oxygen from air prevented to enter the package. Technically, this is commonly solved through a one-way valve to prevent inflation followed by bursting of the package.

Many of the coffee aroma compounds are labile (Blank et al., 2002; Munro et al., 2003) or highly volatile (Karl et al., 2003; Pollien et al., 2003) and after the roasting process is completed, their amount in the bean decreases steadily. Using packaging materials with good barrier

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properties and eliminating oxygen inside the package during the process of coffee packaging help prevent or slow down oxidative degradation of coffee aroma, subsequently reducing loss of freshness (Gloss et al., 2014).

Coffee freshness has been studied in the past both from the perspective of chemical composition as well as taste and smell (Anese et al., 2006; Benkovic & Tusek, 2018; Buffo & Cardelli-Freire, 2004; Cotter & Hopfer, 2018; Gloss et al., 2014; Kallio et al., 1990; Leino et al., 1992; Marin et al., 2008; Nicoli et al., 2009; Ross et al., 2006; Vila et al., 2005). It is defined as a coffee as close as possible to unimpaired retention of the qualities of freshly roasted coffee. Quantitation of loss of freshness is possible by measuring the degassing process (Baggenstoss et al., 2007; Shimoni & Labuza, 2000; Smrke et al., 2018b; Wang & Lim, 2014) or by analyzing changes in coffee aroma. Analyzing the aroma via GC/MS, the freshness of coffee can be assessed by either monitoring the loss of aroma compounds that are markers of the typical coffee aroma or by the appearance of compounds that are indicative of the degradation/oxidation of coffee components (Bignardi et al., 2014; Holscher et al., 1990). The loss of aroma has been quantified previously as the sum of 2-methylpropanal, 3-methylbutanal, 2,3-butanedione, 2-methylfuran, and termed the S-index (J.C. Spadone & R.L., 1989; R. Radtke-Granzer & O.G.P., 1981). Holscher and Steinhart (1992) indicated that methanethiol has a strong impact on aroma freshness, showing a marked decrease after only one day after roasting. Czerny and Schieberle (2001) reported that the staling of coffee is the result of degradation of 2-furfurylthiol.

A simple approach to assessing changes in composition of the headspace (HS) above roasted coffee consists of referring to freshness indices (Gloss et al., 2014; Kallio et al., 1990; Marin et al., 2008). Freshness indices refer to the ratio of the signal intensities of two selected compounds from a single GC/MS chromatogram and hence are inherently less affected by instrumental drifts that may affect the intensity of GC/MS signals over time (Gloss et al., 2014). Since the metric used is a ratio between the instrumental signal intensities of two compounds in single GC runs, the methodology does not necessarily need calibration with standards.

Studies on the loss of freshness and staling have until now mostly been conducted during the primary shelf life of coffee, during storage of unopened packages. Primary shelf life of coffee can be extended by an appropriate packaging solution. However, even the best packaging and oxygen-free conditions cannot prevent loss of freshness, (Clarke, 2001) since coffee is intrinsically an unstable product. This study refers to changes which occur during the secondary shelf life, after the package has been opened and the coffee is being consumed over a period of several weeks (Benkovic & Tusek, 2018). Despite referring to the secondary shelf life, the aim of this contribution was not to determine the threshold at which the secondary shelf life expires. The threshold could be based on consumer acceptance and would therefore most likely vary between different consumer segments. Therefore, we examined how different packaging methods and consumer practices affect the rate of coffee staling during the early period of the secondary shelf life, based on objective and measurable values (freshness-indices) and whether the rate of staling can be reduced by improvements in packaging design.

2. Materials and methods

2.1. Sample description

Roasted Arabica whole beans from a single roast batch provided by Tchibo (Hamburg, Germany) were packaged under inert atmosphere (nitrogen) in 1 kg bags (composition of the bag foil: OPP /Al / PE) with an integrated one-way valve and stored at room temperature. Samples were rested for 3 weeks after roasting, to allow for initial degassing and account for typical distribution systems from roaster to consumer. For the trial samples, the coffee bags were opened, and 49 g of coffee taken out of the packaging twice per week. O_2 and CO_2 content were measured twice per week before each sampling and freshness indices were determined once per week. For the reference sample (coffee stored in closed bag at room temperature), at each measurement's time O_2 and CO_2 content were first measured, then bags were opened, and the freshness indices of the coffee determined. All storage experiments were performed in five parallel replicates.

Once bags were opened after the 3 week resting time, they were re-closed for the duration of the study in four different ways (Fig. 1): **screw cap** – coffee samples were taken for measurements through the on-package integrated closing system; **clip** – once the package was initially opened, it was re-closed using a clip after each time a coffee sample (49 g coffee) was taken; **canister** – after the package was initially opened, the beans were transferred into an airtight metal container and subsequently the beans were taken each time from the container; **tape** – after opening, the package was re-closed after every sampling, using a tape. These four closing options during secondary shelf life were complemented by a **reference** – standard sample, unopened packages; for each analysis a new package was opened. All subsequent data and graphs are labelled by the duration of the study, starting at three weeks after roasting.

2.2. Chemicals

The following chemicals were used as external standards for GC/MS identifications: dimethyl disulfide (>98%), 2-butanone (>99%), 2-methyfuran (99%) supplied by Sigma-Aldrich (Buchs, Switzerland) and methanethiol (5% in triacetin) supplied by Penta (Livingston, USA).

2.3. Determination of O_2 and CO_2 in packages

The O_2 and CO_2 concentration inside packs were measured using a CheckPoint II device (Dansensor, Ringsted, Denmark) to assess the evolution of the atmosphere within each packaging during the trials. Some packages already had elevated oxygen concentration at the start of the trials. Considering that they were initially all packed under inert atmosphere (N_2), packs with an initial O_2 concentration of $> 0.5\%$ were discarded.

2.4. Sample preparation

Whole coffee beans were ground just before GC/MS analysis with an espresso grinder (KED 640, Ditting), set to a grind level 3.75 (1: finest

Fig. 1. Five types of package handling used in this study: reference sample, unopened packages; screw cap, using the on-package integrated closing system; clip, closing packages with a clip; cannister, beans transferred into an airtight metal container and subsequently the beans were taken each time from the container; tape, package was closed after every sampling by taping the package using a tape.

setting, 8: coarse setting). After grinding, 4.0 g of coffee powder were transferred into a 20 mL glass GC/MS vial. Vials were flushed with nitrogen before closing, using a screw cap with silicone/PTFE septa (white/blue), 1.5 mm.

2.5. Headspace gas chromatography mass spectrometry (HS GC/MS)

One sample at a time was prepared per each package (5 different types of packages including the reference, 5 repetitions per type of packaging); the HS above the ground coffee was analyzed using GC/MS (7890 A/5975 C, Agilent Technologies). Samples were handled by a MPS2 autosampler (Gerstel, Germany). Before injection, samples were incubated for 20 min at 70 °C in the vial, 1 mL of gas was sampled with 0.2 mL/s and injected into the GC/MS system. The separation was performed on a DB-WAX column (30 m x 0.25 mm × 0.25 μm), with a He-carrier gas flow 1 mL/min, split 30:1 and injector temperature 250 °C. The temperature program was the following: 20 °C for 6 min, then 10 °C/min until 70 °C, 5 °C/min until 170 °C and 40 °C/min until 220 °C. The compounds used to form the freshness indices were identified by comparing mass spectra and retention times to those of external standards and further checked for each separate sample by comparison of the spectra to the NIST 11 Mass Spectral Database to ensure retention time reproducibility.

2.6. Data analysis

Data analysis was performed using R Statistical Computing (R Foundation for Statistical Computing, Vienna, Austria), version 3.6.1. The reference samples were considered as external standard and the measurements were calibrated relative to the standard samples. The intensities of GC/MS signals (peak area) were used to calculate freshness indices. The evolution of indices was modelled using linear regression and the resulting models were compared using t-test of models with a dummy model interaction variable and ANOVA of nested models. For principal component analysis (PCA), the 55 peaks detected from the GC/MS chromatograms (Table A1, supplementary material) were normalized for total GC/MS ion intensity, centered, and scaled.

3. Results and discussion

Loss of freshness and staling after opening coffee packages and storing under different conditions was examined using GC/MS analysis of HS above ground coffee. GC/MS chromatogram peak area was used for development of freshness indices and PCA. To contextualize the findings based on GC/MS analysis, CO_2 and O_2 content in the packaging was measured and compared for different storage conditions and referenced against the unopened packaging.

3.1. Freshness indices

The staling of coffee in different storage and handling conditions was monitored using two different indices that have been described before: 2-butanone / 2-methylfuran and dimethyl disulfide / methanethiol (Gloss et al., 2014). The indices were calculated relative to the reference sample that was applied as an external standard. The quantification of the coffee staling in a secondary shelf-life scenario is therefore a value relative to the equivalent sample within its primary shelf-life, stored under the same conditions. This use of indices is slightly different from the use described by Gloss et al. (2014), where they were mostly used as an absolute indicator of the freshness of coffee, without calibration or a reference. With the method described here, calibration of the index is performed against the freshness index of an external reference sample to account for the relative changes caused only by the effects of the opened packages, therefore the results were expected to be more robust.

In line with a previous study (Gloss et al., 2014), the 2-butanone / 2-methylfuran index has been shown to be well suited to assess changes of coffee freshness when stored as whole beans. In the study conducted by Gloss et al. (2014), the beans were stored in primary shelf-life conditions for a year. It is important to note that this study only covers a time-period of 9 weeks; the 2-butanone / 2-methylfuran index remained stable for unopened packages, showing a slight downward trend (slope −0.0099, $p < 0.001$). However, in other samples the index increased markedly after correction with the reference sample (Table 1).

The evolution of the 2-butanone / 2-methylfuran index is presented in Fig. 2a. Both compounds are relatively stable in coffee with respect to chemical reactions. Hence, the evolution of this index can be traced back primarily to differences in volatility between both compounds. While 2-methylfuran is one of the most volatile compounds in coffee (Boiling Point: 63–66 °C; Vapor Pressure: 176 mmHg @ 25 °C.; logP (o/w): 1.850), 2-butanone has a slightly lower volatility and is much more polar (Boiling Point: 78–80 °C; Vapor Pressure: 91 mmHg @ 25 °C.; logP (o/w): 0.290). Changes in the index are therefore mainly driven by the different loss via evaporation of both compounds during storage.

The freshness index 2-butanone / 2-methylfuran increases over time for all methods of packaging, due to loss of 2-methylfuran, most probably through evaporation. This index shows significant differences between the four types of packaging closures employed during the study. The highest rate of increase and greatest difference to the reference packaging were for the beans transferred into a canister, followed by the packaging whereby the package was re-closed using tape each time after taking samples. These methods cause the most change in the HS atmosphere when sampling coffee beans and during storage, causing the fastest increase in the freshness index.

The increase in the 2-butanone / 2-methylfuran index was modelled by a simple linear model. Differences in loses of aroma by using different packaging methods after opening are reflected in the slope of the linear model (Table 1). The significance of the difference between the models was found to be high; the slope of the index was significantly different between any two combinations of two linear models. Nested ANOVA of all combinations of two linear models suggests significant differences (Table 1).

The 2-butanone / 2-methylfuran index clearly shows that different ways of closing the packaging during the study lead to varying rate of loss of volatile coffee aroma compounds. Hence, this study demonstrates that the 2-butanone / 2-methylfuran index enables differentiation, based on the loss of highly volatile aroma compounds, between closure methods, employed under secondary shelf-life conditions.

The dimethyl disulfide / methanethiol ratio is a sensitive index to monitor coffee staling. Its sensitivity is due to methanethiol being highly volatile, reactive and sensitive to oxidation (Clarke, 2001; Holscher et al., 1990). Through oxidation of methanethiol, dimethyl disulfide and subsequently dimethyl trisulfide are formed (Chin & Lindsay, 1994; J.C. Spadone & R.L., 1989). While methanethiol decreases during storage, its oxidation product dimethyl disulfide is formed before it further degrades. This behavior of dimethyl disulfide / methanethiol is reflected in a steady increase of the index value for the opened packages (Fig. 2b). Within eight weeks of storage, the GC/MS signal intensity of methanethiol decreased for the opened packages to approx. 15% of the initial values (data not shown), irrespective of the handling of the opened packages. For the coffee in the reference packaging (primary shelf life),

Table 1
Linear models of development of 2-butanone / 2-methylfuran freshness index of coffee during the study.

Sample	Intercept	Slope	Slope t-test model interaction	Nested ANOVA
Cannister	1.04086	0.06669	a*	a*
Tape	0.99949	0.05827	b*	b*
Clip	0.99803	0.03987	c*	c*
Screw cap	1.00248	0.03269	d*	d*

* $p < 0.001$ for all combination of models.

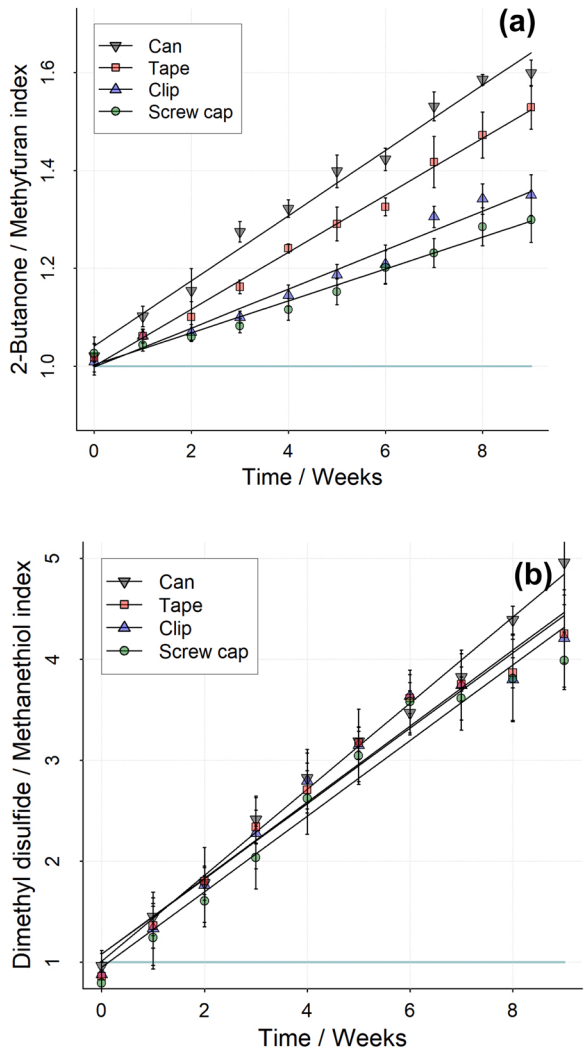

Fig. 2. The development of two coffee freshness indices: (a) 2-butanone / 2-methylfuran; (b) dimethyl disulfide / methanethiol, after opening the package through the duration of the study.

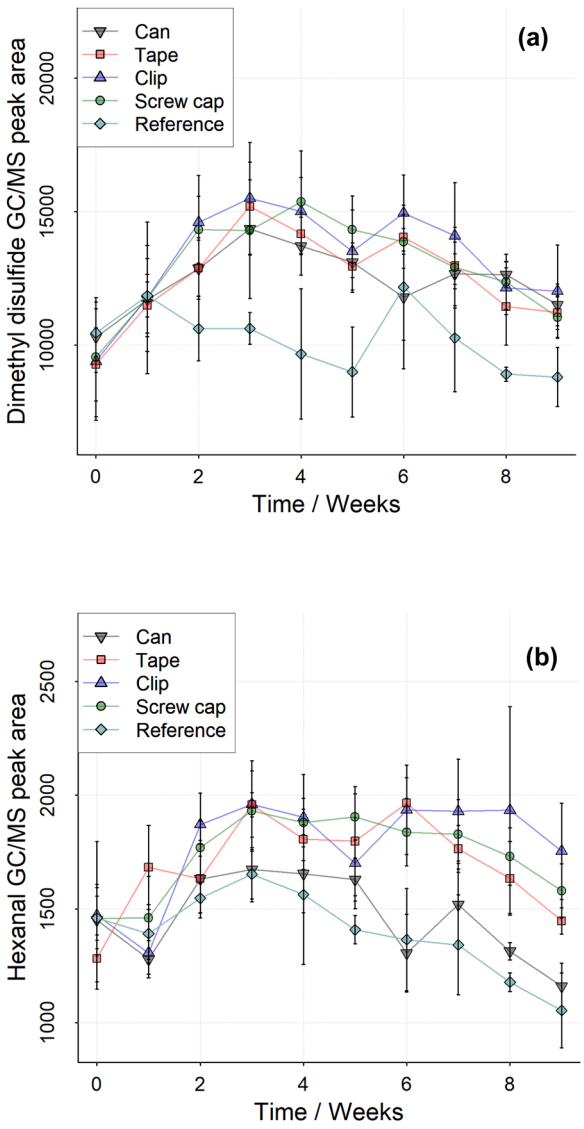

Fig. 3. The MS response of the dimethyl disulfide (a) and hexanal (b) measurements of ground coffee as a function of the time after opening the package.

methanethiol was also significantly reduced to approx. 33% of the original value at the start of the study (data not shown). In the case of beans being stored in inert atmosphere, the decrease of methanethiol observed in the packaging is caused largely by the inherent instability of methanethiol in coffee beans (readily partitioning into the HS), in addition to some reactivity with residual oxygen in the packaging. Because of the high sensitivity and rapid changes of methanethiol, this index does not distinguish between the four samples with opened packages. It appears that the oxygen introduced when opening the package strongly decreased the methanethiol content, without differentiation between the different methods of closure during the secondary shelf-life. The inclusion of methanethiol makes this index clearly differentiate between unopened and opened packages, however this index is unsuitable to distinguish between staling (loss of freshness) in different closing methods of the packaging during secondary shelf life.

3.2. Staling markers

Out of 55 compounds detected with the HS-GC/MS method (Table A1, supplementary material), two compounds significantly and consistently increased in measured signal intensity with storage time, for the opened coffee packages (Fig. 3). First is the dimethyl disulfide, the reason for the dimethyl disulfide increase being that it is an oxidation product of methanethiol. The increase occurs mainly between the start of the study (opening the package) and week 3 (p values for two sample Welch t-test for clip, can and tape samples: $p < 0.01$, screw cap sample: $p < 0.05$). The signal intensity for samples in closed packages remained stable, with an insignificant ($p = 0.12$) tendency to decrease. This indicates that in a secondary shelf-life situation and until week 3, the methanethiol concentration is high enough to produce measurable amounts of dimethyl disulfide, whereas after week 3, there is little methanethiol left for any further increase of dimethyl disulfide content.

Hexanal is the second compound that increased in measured signal intensity with time for samples in opened packages (p values for two sample Welch t-test between first measurement and after 3 weeks: for clip and can samples: $p < 0.05$, tape and screw cap samples: $p < 0.01$, reference sample not significant). Hexanal has been previously suggested as a marker of coffee staling or a component of a freshness index (Marin et al., 2008) since it is a product of fatty acid oxidation. The results of this study show that after package opening there is a small increase in the measured signal for hexanal in the HS above the ground coffee sample. The increase in measured hexanal signal is relatively small and it can be assumed that for the samples in this study, such a small increase is too insignificant to be considered to have an effect of perceived staling or to be considered as a practically applicable staling

marker.

3.3. Principal component analysis

To contextualize the results from the 2-butanone / 2-methyfuran freshness index that differentiates how coffee packaging is used after opening, PCA analysis was performed on the whole GC/MS dataset (55 chromatogram peaks, Table A1, supplementary material). An untargeted approach in the data analysis confirms what can already be seen with the freshness index. The PCA scores (Fig. 4) of the primary shelf-life sample are grouped closely together at lowest PC1 values. The PC1 dimension separates in the time axis the sampling points of opened packages by time after opening. The numbers in Fig. 4a show the weeks after opening the package and consecutive numbering can be seen along the PCA plot. The PC2 dimension further separates the different methods of storing the coffee after initial opening. This separation by PCA strongly indicates that points closest to the reference sample in the PC1-PC2 two-dimensional space should be considered as having lost the least amount of aroma compared to the reference sample.

3.4. CO_2 and O_2 content

The evolution of CO_2 and O_2 in the packages during storage is shown in Fig. 5. The packages used for experiments had an initial concentration, after three weeks of storage of the unopened packaging, of approx. 70% (v/v) in CO_2 and less than 0.5% (v/v) for O_2. Packages with initial O_2 concentrations above 0,5% (v/v) were removed from further experiments. The O_2 concentration in the unopened packaging remained very low and close to zero throughout the experiments, while the CO_2 concentration slightly increased to around 77% at the end of the 9-week storage period due to the degassing process. These results show that the packages had an effective barrier and that the one-way valves integrated into the packages performed as expected.

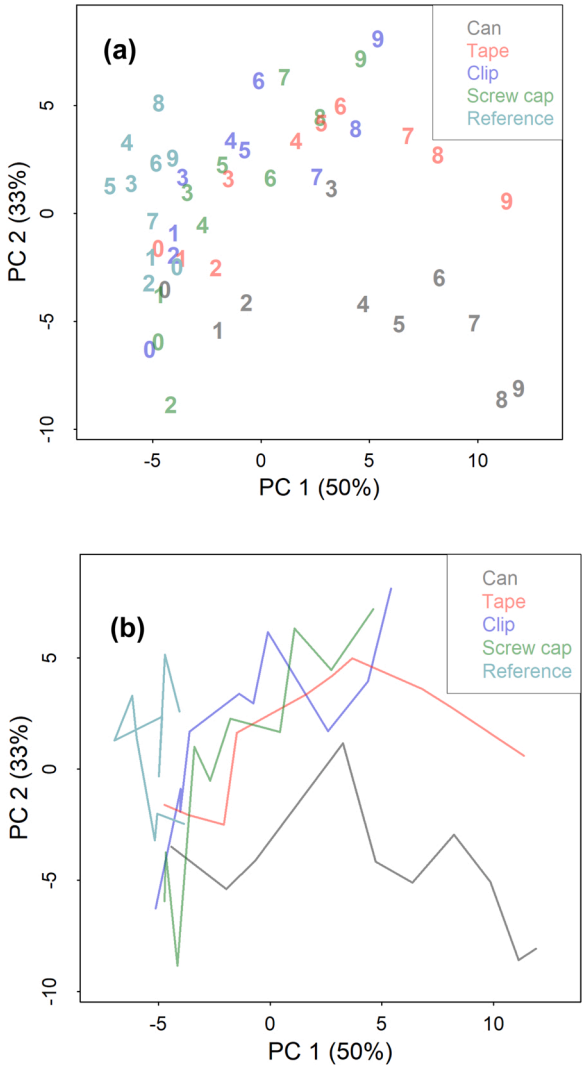

Fig. 4. PCA of 55 GC/MS chromatogram peaks. The numbers on the plots are weeks after package opening, except for the references sample where each time five new packages were opened.

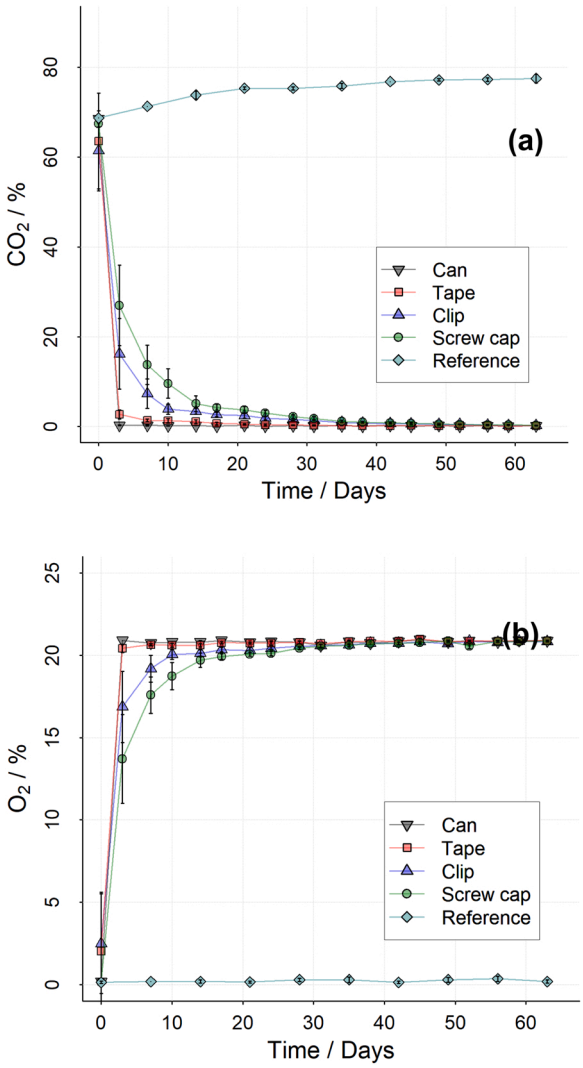

Fig. 5. The change in concentration of CO_2 (a) and O_2 (b) in packages during the study.

The composition of the air inside the opened package samples showed a rapid change already within the first 10 days of storage, with marked differences between the four packaging treatments. For either transferring coffee into a container by fully emptying the coffee bag or closing the pack using a tape after an initial opening the coffee pack causes, a rise of the O_2 concentration to ambient conditions (21%) was observed already within a week of the package being opened (Fig. 5a). In parallel, the CO_2 concentration fell to essentially zero. Because CO_2 and nitrogen represent the protective atmosphere for coffee in the packaging, the immediate change of the package HS composition to essentially air makes the coffee much more exposed to oxidative degradation and staling. The use of a clip or the screw cap packaging provided a slightly better performance. Nevertheless, after initially opening of the package, the O_2 concentration increases to above 10%. After 2 weeks (4

samplings), the O_2 concentrations inside packages were within 1% (v/v) of ambient oxygen levels.

The evolution of the CO_2 concentration (Fig. 5b) mirrors the pattern for O_2, for all four types of packaging. For the transfer to the container or using a tape for re-closing, we observe that the whole HS inside the packaging is rapidly replaced by air. By measuring the drop in CO_2 concentration, the magnitude of the HS replacement during each sampling can be estimated. The HS replacement each time when taking a coffee sample from the package, was found to be approx. 60% when using the screw cap packaging, and 75% when using a clip to close the package. For the cannister and tape samples, each time a sample is taken, effectively the full HS volume is replaced by air. In the case of the container, this is due to the elimination of the protective atmosphere from the original packaging and the large opening surface. For closing the package with a tape, it is assumed that air-tight closure was not achieved and therefore a full exchange occurred by the time the next sampling took place. These observations are consistent with the GC/MS results and substantiate the conclusions based on the HS composition.

4. Conclusion

This is the first systematic study on whole bean coffee staling after opening the package, covering four major consumer coffee handling habits. It complements former work on the shelf life studies of coffee and sheds some light on the main drivers of loss of freshness. Both a targeted and untargeted approach were used to determine how differently stored coffees stale. This study assessed what happens to the coffee under secondary shelf-life and typical coffee handling conditions, from a chemical point of view. The on-package integrated screw cap seems to be an efficient system to best protect the coffee aroma. However, to fully evaluate the duration of the secondary shelf-life and quality acceptance of samples, sensory studies need to be conducted in addition to chemical analyses, to determine an acceptance threshold. Work is underway to better understand the chemistry responsible for the changes involved in the different indices and to develop increasingly detailed strategies and recommendations for preserving freshness and quality of coffee. Furthermore, chemical analysis will be complemented with sensory evaluation to combine both aspects and develop a more comprehensive approach to the study of staling and loss of freshness during secondary shelf-life.

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CRediT authorship contribution statement

Samo Smrke: Formal analysis, Visualization, Writing – original draft. **Jan Adam:** Resources. **Samuel Mühlemann:** Investigation. **Ingo Lantz:** Resources, Writing – review & editing. **Chahan Yeretzian:** Project administration, Funding acquisition, Writing – review & editing.

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.fpsl.2022.100893.

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