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EXPLORING THE IMPACT OF DIFFERENT PACKAGING TYPES AND REPEATED PACKAGE OPENING ON VOLATILE COMPOUND CHANGES IN GROUND ROASTED COFFEE

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ABSTRACT

In this study, the dynamics of volatile compound alterations during the storage of ground roasted coffee in various packaging were investigated using solid-phase microextraction-gas chromatography-mass spectrometry. Each type of packaging underwent repeated opening and closing, simulating the gradual consumption behavior of the consumer who consumes ground coffee in smaller doses over time. The experimental results revealed that after 30 days of storage, 35 to 45% of the volatile substances originally present in the coffee were lost regardless of the type of packaging used. Notably, storing coffee in a paper sachet proved to be the least effective method, resulting in a loss of over 80% of all volatile coffee substances during this storage period.

Keywords: coffee, storage, packaging, GC-MS, shelf life, volatile substances, moisture

INTRODUCTION

Coffee, revered for its rich aroma and bold flavor, is a staple beverage enjoyed worldwide. The sensory experience of coffee is intricately linked to its volatile compounds, which contribute to its distinctive fragrance and taste (**Mahmud *et al*., 2020**). However, the delicate balance of these compounds can be easily altered, especially during storage, leading to changes in quality that may affect consumer satisfaction (**Glöss *et al*., 2020**). Ground coffee is a relatively stable product in terms of spoilage. Thanks to the high temperature used when roasting coffee beans, coffee exhibits low water activity, and its stability is also aided by the products generated during Maillard reactions, which exhibit antimicrobial activity (**Anese *et al*., 2006; Supanivatin *et al*., 2023**). In order to maintain quality for a long time, it is important to choose a suitable packaging material for the packaging of ground coffee. Effective ground coffee packaging acts as a barrier against oxygen, which can lead to oxidation and degradation of flavor compounds, resulting in stale and lackluster coffee. Secondly, it shields the coffee from moisture, preventing it from absorbing excess water that can accelerate spoilage and the growth of mold and bacteria. Additionally, packaging plays a role in protecting coffee from exposure to light, which can cause undesirable flavor changes and deterioration over time. Lastly, packaging materials and designs also influence the coffee's susceptibility to temperature fluctuations, which can accelerate chemical reactions and compromise its quality (**Han, 2014**).
The rate of degradation of ground coffee can increase significantly after the package is opened by the consumer. This degradation affects the so-called secondary shelf life of packaged food (**Makri *et al*., 2011; Nicoli & Calligaris, 2018**). For ground coffee, the secondary shelf life is an important parameter since consumers typically do not consume the entire package at once; instead, they consume the coffee gradually in smaller doses. The consumer stores ground coffee in various ways. Often, coffee is kept in its original packaging, which is repeatedly sealed in different manners after being opened. Another frequently used option is to transfer the ground coffee from the original packaging to a new container, commonly glass or metal (**Smrke *et al*., 2022**). Hence, this study aims to clarify the intricate connection between consumer habits, packaging selections for ground coffee, and the fluctuating dynamics of volatile compound alterations during storage of roasted ground coffee.

MATERIAL AND METHODS

For experiments, coffee from Brazil (100% Arabica) was roasted in the roastery and immediately after grinding the coffee was packed into individual packages purchased from a coffee shop in Brno, Czech Republic. A glass container with twist-off lid, steel can with polypropylene lid, paper sachet (70 µm) closed by aluminum sealing liner, zip-lock stand up composite pouch consisting of paper (50 µm), aluminum foil (7 µm) and cast polypropylene (60 µm), zip-lock stand up composite pouch consisting of paper (70 µm), biaxial oriented polyamide (15 µm) and cast polypropylene (50 µm) were used for coffee storage experiment. All packages were sized for storage of 250g ground coffee. From individual packages a sample was taken for analysis at five-day intervals for up to 30 days. All storage experiments were performed in triplicate. For the volumetric titration high purity methanol (purity>99.9 %, Honeywell, Hopewell, USA), Karl-Fisher titrant (Hydranal, Honeywell) and analytical standard containing 1 % of water (Honeywell) were used.
The moisture of coffee samples was determined gravimetrically by drying the pre-weighed samples in an aluminium dish in a hot air oven at 105 °C. Water content in all samples was determined by Karl-Fisher volumetric titration method **(International Organization for Standardization, 1978)** using Titrino 701 (Mtrohm, Herisau, Switzerland) titrator. Solid phase microextraction (SPME) and analysis by gas chromatography coupled with mass spectrometer (GC-MS) were performed according to the method described by **Burdějová and Vítová (2019)**. For SPME extraction, 1 g of coffee sample was placed into a 10 mL screw cap glass vial (Sigma Aldrich, Darmstadt, Germany) and immediately analysed. SPME was performed using Divinylbenzene/Carboxen/Polydimethylsiloxane (DVB/CAR/PDMS) fibre 50/30 µm (Supelco, Bellefonte, Pennsylvania, USA) in headspace (HS) mode. Final analyses were carried out on Trace™ 1310 GC system with split/splitless injector (Thermo Fisher Scientific Inc., Waltham, Massachusetts, USA), capillary column TG-WaxMS (30 m × 0.25 mm i.d., 0.5 µm film thickness, Thermo Fisher Scientific Inc., Waltham, Massachusetts, USA) coupled with mass spectrometer ISQ™ LT Single Quadrupole (Thermo Fisher Scientific Inc., Waltham, Massachusetts, USA). All analyses were performed in triplicate.
The statistical variability of the data was managed using Microsoft Excel (Microsoft, USA) and XL-Stat (Addinsoft, France). Mean values representing changes in water, moisture, and volatile content during the storage experiment were compared using Tukey's test ($p < 0.05$).

RESULTS AND DISCUSSION

Water and moisture content in coffee during storage

Changes in water content and moisture during coffee storage are summarized in Table 1. The water content in fresh coffee was 2,38±0,02%. The type of packaging significantly influenced water content during coffee storage; however, except for coffee stored in a paper sachet, the differences were small. The water content of coffee during storage increased by an average of 0,98%. In the case of coffee stored in a paper sachet, there was an observed increase in water content of 1,68% during storage. These results correspond to the water vapor permeabilities of the

individual packaging materials, with paper having the highest permeability for water vapor.
Due to the hygroscopic nature of roasted coffee, when explaining the increase in water content, it is necessary to consider the adsorption of water from the air upon opening the packaging and handling the coffee, in addition to water vapor passing through a sealed package. Another factor to consider is the different types of closures used in this study. Literature lacks data on the water content of ground roasted coffee; instead, moisture in coffee is often reported. In this study, the moisture content in fresh coffee was 4,64±0,05% and increased during coffee storage up to 5,29±0.07%. This result is consistent with results published by **Nakilcioglu and Ötles (2019)**. Increased water content in ground coffee leads to a shorter product shelf-life due to the faster degradation of aroma compounds (**Baggenstoss *et al*., 2008**). The legal limit for moisture content in ground coffee is usually 5% (**Corrêa *et al*., 2016; Leal *et al.* 2021**). The volatile content in fresh coffee was 2,26%, and during coffee storage, a loss of 0,6% to 1,03% of volatiles was observed depending on the packaging material used. As expected, the most volatiles were lost in the coffee stored in the paper sachet (Table 1).

Table 1 Changes in water content and moisture during coffee storage

	G		M		C1		C2		PAP	
day	**wat**	**moist**	**wat**	**moist**	**wat**	**moist**	**wat**	**moist**	**wat**	**moist**
0	2,38	4,64	2,38	4,64	2,38	4,64	2,38	4,64	2,38	4,64
5	2,67	4,79	2,64	4,78	2,48	4,68	2,45	4,68	2,64	4,58
10	2,78	4,82	2,72	4,82	2,61	4,72	2,58	4,74	2,72	4,62
15	2,89	4,88	2,53	4,85	3,08	4,81	3,05	4,82	3,33	4,67
20	3,01	4,91	2,68	4,88	3,30	4,91	3,29	4,91	3,68	4,65
25	3,09	4,89	3,10	4,90	3,31	4,88	3,46	4,89	3,90	4,69
30	3,27	4,93	3,36	4,91	3,34	4,96	3,51	4,98	4,06	4,78

Note: G=glass container, M=steel can, C1= composite pouch consisting of paper, aluminium foil and cast polypropylene, C2= composite pouch consisting of paper, biaxial oriented polyamide and cast polypropylene, PAP=paper sachet, wat=water content (%), moist=moisture content (%). The relative standard deviation was less than 5 % for all results.

Table 2 Relative changes in water content, moisture and volatiles during coffee storage

	G	M	C1	C2	PAP
water Δ30-0 (%)	+0,89[d]	+0,98[c]	+0,96[c]	+1,13[b]	+1,68[a]
moisture Δ30-0 (%)	+0,29[b]	+0,27[b]	+0,32[a]	+0,34[a]	+0,14[c]
volatiles Δ30-0 (%)	-0,60[e]	-0,71[c]	-0,64[d]	-0,79[b]	-1,54[a]

Note: G=glass container, M=steel can, C1= composite pouch consisting of paper, aluminium foil and cast polypropylene, C2= composite pouch consisting of paper, biaxial oriented polyamide and cast polypropylene, PAP=paper sachet. Relative standard deviation was less than 5 % for all results. [a-d] Different superscripts within one line means statistically significant differences at $P<0.05$

Identification of volatile compounds in coffee

A total of 59 aromatic compounds were detected in the fresh ground coffee sample, as shown in Fig. 1. Out of the total detected volatile compounds, 50 were identified through mass spectral matching. The total number of identified compounds in the coffee sample aligned with the results of other authors who have studied the aromatic profile of coffee (**Mondello *et al*., 2004; Marin *et al*., 2008, Toledo *et al*. 2016; Wang *et al.* 2021, Carcea *et al.* 2023**).
The most prevalent group of volatile substances in the coffee sample were pyrazines, including 2-methylpyrazine, 2,3-dimethylpyrazine, 2,5-dimethylpyrazine, 2,6-dimethylpyrazine, 2-ethylpyrazine, 2-ethyl-6-methylpyrazine, 2-ethyl-5-methylpyrazine, 2-ethyl-3-methylpyrazine, 2-ethyl-3,5-dimethylpyrazine, 2,6-diethylpyrazine, and 2-propylpyrazine (2-13 in Fig 1). Other significant compounds found in the coffee sample included 1-methylpyrrole (no. 1 in Fig.1), acetic acid (no.14 in Fig.1), 2-oxobutylacetate (no.15 in Fig.1), furfural (no.16 in Fig.1), furfurylacetate (no.17 in Fig.1), 5-methylfurfural (no.18 in Fig.1), 2-furanmethanol (no.19 in Fig.1), 1-furfuryl pyrrole (no.20 in Fig.1), 2-methoxy-4-methylphenol (no. 21 in Fig.1), and 2-methoxy-4-vinylphenol (no. 22 in Fig.1).
Pyrazines are formed in coffee during the roasting process through the transformation of fructose and glucose and ongoing Strecker reactions, providing the coffee with an earthy-roasty, coffee-like, and walnut aroma (**Caporaso *et al.*, 2014**). 2-furanmethanol, a Maillard-reaction product, primarily arises from the thermal degradation of glucose and sugar polymers, contributing to the caramelic, burnt, and smoked aroma of coffee (**Caporaso *et al*., 2014**). 2-oxobutylacetate, a compound with a typical coffee-like aroma, may be formed in coffee through the esterification of acetic acid resulting from the degradation of sucrose (**Ginz *et al*., 2000**). Phenol derivatives are formed in roasted coffee through the thermal degradation of chlorogenic acids (**Yeager *et al*., 2021**), providing the coffee with a characteristic smoky aroma (**Caporaso *et al*., 2014**).

The identified compounds were categorized according to their chemical nature into five groups: pyrazines, furans, aldehydes and ketones, pyrroles, and phenolic compounds for a clearer presentation of the achieved results.

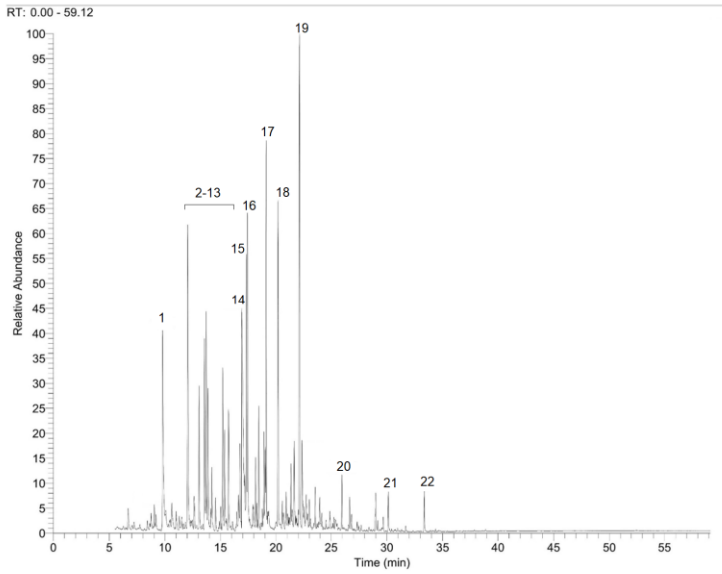

Figure 1 HS-SPME-GC-MS chromatogram representing the most abundant volatile compounds in roasted coffee used in the experiment. The peak numbers correspond to the substances described in the chapter above.

Changes in volatile compounds in coffee during storage

As results from Fig. 2, none of the packaging used did not prevent the loss of volatiles from stored coffee. The reason for this loss is evaporation or degradation of volatiles during storage as well as different barrier properties of the packaging for the oxygen and air humidity. Based on the peak area's changes between storage time 0 and 30 days it can be concluded that the amount of degraded or evaporated substances in all packages except the paper sachet was between 35–45%. Coffee stored in a paper sachet lost more than 80% of volatiles after 30 days of storage. In terms of barrier properties, metal and glass packaging provide the best protection for packaged coffee against oxygen and air humidity from the surrounding environment. However, when comparing the results obtained during the storage of ground coffee in various packaging, it is essential to consider that these types of packaging also have the largest free space between the packed ground coffee and the container's cover, as well as the largest surface area through which volatile substances from the ground coffee can migrate into this space. These properties result in a more intense exchange of volatile substances accumulated in the headspace with the surrounding air each time the metal or glass packaging is opened.

The most abundant volatiles in coffee were pyrazines. Figure 3 shows the course of the change in the peak area of 2-methylpyrazine in ground roasted coffee during storage. Other pyrazines showed a similar course. The 2-methylpyrazine peak area in the coffee stored in tested packaging decreased by about 40%. The paper packaging could not prevent the evaporation or degradation of 2-methylpyrazine and already after 5 days of storage, the 70 % decrease of 2-methylpyrazine peak area in the stored coffee was observed.

The second most abundant group of volatile substances in coffee were furans. Among the furans 2-furanmethanol was the most abundant. The course of the change in the 2-furanmethanol peak area in coffee stored in different packages is shown in Figure 3. According to **Perez-Martinez *et al.*, (2008)**, 2-furanmethanol is a relatively stable substance and the content of this substance does not change significantly during storage. These results were confirmed in this work because the loss of 2-furanmethanol in stored coffee after 30 days ranged between 10 and 20 %. A more significant loss (about 35 %) was recorded for coffee stored in the paper sachet.

The third most abundant group of volatiles in ground roasted coffee were aldehydes and ketones. Within this group of compounds, 2,3-pentanedione was the most abundant. Pentanedione is considered to be a relatively unstable substance in coffee (**Perez-Martinez *et al*., 2008**), which has been confirmed in present study. The change in the content of 2,3-pentanedione in stored coffee was very fast and after 30 days of storage a decrease of the 2,3-pentanedione peak area by up to 80% was recorded. In coffee stored in paper sachet, almost 100% of the original 2,3-pentanedione content was lost (results not shown). A significant decrease in the concentration of pentadione in the aromatic profile of coffee during its storage was also noted by the authors **Leino *et al.*, (1992)**. Ketones and aldehydes can also be among the few substances which arise in coffee during storage as products of degradation reactions. In this work, a significant increase in hexanal (about 50 %) was observed in coffee during storage (Fig.3). Hexanal is the product of oxidative degradation of unsaturated fatty acids in roasted coffee, and its formation and increase in coffee during storage has also been described by **Marin *et al.* (2008)** or by **Smrke *et al.* (2022).**

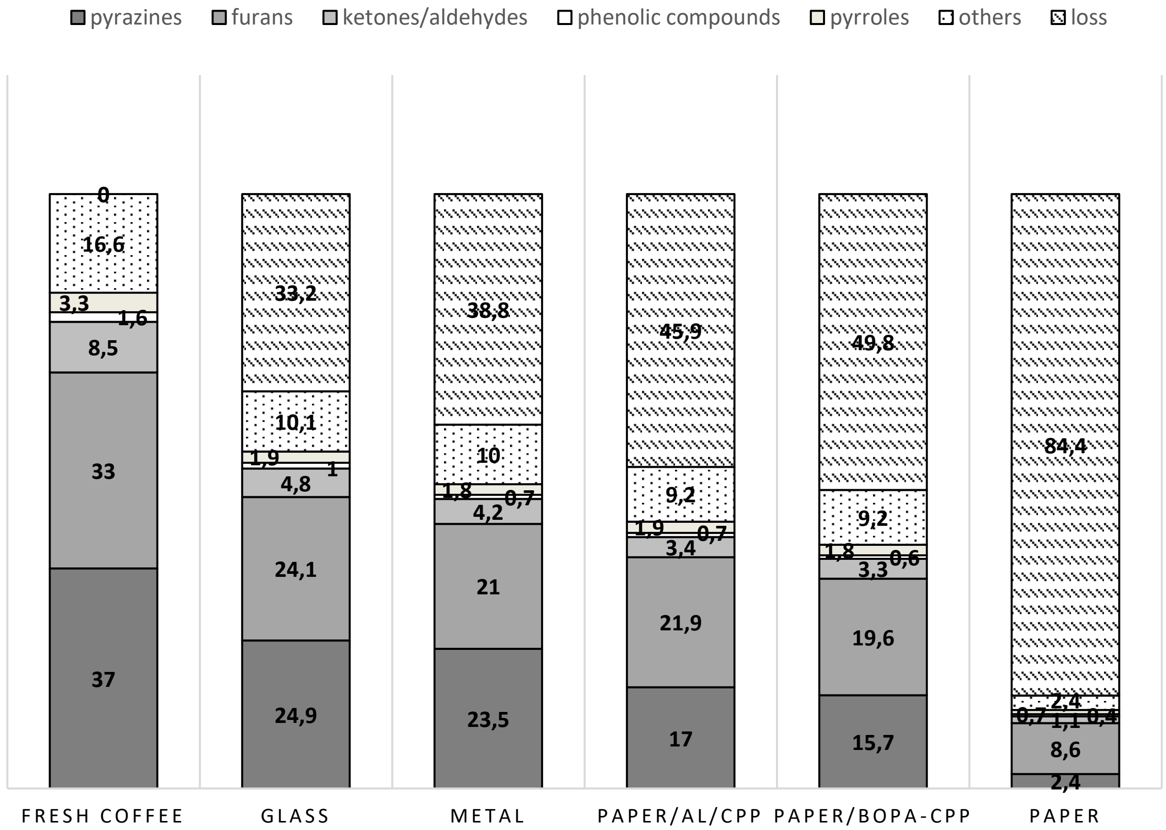

Figure 2 The percentage of groups of volatile substances in fresh ground coffee and coffee after 30 days of storage in various packaging. The relative standard deviation was less than 10 % for all results.

From the group of pyrroles, 1-methylpyrrole was the most represented in the coffee samples. Other pyrroles identified were 2-formyl-1-methylpyrrole, 1-furfurylpyrrole and 1-acetylpyrrole. The change in the content of 1-methylpyrrole during the storage experiment is shown in Figure 3. From the results, it can be seen that there was a noticeable loss of this substance during storage. In the case of coffee storage in a paper sachet, the content of 1-methylpyrrole was reduced by 90% after just 10 days of coffee storage. In other packages,

1-methylpyrrole gradually decreased to approximately 50% of the original amount in fresh coffee. The mechanism of reducing the concentration of 1-methylpyrrole could not be clarified by studying the available literature, so it is not possible to determine whether it is a loss by leakage or whether this substance is degraded during storage. A sharp decrease in the concentration of pyrroles was also observed by **Perez-Martinez *et al*., (2008)** in studying changes in brewed coffee during storage of this beverage.

The most significant volatile phenolic substances in coffee include 2-methoxyphenol, 2-methoxy-4-methylphenol, and 2-methoxy-4-vinylphenol. These compounds are generally considered relatively stable under typical storage conditions. However, like other volatile compounds, they can undergo degradation over time due to factors such as exposure to oxygen, light, and high temperatures. The degradation of volatile phenolic compounds in coffee can result in changes to its aroma profile, potentially leading to a loss of the spicy, smoky, and clove-like notes they contribute. Figure 3 depicts changes in the content of 2-methoxy-4-methylphenol during the storage experiment. The results indicate that the content of 2-methoxy-4-methylphenol gradually decreases over time. After 30 days of storage, the content of 2-methoxy-4-methylphenol decreased by approximately 20% regardless of the packaging used. An exception was observed for ground coffee packaged in a paper sachet, where a decrease of 50% in the original amount of 2-methoxy-4-methylphenol was recorded after 30 days. The monitored changes in the content of 2-methoxy-4-methylphenol are due to the relative stability of this substance caused rather by its diffusion into the surrounding space when the packaging is repeatedly opened. There are relatively few studies that investigate changes in volatile phenolic substances in ground coffee during storage. **Schedig et al. (2007)** point out in their work that the content of 2-methoxy-4-methylphenol may even increase during coffee storage, with this effect being significantly influenced by an increased water content in the coffee.

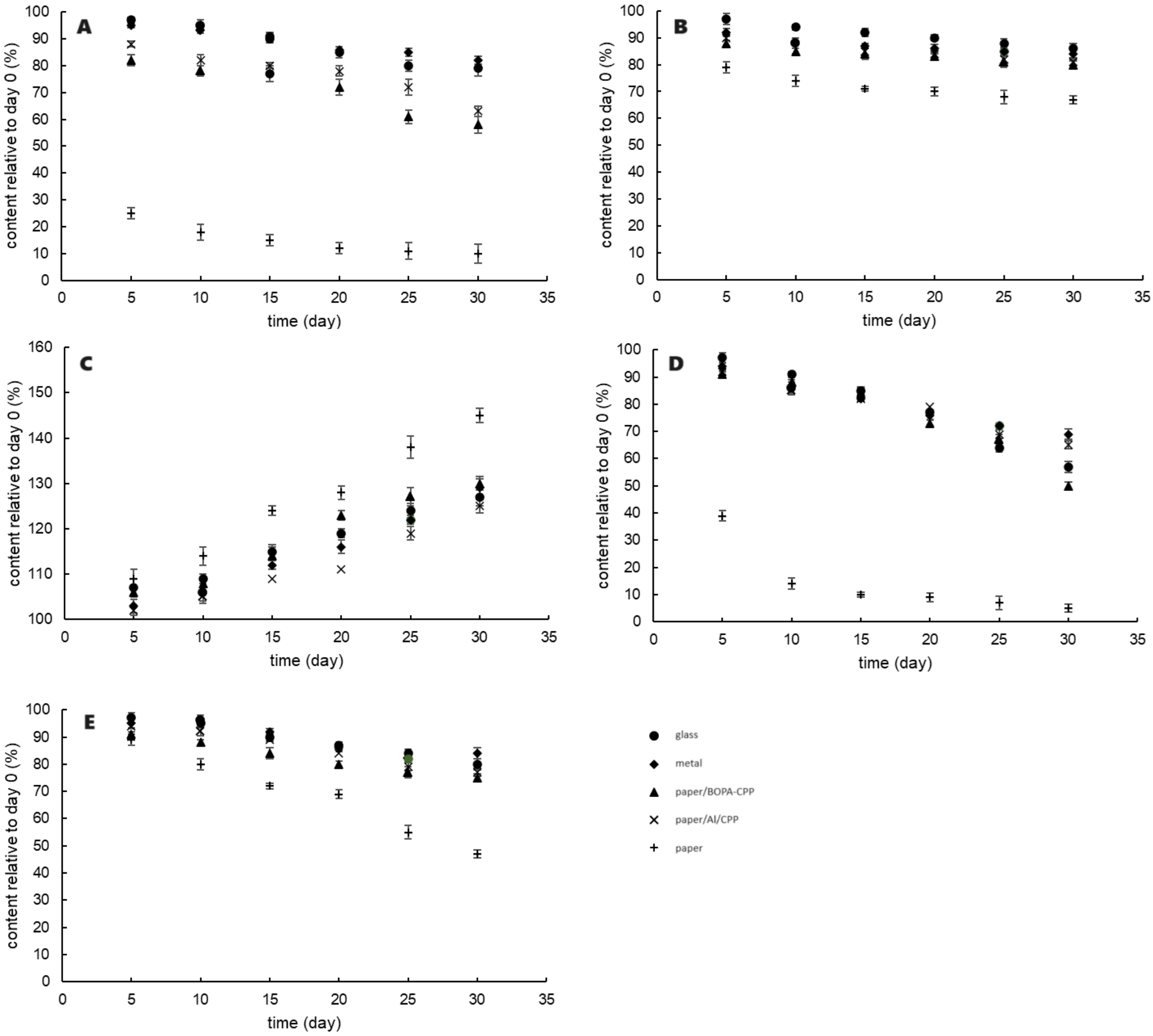

Figure 3 Change in the relative content of selected volatile substances in ground coffee during a storage experiment. A= 2-methylpyrazine, B= 2-furanmethanol, C= hexanal, D= 1-methypyrrol, E= 2-methoxy-4-vinylphenol

CONCLUSION

This study provides information about changes in water and volatile substance content in ground roasted coffee stored under typical conditions modeling consumer behavior. This storage method includes repeated opening and closing of the packaging. The results of this work have shown that even storing ground coffee in packaging with excellent barrier properties and quality closures will not prevent the loss of volatile substances or the creation of some degradation products resulting from oxidation reactions, due to the exchange of volatile substances accumulated in the headspace with the surrounding environment after opening the packaging. Storing coffee in a paper bag proved to be the least suitable option. Paper has the worst barrier properties for water vapor and gases among all other packaging materials used in this study (laminated plastic packaging, glass, or metal containers). Therefore, complete loss of some characteristic aroma substances was recorded after a few days of storage, and up to 80% loss of these substances after 30 days of storage. The amount of water in the ground coffee in the simulated storage conditions increased very slowly, and only in the case of storing ground coffee in a paper bag did it exceed an acceptable water content rate set at 5% after 30 days. The issue of food packaging is constantly evolving, and currently, there are packaging options available on the market for storing ground coffee by consumers, which minimize headspace and the total amount of air in contact with packaged coffee. Such packaging can extend the secondary shelf-life of roasted ground coffee, although the decision on whether ground coffee still meets sensory requirements ultimately lies with the consumer.

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