

## EVALUATION OF A HEADSPACE SOLID-PHASE MICROEXTRACTION WITH DIFFERENT FIBRES FOR VOLATILE COMPOUND DETERMINATION IN SPECIALTY COFFEE BREWS

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### Abstract

The aroma of coffee is the main quality factor. Compounds with floral, fruity, citrus and sometimes fermented aroma notes are defined as high quality characteristics for the specialty coffee brew. Commercial coffees mostly are at medium roast and the aroma profile is focused on balanced chocolate, caramel, toasted bread and almond bitterness aroma notes. These sensory characteristics set the focus for volatile compound profile analysis by Headspace solid-phase microextraction (HS-SPME). The most popular fibres for commercial coffee aroma profile analysis are DVB/CAR/PDMS, CAR/PDMS, PDMS/DVB and PA. There is limited research done about specialty coffee aroma profile and evaluation of more suitable fibres for solid-phase microextraction. The aim of the research is to evaluate the aroma composition of specialty coffee brews using different SPME fibres. Results demonstrated that CAR/PDMS fibre, compared to other fibres, can extract significantly more volatile compounds with higher peak areas in all chemical compound groups, except phenols. The CAR/PDMS was the only fibre which could detect all 17 important volatile compounds for specialty coffee. In conclusion, from the given research evidence, CAR/PDMS fibre is suggested as the most suitable SPME fibre coating for volatile compound extraction for specialty coffee brew import. The result provides evidence for improved specialty coffee aroma profile analysis by SPME.

**Key words:** specialty coffee, volatile compounds, SPME, fibre coating.

### Introduction

HS-SPME is a simple, rapid but sensitive method for volatile compound extraction from food products and beverages (Chen, Chiang, & Chung, 2019). There are commercially available 7 SPME coatings: polydimethylsiloxane (PDMS), polyacrylate (PA), carboxen/polydimethylsiloxane (CAR/PDMS), polydimethylsiloxane/divinylbenzene (PDMS/DVB), carbowax/divinylbenzene (CW/DVB), carbowax/template resin (CW/TR) and divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) (Spitelun *et al.*, 2010). It is important to evaluate SPME coatings, extraction and desorption technique for a specific product (Chen, Chiang, & Chung, 2019). The most popular fibres for commercial coffee aroma profile analysis are DVB/CAR/PDMS, CAR/PDMS, PDMS/DVB and PA. The DVB/CAR/PDMS fibre was selected for Mestdagh *et al.* (2014) study about the kinetics of coffee aroma extraction and Lee, Kim, & Lee (2017) study for coffee brew volatile compound profile determination of reversed coffee grinding and roasting process. The PDMS/DVB fibre was confirmed in Roberts, Pollien, & Milo (2000) research as the most suitable SPME fibre coating for wider range coffee volatile compound extraction. Petisca *et al.* (2012) study suggested CAR/PDMS as a beneficial SPME fibre coating for coffee furan compound detections. The CAR/PDMS fibre also was chosen by Kim *et al.* (2018) for the prediction of key aroma compounds in differently roasted coffee samples. Commercial coffees aroma profile is focused on balanced chocolate, caramel, toasted bread and almond bitterness aroma notes.

These sensory characteristics set the focus for volatile compound profile by HS-SPME. Specialty coffee focus is on highlighting specific aroma and flavour notes from coffee by high-quality standards. From niche product specialty coffee has become a whole industry. Specialty coffee is defined as high quality coffee (Donnet, Weatherspoon, & Hoehn, 2008; Lee, Bonn, & Cho, 2013). Compounds with floral, fruity, citrus and sometimes fermented aroma notes are compared as high quality characteristics for the coffee brew (Fassio *et al.*, 2017; Piccino *et al.*, 2014; Poltronieri & Rossi, 2016; Silveira *et al.*, 2016). There is limited research done about specialty coffee aroma profile and evaluation of more suitable fibres for solid phase microextraction. The aim of the research is to evaluate the volatile composition of specialty coffee brews using different SPME fibres.

### Materials and Methods

#### *Coffee samples*

Five samples of coffee (*Coffea arabica L.*) beans were collected from two different coffee roasteries in Latvia. The main characteristics of the coffee samples are summarized in Table 1. Natural processing method was applied for Honduras\_1, Kenya, Columbia, Ethiopia coffee samples, and anaerobic processing method – Honduras\_2. All coffees are roasted at light-medium roast level and rated as specialty coffees: specialty coffee grade applies if the total specialty cup quality score is 80 points or above (Donnet, Weatherspoon, & Hoehn, 2008; Silveira *et al.*, 2016). Both roasters, according to The World Coffee Research Sensory Lexicon (2017), gave sensory characteristic

Table 1

**Main characteristics of the samples**

Sample name	Roastery	Origin	Roasting level	Sensory description from roasters
Honduras_1	roastery 1	Honduras	light-medium	dark plum, grapes, red pepper, toffee (caramel)
Kenya	roastery 1	Kenya	light-medium	blackberry, red pepper, roses, dark chocolate
Columbia	roastery 1	Columbia	light-medium	pineapple, dried apricot, elderflower
Ethiopia	roastery 2	Ethiopia	light-medium	lime, jasmine, chocolate cream, cherry brandy
Honduras_2	roastery 2	Honduras	light-medium	dried fruits, passion fruit, melon, kombucha

descriptions and the descriptions are also stated on the specialty coffee label as sensory guidelines for consumers.

**Brewing method**

Coffee beans were immediately ground and prepared by French Press brewing technique (150 mL of 93 °C water (Neptunas/Lithuania) was added to 16 g of coarse grind coffee, with extraction time: 4 minutes). Each sample after brewing was immediately analysed. All brewed coffee samples were prepared in duplicates.

**Headspace Volatile Compound Extraction by SPME**

Aroma compounds were extracted by solid phase microextraction (SPME). The coffee extract samples (5 mL) were transferred to 20 mL glass container. Extraction parameters: extraction temperature  $+50 \pm 1$  °C; incubation time: 4 minutes; extraction time: 7 minutes. In the research four different fibres were used: divinylbenzene/carboxen/ polydimethylsiloxane (DVB/CAR/PDMS), carboxen/ polydimethylsiloxane (CAR/PDMS), polydimethylsiloxane/ divinylbenzene (PDMS/DVB) and polyacrylate (PA). All fibres were purchased from Supelco (Bellefonte, PA, USA).

**HS-SPME-GC-MS chromatographic parameters**

'Perkin Elmer Clarus 500' chromatograph with mass spectrometer and 'Elite-Waw ETR' (60 mx 0,25mm internal diameter; DF 0.25 column) were used for gas chromatography method. Injection parameters: desorption time – 15 minutes; temperature  $+250$  °C. Working conditions were as follows: injector 250 °C; transfer line to MSD 260 °C; oven temperature start 40 °C, hold 7 min, programmed from 40 – 160 °C at 6 °C min<sup>-1</sup> hold 10 min, and from 160 – 210 °C at 10 °C min<sup>-1</sup>, hold 15 min. The column initial flow rate of 1 mL min<sup>-1</sup> was held using helium as carrier gas. The outlet split 1:2 and between 40 and 300 mass-to-charge ratios were scanned. As a quantitative measure, the share in the total GC peak area for each compound is given. The compounds were identified using mass spectral database 'Nist98' (Gloess *et al.*, 2013; Steen *et al.*, 2017).

**Statistical analysis**

The data were express as mean. Analysis of variance was performed, and the significant

differences were stated if  $p < 0.05$ . The data were analysed with Microsoft Office Excel 2013. Principal component analysis (PCA) was used for reduction of variables to a smaller set called components. The first principal component demonstrates the highest variability, followed by second component with less variability. PCA was performed including volatile profile of all tested coffees. Data were treated using SPSS 23 software.

**Results and Discussion****Evaluation of coffee volatile compound chemical groups, detected with different SPME fibres**

From 16 chemical compound groups, 76 compounds were detected (compounds are summarized by their chemical groups in Table 2). CAR/PDMS fibre extracted significantly more volatile compounds compared to other fibres and showed the highest peak areas in all chemical compound groups, except for phenols. CAR/PDMS has shown lack of extracting phenol compounds from ground coffee also in Akiyama *et al.* (2007) study. The phenol compounds as 4-vinylguaicol or 4-ethyl-2-methoxyphenol are not common compounds in specialty coffee aroma profile because of the smoky, spicy unpleasant aroma notes and increasing concentration is only detected in medium-dark roasted coffees (Dorfner *et al.*, 2003; Yang *et al.*, 2016). Roberts, Pollien, & Milo, (2000) research also reports CAR/PDMS fibre as the most suitable for small molecule extraction as 2-methylpropanal, acetaldehyde and organic acid as acetic acid and propionic acid extraction. The CAR has a large surface area with different range pores (micro- and macro-pores), which allows extracting also trace level volatile compound, compared to DVB with limited micropores (Chen, Chiang, & Chung, 2019; Salum *et al.*, 2017). The results also confirm this statement because using DVB/CAR/PDMS and CAR/PDMS fibre volatile compounds showed a higher peak area range compared to PDMS/DVB. DVB/CAR/PDMS fibre is the most commonly chosen fibre for aroma analysis of coffee brews. However, these studies used distilled water for coffee extraction (Caporaso *et al.*, 2018; Mestdagh *et al.*, 2014). It is known that distilled water cannot extract beneficial

Table 2

**Chemical compound groups extracted from coffee brews using four different SPME fibres**

No.	Chemical compound groups (number of compounds)	GC–MS peak area (number of compounds)			
		DVB/CAR/PDMS fibre	CAR/PDMS fibre	PDMS/DVB fibre	PA fibre
1.	Alcohols (7)		5.68E + 07 <sup>a</sup> (3)	2.89E + 07 <sup>c</sup> (3)	3.01E + 07 <sup>b</sup> (3)
2.	Aldehydes (11)	3.18E + 08 <sup>b</sup> (3)	9.61E + 08 <sup>a</sup> (8)	2.39E + 08 <sup>c</sup> (6)	3.07E + 07 (5)
3.	Alkanes (2)	6.37E + 06 <sup>b</sup> (1)	2.89E + 07 <sup>a</sup> (1)		
4.	Ketones (9)	6.71E + 07 <sup>b</sup> (4)	2.95E + 08 <sup>a</sup> (7)	4.17E + 07 (2)	4.32E + 05 <sup>c</sup> (1)
5.	Amides (1)		9.61E + 07 (1)		
6.	Esters (8)	7.09E + 07 <sup>b</sup> (5)	3.19E + 08 <sup>a</sup> (6)	1.53E + 07 <sup>c</sup> (1)	
7.	Furans (6)	4.09E + 08 <sup>b</sup> (5)	1.43E + 09 <sup>a</sup> (6)	3.97E + 08 <sup>c</sup> (4)	8.46E + 07 (3)
8.	Organic acids (10)	1.90E + 07 <sup>c</sup> (4)	3.07E + 08 <sup>a</sup> (3)	1.02E + 07 (5)	3.02E + 07 <sup>b</sup> (2)
9.	Oxides (1)	1.12E + 07 (1)	1.02E + 08 <sup>a</sup> (1)	1.69E + 07 <sup>c</sup> (1)	1.01E + 08 <sup>b</sup> (1)
10.	Phenols (3)		4.77E + 05 <sup>c</sup> (1)	3.50E + 06 <sup>b</sup> (1)	3.72E + 06 <sup>a</sup> (1)
11.	Pyrazines (9)	7.96E + 06 (1)	1.13E + 08 <sup>a</sup> (7)	4.10E + 07 <sup>b</sup> (6)	
12.	Pyridine (1)	2.37E + 07 <sup>b</sup> (1)	7.03E + 07 <sup>a</sup> (1)	2.47E + 06 (1)	
13.	Pyrimidine (2)	3.33E + 07 <sup>b</sup> (1)	8.07E + 07 <sup>a</sup> (1)	4.67E + 06 <sup>c</sup> (1)	5.10E + 05 (1)
14.	Pyrroles (2)	1.20E + 07 <sup>c</sup> (2)	5.21E + 07 <sup>a</sup> (2)	2.87E + 07 <sup>b</sup> (1)	
15.	Sulfur containing compounds (2)		1.77E + 07 <sup>a</sup> (1)	2.59E + 06 <sup>b</sup> (1)	
16.	Terpenes (2)		1.56E + 07 <sup>a</sup> (1)	3.41E + 06 <sup>b</sup> (2)	
Total compounds (76)		9.79E + 08 <sup>b</sup> (28)	3.94E + 09 <sup>a</sup> (50)	8.33E + 08 <sup>c</sup> (35)	2.81E + 08 (17)

Different letters (a–c) in the same row indicate significant differences ( $p < 0.05$ ) in GC–MS peak area.

aroma and flavour compounds because of the lack of minerals like calcium or magnesium (Hendon, Colonna–Dashwood, & Colonna–Dashwood, 2014). From this point of view, DVB/CAR/PDMS fibre suitability for coffee brew aroma extractions was not proven.

The PA fibre was able to extract only 17 volatile compounds from 8 chemical compound groups, showing the lowest total compound peak area, but the advantage of current fibre is the extraction of phenols with a higher peak area. It has been reported that fibre coatings with polar and non–polar materials are more efficient for polar compound extraction from samples with polar matrix composition. It could be one of the reasons why using PA fibre alkanes, amides, esters, pyrazines, pyridines, pyrroles, sulphur containing compounds and terpenes were not detected. Chen, Chiang, & Chung, (2019) study also confirmed that using PA fibre compounds as ketones, pyrazines, sulphur containing compounds were not detected. The DVB/CAR/PDMS, CAR/PDMS and PDMS/DVB fibres are bipolar and the results confirm their ability to extract wider composition of volatile compounds (Mestdagh *et al.*, 2014; Spietelun *et al.*, 2010).

*Important volatile compounds in specialty coffee brew*

It is noted that from approximately 950 coffee volatile compounds only 20 can significantly

influence the aroma and flavour profile. These 20 compounds are mostly detected in specialty coffees (Kim *et al.*, 2018; Laukaleja & Kruma, 2018). In this study, using CAR/PDMS fibre 17 important specialty coffee volatile compounds were detected: DVB/CAR/PDMS detected 15, PDMS/DVB detected 13 and PA fibre only detected 6 compounds. A higher peak area of volatile compounds was detected using CAR/PDMS and DVB/CAR/PDMS fibres (the results are illustrated in Table 3). Both fibres detected a significantly higher peak area for 2,6–dimethyl–4–thiopyrone (caramel aroma notes), 2–methylfuran (roasted coffee aroma notes). The CAR/PDMS fibre is suggested for detection of compounds with low boiling point, especially aldehydes (Salum *et al.*, 2017). In this research, the CAR/PDMS was the only fibre which could extract acetaldehyde (fruity aroma notes) and had a higher peak area for 3–methylbutanal (fruity), 2–methylpropanal (roasted, sweet almond aroma notes). The overall volatile compound profile in this research corroborates with Wei *et al.* (2017) aroma analysis using CAR/PDMS fibre; the furan and aldehyde compound peak areas were similar and proportional. Petisca *et al.* (2012) study reports supporting evidence that CAR/PDMS is the most efficient for furan compound extraction from coffee. The DVB/CAR/PDMS was the only

Table 3

Identified important volatile compounds in specialty coffee brews using four different SPME fibres

No.	Compounds	GC–MS peak area (x10 <sup>8</sup> )				Compound sensory description
		DVB/CAR/ PDMS	CAR/ PDMS	PDMS/ DVB	PA	
1.	1–(2–Furanylmethyl)–1H–pyrrole	19.64 <sup>b</sup>	48.81 <sup>a</sup>	7.08 <sup>c</sup>		savoury, vegetables
2.	2,3–Pentanedione	5.01				buttery
3.	2,6–Dimethyl–4–thiopyrone	56.01 <sup>b</sup>	127.43 <sup>a</sup>	33.73 <sup>c</sup>	5.01	caramel
4.	5–Methyl–2–furanaldehyde	35.85 <sup>b</sup>	73.31 <sup>a</sup>	20.29 <sup>c</sup>	10.35	caramel
5.	2–Furanmethanol	60.46 <sup>a</sup>		42.15 <sup>b</sup>	3.66 <sup>c</sup>	floral
6.	Furfuryl acetate	27.37 <sup>b</sup>	34.29 <sup>a</sup>	11.89 <sup>c</sup>	3.06	floral, fruity
7.	3–Methyl–butanal	7.14 <sup>b</sup>	12.39 <sup>a</sup>	6.57 <sup>c</sup>	0.86	fruity
8.	1–(2–Furanyl), ethanone	7.85 <sup>a</sup>	3.29 <sup>b</sup>			nutty
9.	2–(Methoxymethyl)furan	95.72 <sup>a</sup>	34.00 <sup>b</sup>	2.93		nutty, ground coffee–like
10.	2–Methylfuran	73.77 <sup>b</sup>	164.31 <sup>a</sup>	27.06 <sup>c</sup>	6.86	roasted almond, coffee
11.	Furfural	14.57 <sup>b</sup>	16.84 <sup>a</sup>	5.16 <sup>c</sup>		caramel
12.	2–Methylpropanal		3.98 <sup>a</sup>	3.28 <sup>b</sup>		roasted sweet almond
13.	2,5–Dimethylpyrazine	7.96 <sup>a</sup>	5.48 <sup>b</sup>	3.56 <sup>c</sup>		roasted
14.	Ethyl–pyrazine	16.63 <sup>b</sup>	32.29 <sup>a</sup>	9.35 <sup>c</sup>		chocolate–peanut, nutty
15.	4–Methyl–pyrimidine		18.20			popcorn
16.	Acetaldehyde		6.15			fruity
17.	Furfurylformate	19.64 <sup>b</sup>	48.81 <sup>a</sup>	7.08 <sup>c</sup>		malt, fruity

Different letters (a–c) in the same row indicate significant differences (p<0.05) in GC–MS peak area.

fibre which could detect 2,3-pentanedione (buttery aroma notes) in coffee brews and showed the highest peak area for 2–furanmethanol (floral aroma notes), 2–(methoxymethyl) furan (nutty, ground coffee like aroma notes), 2,5–dimethylpyrazine (roasted aroma notes) compounds.

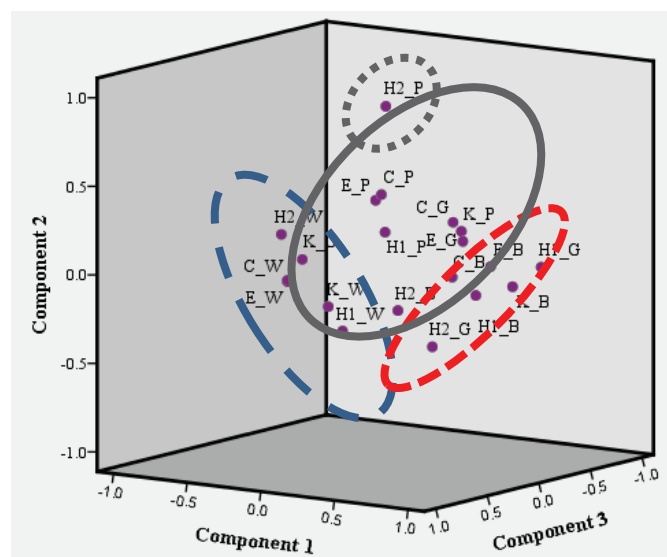


Figure 1. Component Plot in Rotated Space by principal component analysis (PCA). Code names: E\_W – Ethiopia PA fibre; C\_W – Columbia PA fibre; H1\_W – Honduras\_1 PA fibre; H2\_W – Honduras\_2 PA fibre; K\_W – Kenya PA fibre; E\_P – Ethiopia PDMS/DVB fibre; C\_P – Columbia PDMS/DVB fibre; H1\_P – Honduras\_1 PDMS/DVB fibre; H2\_P – Honduras\_2 PDMS/DVB fibre; K\_P – Kenya PDMS/DVB fibre; E\_B – Ethiopia CAR/PDMS fibre; C\_B – Columbia CAR/PDMS fibre; H1\_B – Honduras\_1 CAR/PDMS fibre; H2\_B – Honduras\_2 CAR/PDMS fibre; K\_B – Kenya CAR/PDMS fibre; E\_G – Ethiopia DVB/CAR/PDMS fibre; C\_G – Columbia DVB/CAR/PDMS fibre; H1\_G – Honduras\_1 DVB/CAR/PDMS fibre; H2\_G – Honduras\_2 DVB/CAR/PDMS fibre; K\_G – Kenya DVB/CAR/PDMS fibre.

This study result disproves Bressanello et al. (2017) and Gloess et al. (2013) researches; they reported a positive correlation between sensory (cupping) results and important coffee volatile compounds determined by HS-SPME using PDMS/DVB. Ribeiro et al. (2009) using PDMS/DVB fibre for prediction of sensory properties of Brazilian *Coffea arabica* L. roasted coffees concluded the importance of the method for future alternative coffee beverage quality monitoring.

*PCA of the specialty coffee brews with different fibres*

The Principal component analysis reduced variables in components and the first two components represented 83.84% of total variance. In the score plot, coffee samples extracted with Carboxen/PDMS (CAR/PDMS) and Divinylbenzene/Carboxen/PDMS (DVB/CAR/PDMS) were classified in one principal component showing the highest content of the most important coffee volatiles (Figure 1). In separate principal components, samples extracted with polyacrylate (PA) and Polydimethylsiloxane/Divinylbenzene (PDMS/DVB) fibre were classified. Due to the specific volatile profile, Honduras\_2 samples were classified differently – the sample extracted with Carboxen/PDMS was classified together with other coffee samples extracted with Polydimethylsiloxane/Divinylbenzene (PDMS/DVB), and the Honduras\_2 sample, extracted with Polydimethylsiloxane/Divinylbenzene (PDMS/DVB), was classified separately from all tested samples. The results can be explained by the applied processing method. Honduras\_2 was the only coffee sample with different processing method (anaerobic), while for the other four coffees natural processing method was applied. Natural process method allows the coffee cherries to dry in the sun, while in anaerobic

process coffee cherries are washed and coffee beans are afterwards sealed in fermentation tanks. During the anaerobic process coffee develops the fermented aroma and flavour notes (Geromel *et al.*, 2008; Taveira *et al.*, 2015).

The Principal component analysis allowed to reduce data of coffee volatiles in principal components, and results confirmed the importance of SPME fibre coating selection for analysis of coffee volatile profile, because samples were classified based on fibre coating, not the origin of coffees.

**Conclusions**

The results demonstrated that CAR/PDMS, compared to other fibres, was able to extract significantly more volatile compounds with higher peak areas in all chemical compound groups, except phenols. The CAR/PDMS was the only fibre which could detect all 17 important volatile compounds for specialty coffee. The PA fibre showed the largest phenol peak area but could only detect 6 from 17 important volatile compounds. This research disproves previous reports, showing that PDMS/DVB and DVB/CAR/PDMS fibres detect a wider and higher concentration of coffee volatile compounds compared to CAR/PDMS fibre. The result provides evidence for specialty coffee aroma profile analysis by SPME.

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