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The kinetics of coffee aroma extraction

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ABSTRACT

To obtain a cup of coffee with a balanced aroma, every step in the coffee production chain is crucial, including the final brew preparation, in order to achieve the optimal result in-cup. In this study, the role of the physico-chemical properties (volatility and polarity) of coffee odorants during extraction was investigated. The extraction kinetics of 20 key coffee odorants from a coffee bed were measured using quantitative in-cup aroma analysis. The kinetics differed depending on the coffee odorants' properties. The extraction speed could be correlated with the odorants' polarity. Higher polar components, such as 2,3-butanedione, were released much faster from the coffee bed as compared to lower polar ones such as β-damascenone. In contrast, the odorants' volatility did not seem to play a major role. Due to the different kinetics of extraction of the coffee odorants, the in-cup aroma balance changed continuously as more water passed through the coffee bed.

1. Introduction

A good cup of coffee is characterized by a subtle equilibrium in aroma, taste and mouthfeel. Several steps throughout the coffee production chain influence these sensory properties to a different extent. The overall quality of a cup of coffee depends largely on the quality of the raw material. It is the precursor composition (e.g. sugars, amino acids) which triggers the level of the odour- and taste active molecules formed during the roasting process. After harvesting of the cherries, the quality can merely be maintained and it is only by treating the coffee beans in the optimal way at every step along the value chain that one can avoid quality loss due to, e.g., degradation of precursors. The aim of roasting is to release all the aromatic potential of a specific green coffee origin. Over 1000 volatile compounds are formed, but only about 25–35 are considered as key odorants, responsible for the coffee flavour (Blank, Sen, & Grosch, 1992; Czerny, Mayer, & Grosch, 1999). After roasting, the coffee needs to be optimally stored in the absence of oxygen in order to avoid 1) the loss of aroma and 2) the oxidation of certain compounds giving rise to an off-note in the cup (Holscher & Steinhart, 1992). Finally it is by extracting the roast and ground coffee that the odorants and taste molecules are brought to the consumer's cup. Different brewing and extraction methods exist and for each of the methods various parameters play a role in delivering a brew with a balanced aroma. The coffee parameters such as roast profile and granulometry have to be optimised with respect to the extraction parameters such as pressure and coffee-to-water ratio, water minerality and temperature. Some studies have investigated the impact of extraction temperature and pressure on the final in-cup quality (Albanese, Matteo, Poiana, & Spagnamusso, 2009; Andueza et al., 2002, 2003; Caprioli et al., 2012), or alternatively different extraction methods or tools were compared (Gloess et al., 2013; Parenti et al., 2014). Overall, these studies show that every extraction method has its own characteristics and needs to be fine-tuned in accordance with all previous steps in the coffee production chain in order to bring the desired result in the cup. No balanced coffee can be created without excellent starting material, ideal roasting conditions and appropriate grinding. Because of this inter-relationship throughout the production chain, no universal, best extraction method exists and different studies indicate different 'optima'. To fine-tune extraction, the knowledge of the physico-chemistry governing the extraction phenomena is crucial. These fundamental mechanisms behind coffee extraction were, to our knowledge, however never investigated. Therefore, the objective of this study is to follow how key coffee odorants are extracted in-cup from a coffee bed by investigating the extraction kinetics using quantitative in-cup aroma analysis by SPME–GC–MS and employing stable isotope dilution analysis.

2. Materials and methods

2.1. Coffee sample

A commercially available portioned coffee (Nespresso capsule) was selected, which is developed to prepare a 110 mL lungo coffee. The

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capsule was filled with approximately 6.3 g of fresh roast and ground coffee.

2.2. Preparation of coffee beverage

Extraction was performed on a modified commercial coffee machine (TurMix, Modified Citiz TX270, Zürich, Switzerland), from which the pump was replaced with a pressure-programmable one, enabling an extraction of the capsule at a constant pressure of 20 bars. Acqua Panna water (Sanpellegrino S.p.a., Milano, Italy) was used and coffees with final volumes of approximately 10-, 20-, 40-, 80-, 110 and 150 mL were prepared. For each volume, an average of 3 extractions having similar extraction times and volumes was performed. The 3 extracts were pooled prior coffee odorant analysis.

2.3. Quantitative in-cup coffee odorant analysis

20 key coffee odorants were selected having a different volatility and polarity (Blank et al., 1992; Czerny et al., 1999). These were quantified in-cup by SPME–GC–MS using the stable isotope dilution analysis (SIDA). The amount is calculated in μg extracted into the cup and standardized towards the amount found at the end of the extraction experiment (150 mL). As the coffee odorants have a different concentration in-cup, appropriate dilutions need to be prepared. Moreover to obtain a good chromatographic separation with GC–MS, operating in single ion monitoring mode, the analysis was split into two groups of analytes (Table 1). Sample preparation was carried out in triplicate for each extraction volume. The overall variability of the analysis was less than 10%.

2.3.1. Group 1

Once extracted, the coffees were quickly cooled on ice and diluted with distilled water until a final volume of 150 mL. 100 mg of cysteine was added to the coffee samples to avoid thiol binding to the coffee matrix during sample work up. Definite amounts of isotope labelled standards were spiked into the water phase (Table 1) and the solutions were stirred for 10 min (at room temperature). Subsequently, portions of 7 mL were transferred to headspace vials.

After equilibration (40 °C, 1 min), the aroma compounds were extracted from the headspace during 10 min at 40 °C under agitation (350 rpm) using a divinylbenzene–carboxen–polydimethylsiloxane fibre (StableFlex DVB/CAR/PDMS; 2 cm; film thickness 50/30 μm; Supelco, Buchs, Switzerland). The extracted compounds were thermally desorbed for 3 min into a split/splitless injector maintained at 240 °C and operated in splitless mode. For separation of compounds, an Agilent 7890A gas chromatograph (Agilent Technologies, Morges, Switzerland) with a 60 m × 0.25 mm × 0.25 μm DB-Wax column was used (Agilent Technologies, Morges, Switzerland). Helium was used as a carrier gas with a constant flow of 1.3 mL/min, and the following temperature programme was applied: 40 °C (6 min), 4 °C/min, 140 °C (0 min), 20 °C/min, and 240 °C (10 min). The gas chromatograph was coupled to a 5975C mass spectrometer (Agilent Technologies, Morges, Switzerland) operating in single ion monitoring (SIM) mode using electron ionization and an ionization potential of 70 eV. All GC–MS measurements were run in triplicate. Data were analysed using the MassHunter Quant software (Version B.05.02, Agilent Technologies).

2.3.2. Group 2

After cooling the coffees on ice and diluting until a final volume of 150 mL, a 4:1 additional dilution was performed. Definite amounts of isotope labelled standards were spiked in-cup (Table 1) and the solutions were stirred for 10 min (at room temperature). Portions of 7 mL were transferred to headspace vials.

The coffee volatiles were extracted and injected into a Thermo Trace Ultra gas chromatograph (Brechbühler, Schlieren, Switzerland) as described above for Group 1. In contrast to Group 1, volatiles were separated on a 60 m × 0.25 mm × 1.4 μm DB-624 column (Agilent Technologies, Morges, Switzerland). Helium was used as a carrier gas with a constant flow of 1.3 mL/min, and the following temperature programme was applied: 40 °C (6 min), 6 °C/min, 140 °C (0 min), 20 °C/min, and 240 °C (10 min). The gas chromatograph was coupled to a Thermo Scientific ISQ mass spectrometer (Brechbühler, Schlieren, Switzerland) operating in single ion monitoring (SIM) mode using electron ionization and an ionization potential of 70 eV. All GC–MS measurements were run in triplicate. Data were analysed using the Xcalibur 2.1 software (Thermo Scientific).

Table 1
Key coffee odorants and labelled standards used for quantification.

Analyte	Aroma quality	Polarity log(Po/w)[a]	Volatility vapour pressure (mm Hg at 25 °C)[a]	m/z	Standard for quantification	m/z
Group 1						
2-Acetylpyrazine	Popcorn	0.10	0.17	122	2-Acetylpyrazine-[$^2H_5$]	127
Methanethiol	Sulfury, cabbage	0.93	1901	48	Methanethiol-[$^2H_3$]	51
Dimethyl sulfide	Sulfury, cabbage	0.98	647	62	Dimethyl sulfide-[$^2H_6$]	68
2-Ethyl-3,5-dimethylpyrazine (EDMP)	Earthy	1.46	0.81	135	EDMP-[$^2H_6$]	141
3-Mercapto-3-methylbutyl formate (MMBF)	Catty, sweaty	1.63	0.42	102	MMBF-[$^2H_6$]	108
2-Furfurylthiol	Roasty	1.73	4.0	114	2-Furfurylthiol-[$^2H_2$]	116
Hexanal	Green, grassy	1.93	11	72	Hexanal-[$^2H_4$]	76
Ethyl-2-methylbutanoate	Fruity	2.16	7.9	102	Ethyl-2-methylbutanoate-[$^2H_5$]	107
β-Damascenone	Cooked apple	3.10	0.02	190	β-Damascenone-[$^2H_4$]	194
Group 2						
2,3-Butanedione	Buttery	−1.34	57	43	2,3-Butanedione-[$^{13}C_4$]	45
2,3-Pentanedione	Buttery	−0.83	26	100	2,3−Pentanedione-[$^{13}C_2$]	102
Furfural	Bready	0.71	2.2	96	2-Methoxyphenol-[$^2H_3$]	127
Pyridine	Fishy	0.84	23	79	2-Methoxyphenol-[$^2H_3$]	127
2-Methylbutanal	Malty, cocoa	1.27	49	86	3-Methylbutanal-[$^2H_2$]	88
3-Methylbutanal	Malty	1.27	49	71	3-Methylbutanal-[$^2H_2$]	73
2-Methoxyphenol	Smoky, phenolic	1.34	0.18	124	2-Methoxyphenol-[$^2H_3$]	127
N-Methylpyrrole	Roasty	1.35	26	81	*N*-Methylpyrrole-[$^2H_3$]	84
Phenylacetaldehyde	Floral	1.76	0.37	120	Phenylacetaldehyde-[$^{13}C_2$]	122
4-Ethyl-2-methoxyphenol	Spicy, medicinal	2.43	0.017	152	4-Vinyl-2-methoxyphenol-[$^2H_3$]	153
4-Vinyl-2-methoxyphenol	Smoky, medicinal	2.57	0.019	150	4-Vinyl-2-methoxyphenol-[$^2H_3$]	153

[a] Values taken from SciFinder database (https://scifinder.cas.org/).