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AROMA IMPACT COMPOUNDS OF ARABICA AND ROBUSTA COFFEE. QUALITATIVE AND QUANTITATIVE INVESTIGATIONS

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Introduction

The volatile fraction of roasted coffee is extremely complex, consisting of more than 700 compounds [1] with a wide variety of functional groups.

During the last decade, efforts have been undertaken to evaluate those volatile compounds which contribute significantly to the aroma of roasted coffee. On the basis of the odor unit concept [2], *Tressl* [3] has suggested that 2-furfurylthiol is the most important odorant. In addition, he reported that the other compounds listed in Table 1 are of significance for the coffee flavor. Recently, *Holscher et al.* [4, 5], using gas chromatography/olfactometry (GC/O) of serial dilutions of the volatile fraction (aroma extract dilution analysis, AEDA [6]), confirmed that some of the odorants suggested by *Tressl* [3] are indeed intensely involved in the coffee flavor (Table 1). In addition, these authors identified further character impact compounds which are summarized in Table 1.

It is well-known [review in 7] that the two varieties of coffee, Arabica and Robusta, differ in their aromas. Several authors have compared the volatile fractions of the two varieties, but they have not evaluated the contribution of the identified compounds to the flavor differences of the two coffees. *Vitzthum et al.* [8] have recently reported, that 2-methylisoborneol resembles the typical earthy aroma impression of the Robusta coffee.

Table 1: Important odorants of the coffee flavor according to *Tressl* [3] and *Holscher et al.* [4, 5]

Compound	*Tressl* [3]	*Holscher et al.* [4, 5]
2-Furfurylthiol	+	+
2-Methyl-3-furanthiol	-	+
5-Methyl-2-furfurylthiol	+	-
3-Methyl-2-buten-1-thiol	-	+
Furfurylmethyldisulphide	+	-
3-Mercapto-3-methylbutylformate	-	+
3-Mercapto-3-methyl-1-butanol	-	+
Methional	-	+
Kahweofuran	+	-
Ethyldimethylpyrazine	+	+
Acetylpyrazine	+	-
Trimethylpyrazine	-	+
2-Methoxy-3-isobutylpyrazine	+	+
2-Methoxy-3-isopropylpyrazine	-	+
Linalool	-	+
Guaiacol	+	+
4-Vinylguaiacol	+	+
ß-Damascenone	-	+
Furaneol	+	+
2,3-Pentandione	+	+
2,3-Butandione (diacetyl)	-	+
(E)-2-Nonenal	+	-
2-/3-Methylbutanal	+	+
2-/3-Methylbutanoic acid	-	+
Acetic acid	-	+

The aim of the following study was to identify the potent odorants of Arabica and Robusta coffee (powder and brew) and to show the differences of these two varieties. The identification experiments were focussed on those compounds which were evaluated by AEDA as important odorants. As AEDA is only a screening method [9], some odorants, contributing to important notes within the odor profile of the two varieties, were quantified and the odor units were calculated on the basis of odor/taste threshold values in water.

Methods

The methods used for the analysis of the coffee aroma are summarized in Table 2. The volatiles were isolated by solvent extraction from both, the roasted ground powder and the brew. The aroma extract was separated from the non-volatile compounds by high vacuum transfer and the volatile fraction obtained was analysed by GC/O. The odorants were characterized by their retention index, odor quality and relative odor activity (FD-factor). The odorants with high FD-factors were identified and their odor threshold values were determined. Important odorants were quantified using stable isotope dilution methods and then compared on the basis of odor activity values calculated by dividing the concentration levels in coffee (powder and brew) through the flavor thresholds in water.

Table 2: Methods used for the analysis of coffee aroma

Method	Result
Solvent extraction, high vacuum transfer [10]	Aroma extract
Gas chromatography/olfactometry (GC/O [11])	Retention index and odor of the volatiles
Aroma extract dilution analysis (AEDA [6])	FD-Chromatogram (FD-factors: ranking of the volatiles on the basis of their odor units determined by GC/O)
Enrichment procedures (column chromatography, HPLC, preparative GC)	Pure odorants
Identification experiments (Capillary GC, MS, NMR)	Chemical structure
Sensory characterization [11]	Threshold values (in air, water)
Synthesis	Reference compounds, labeled compounds
Stable isotope dilution assay (SIDA [12]); calculation of odor units [2]	Quantitative data of some important odorants; their flavor significance expressed as odor units

Results and Discussion

Identification of important odorants

About 50-60 odorants were found in the GC-effluent of the aroma extracts of Arabica and Robusta coffee. As examplified for the Arabica coffee (roasted powder) the FD-chromatogram (Fig. 1) indicated 38 odorants with FD-factors ≥ 16. Most of the 38 odorants were identified on the basis of GC and MS data (footnote "d" in Table 3). Only the amounts of the compounds nos. 5, 21 and 37 were so low in the volatile fraction that no clear MS signals were obtained. The identification of these compounds as 2-methyl-3-furanthiol (no. 5), 2,3-diethyl-5-methylpyrazine (no. 21) and bis(2-methyl-3-furyl)disulphide (no. 37) was based only on comparison of GC and sensory data with that of the corresponding reference substances. Their sensory importance can be explained by the low threshold values (0.001-0.01 ng/l air) [13].

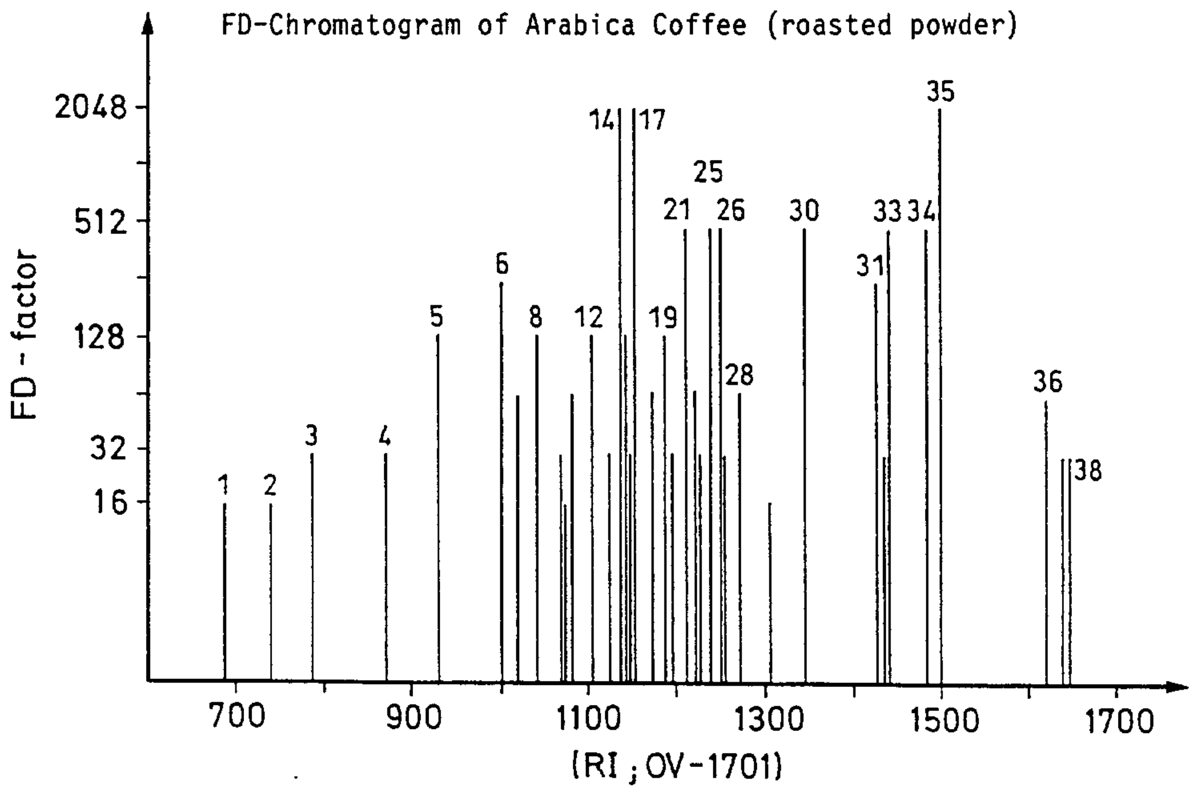

Figure 1. Flavor dilution chromatogram of the volatiles isolated from the roasted powder of Arabica coffee. Numbering of the flavor compounds as in Table 3; FD-factor: flavor dilution factor; RI: retention index.

Three odorants (nos. 14, 17 and 35) appeared with the highest FD-factor (FD = 2048) in the FD-chromatogram (Fig. 1). As shown in Table 3 they were identified as 3-mercapto-3-methylbutylformate (no. 14), 3,5-dimethyl-2-ethylpyrazine (no. 17) and (E)-ß-damascenone (no. 35). Their odor qualities were described as "catty", "earthy-roasty" and "honey-like", respectively.

Further odorants summarized in Table 3 were sotolon (no. 30), abhexon (no. 33), 4-methoxybenzaldehyde (no. 32) and bis(2-methyl-3-furyl)disulphide (no. 37). These compounds were identified for the first time in the coffee aroma. Sotolon and abhexon smelled "seasoning-like" and, in higher dilution, "caramel-like". Other volatiles contributing with "caramel-like, sweet" odor qualities to the aroma of coffee were furaneol (no. 24), 3,4-dimethyl-2-cyclopentenol-1-one (no. 22) and an unknown compound (no. 29).

The identification of such polar enoloxo compounds in low concentrations is difficult, since they are more or less adsorbed at the capillary during the GC-procedure [14]. It was observed that the lower the amount injected on the capillary the higher the proportion which was adsorbed. These effects were the smallest on the FFAP stationary phase which was, therefore, used for the AEDA of the enoloxo compounds.

Odorants with the impression "sweet" in combination with an additional odor quality were diacetyl (no. 1, buttery), 2,3-pentandione (no. 3, buttery), methional (no. 8, potato-like), 5H-5-methyl-6,7-dihydrocyclopentapyrazine (no. 27, nutty), p-anisaldehyde (no. 32, minty), bis(2-methyl-3-furyl)disulphide (no. 37, meaty) and vanillin (no. 38, vanilla-like).

About one third of the potent odorants were described as "roasty" in combination with an additional odor quality. 2-Furfurylthiol (no. 6, coffee-like), 3-mercapto-3-methylbutylformate (no. 14, catty), 2-methoxy-3-isopropylpyrazine (no. 15, earthy), 3,5-dimethyl-2-ethylpyrazine (no. 17, earthy), 2,3-diethyl-5-methylpyrazine (no. 21, earthy) and two unknown compounds (nos. 19 and 26, earthy) belonged to this group.

Important odorants with "honey-like" or "spicy" aroma qualities were (E)-ß-damascenone (no. 35), phenylacetaldehyde (no. 18) and guaiacol (no. 23), 4-ethyl- and 4-vinylguaiacol (nos. 31 and 34), respectively.

The compounds found in this study as important for the flavor of roasted coffee powder are in good agreement with those reported by *Holscher et al.* [4, 5] (Table 1). The presence of 2-furfurylthiol, ethyldimethylpyrazine, 2-methoxy-3-isobutylpyrazine, 4-vinylguaiacol, furaneol and 2,3-pentandione

Table 3: Important odorants of the roasted powder of Arabica coffee (FD ≥ 16)[a]

No.	Compound	Retention index[b]			Aroma quality[c]
		OV-1701	SE-54	FFAP	
1	2,3-Butandione (Diacetyl)[d]	686	580	990	buttery
2	3-Methylbutanal[d]	739	650	950	malty
3	2,3-Pentandione[d]	791	695	1060	buttery
4	3-Methyl-2-buten-1-thiol	874	821		amine-like
5	2-Methyl-3-furanthiol[e]	930	870		meaty
6	2-Furfurylthiol[d]	1004	913	1440	roasty (coffee)
7	2-/3-Methylbutanoic acid[d]	1022	860		sweaty
8	Methional[d]	1040	909	1455	potato-like, sweet
9	Unknown	1073		1365	fruity
10	2,4,5-Trimethylthiazole[d]	1074	997	1370	roasty, earthy
11	2,3,5-Trimethylpyrazine[d]	1078	1000	1395	roasty, earthy
12	Unknown	1107	1055		roasty, sulfury
13	3-Methyl-3-mercapto-1-butanol[d]	1127	972	1655	meaty (broth)
14	3-Methyl-3-mercapto-butylformate[d]	1138	1023	1517	catty, roasty
15	2-Methoxy-3-isopropylpyrazine[d]	1146	1097	1428	earthy, roasty
16	2,4-Dimethyl-5-ethylthiazole[d]	1149	1078	1435	earthy, roasty
17	3,5-Dimethyl-2-ethylpyrazine[d]	1154	1083	1453	earthy, roasty
18	Phenylacetaldehyde[d]	1178	1053	1635	honey-like
19	Unknown	1185	1103		roasty, earthy
20	Linalool[d]	1193	1102		flowery
21	2,3-Diethyl-5-methylpyrazine[e]	1218	1155	1485	earthy, roasty
22	3,4-Dimethyl-2-cyclopentenol-1-one[d]	1226	1075	1840	caramel-like
23	Guaiacol[d]	1228		1990	phenolic, spicy
24	2,5-Dimethyl-4-hydroxy-3(2H)-furanone (Furaneol)[d]	1235	1065	2035	caramel-like
25	2-Methoxy-3-isobutylpyrazine[d]	1237	1186	1520	earthy
26	Unknown	1254	1184		roasty, earthy
27	5H-5-Methyl-6,7-Dihydrocyclopentapyrazine[d]	1260	1145		roasty, sweet
28	(E)-2-Nonenal[d]	1275	1160		fatty
29	Unknown	1305		2090	caramel-like
30	4,5-Dimethyl-3-hydroxy-2(5H)-furanone (Sotolon)[d]	1347	1107	2200	seasoning-like
31	4-Ethylguaiacol[d]	1424	1287	2032	spicy
32	p-Anisaldehyde[d]	1431	1263	2030	sweet, minty
33	4-Methyl-5-ethyl-3-hydroxy-2(5H)-furanone (Abhexon)[d]	1433	1193	2270	seasoning-like
34	4-Vinylguaiacol[d]	1482	1323	2205	spicy
35	(E)-ß-Damascenone[d]	1502	1395	1815	honey-like, fruity
36	Unknown	1620		2355	amine-like
37	Bis(2-methyl-3-furyl)disulphide[e]	1640	1540	2150	meaty, sweet
38	Vanillin[d]	1645	1410	>2500	sweet (vanilla)

Footnotes of Table 3

a Numbering as in Fig. 1.

b RI: Retention index on the capillary [10].

c Odor quality perceived at the sniffing-port.

d The compound was identified by comparing it with the reference substance on the basis of the following criteria: MS/EI, MS/CI, RI data (on OV-1701, SE-54 and FFAP) as well as of the odor quality and threshold, which was perceived at the sniffing-port.

e The MS signals of the substance were too weak for an interpretation; the compound was only identified by comparing it with the reference substance on the basis of the resting criteria reported in footnote d.

also agrees with the suggestion of Tressl [3], that these odorants belong to the key compounds of the coffee flavor. In the capillary gas chromatograms of the coffee volatiles, *Holscher et al.* [5] have localized two additional odorants of unknown structure with high FD-factors. In the present study, they were identified as sotolon and abhexon.

Differences between the powders of roasted Arabica and Robusta coffee

The odorants of roasted Arabica and Robusta coffee powders were compared on the basis of their FD-factors. All of the odorants identified with FD-factors ≥ 16 contribute to the aroma of both coffee varieties. Linalool was an exception occurring only in Arabica coffee. According to the data summarized in Table 4 3,5-dimethyl-2-ethylpyrazine appeared with the highest FD-factor in both coffee varieties. However, differences were found in the concentration levels of some important coffee odorants.

2,3-Diethyl-5-methylpyrazine and 4-ethylguaiacol were predominant in the Robusta coffee and 3-mercapto-3-methylbutylformate, sotolon and abhexon in the Arabica coffee. Further significant differences were found for 2-methyl-3-furanthiol, phenylacetaldehyde, 3,4-dimethyl-2-cyclopentenol-1-one, 2-/3-methylbutanoic acid and linalool, all predominating in the Arabica coffee, and for 3-methyl-2-buten-1-thiol, which prevailed in the Robusta coffee.

The comparison of the coffee varieties indicated in addition that compounds causing "caramel-like, sweet-roasty" odor qualities were high in Arabica coffee, while those having "spicy" and "earthy-roasty" qualities contributed more significantly to those of the Robusta species. These

Table 4: Important odorants of roasted Arabica and Robusta coffee (powder and brew)

No.[a]	Compound	FD-factor$_{rel}$[b] Powder[c] Arabica	Robusta	Brew[d] Arabica	Robusta
17	3,5-Dimethyl-2-ethylpyrazine	100	100	50	100
35	(E)-ß-Damascenone	100	50	3	6
14	3-Mercapto-3-methylbutylformate	100	25	13	3
21	2,3-Diethyl-5-methylpyrazine	25	100	6	50
34	4-Vinylguaiacol	25	50	25	50
25	2-Methoxy-3-isobutylpyrazine	25	25	6	3
19	Unknown	25	25	6	25
30	Sotolon	25[e]	2[e]	100[e]	6[e]
33	Abhexon	25[e]	3[e]	50[e]	3[e]
31	4-Ethylguaiacol	13	50	25	50
26	Unknown	13	25	2	6
6	2-Furfurylthiol	13	13	3	3
8	Methional	6	3	25	13
15	2-Methoxy-3-isopropylpyrazine	6	3	2	3
5	2-Methyl-3-furanthiol	6[f]	2[f]	<1[f]	<1[f]
11	2,3,5-Trimethylpyrazine	3	2	2	3
28	(E)-2-Nonenal	3	2	<1	<1
36	Unknown	3	2	3	6
18	Phenylacetaldehyde	3	1	2	1
22	3,4-Dimethyl-2-cyclopentenol-1-one	3[e]	<1[e]	6[e]	2[e]
7	2-/3-Methylbutanoic acid	3[e]	<1[e]	3[e]	<1[e]
20	Linalool	3[e]	-	<1	-
5	3-Methyl-2-buten-1-thiol	2	6	<1	<1
27	5H-5-Methyl-6,7-dihydrocyclopenta-pyrazine	2	3	<1	<1
23	Guaiacol	2[e]	3[e]	1[e]	3[e]
13	3-Mercapto-3-methyl-1-butanol	2[e]	<1[e]	3[e]	2[e]
38	Vanillin	2	2	25[e]	13[e]
37	Bis(2-methyl-3-furyl)disulphide	2	2	6	3
24	Furaneol	1[e]	<1[e]	13[e]	3[e]

[a] Numbering as in Fig. 1 and Table 3.

[b] The FD-factor (OV-1701) of each compound (Table 3) was related to the compound with highest FD-factor which was set to 100.

[c] Both coffee varieties were roasted 3 min using a jetzone roaster, particle size of the coffee powder: 300-500 µm.

[d] The brew was obtained by extracting 54 g of the powder with 1 L of hot water (80-100°C).

[e] The FD-factor was determined on FFAP.

[f] The FD-factor was determined on SE-54.

differences in the composition of the important odorants corresponded to the differences in the overall aromas of the two varieties.

Differences between the brews of Arabica and Robusta coffee

A comparison of the odorants isolated from the brews of Arabica and Robusta coffee (Table 4) revealed a shift in the predominating flavor compounds. Sotolon, abhexon, furaneol and 3,4-dimethyl-2-cyclopentenol-1-one showed significant higher FD-factors in the Arabica than in the Robusta coffee. This difference suggested that these odorants were mainly responsible for the "sweet, mild" aroma of the Arabica coffee.

During the extraction with hot water the water-soluble enoloxo compounds were enriched in the brew and, thus, enhanced the caramel-like flavor notes, in particular of the Arabica coffee. *Hodge* [15] has reported that a planar enoloxo structural element in a volatile compound is responsible for the caramel-like odor impression.

The aroma of the Robusta coffee brew was mainly influenced by compounds having "roasty-earthy" and "spicy" odor qualities like 2,3-diethyl-5-methylpyrazine, 3,5-dimethyl-2-ethylpyrazine, 4-ethylguaiacol, 4-vinylguaiacol and the odorant no. 19. These odorants, in combination with the lower amounts of compounds having caramel-like aromas, were responsible for the "harsh, earthy, less pleasant" flavor notes of Robusta coffee.

Compared to the powders, vanillin, methional, furaneol and sotolon increased in the brews, in particular of the Arabica coffee. On the other hand (E)-ß-damascenone, (E)-2-nonenal, the temperature labile thiols 3-mercapto-3-methylbutylformate, 2-furfurylthiol, 2-methyl-3-furanthiol, 3-methyl-2-buten-1-thiol, and also linalool decreased strongly. This effect was also observed for 2,3-diethyl-5-methylpyrazine and 2-methoxy-3-isobutylpyrazine, which also decreased especially in the Arabica coffee.

Quantitative data

The quantitative analysis of the compounds were performed by means of a stable isotope dilution analysis (SIDA) in order to compensate for losses during the isolation procedure [12]. In the SIDA the odorant labeled with a stable isotope is used as internal standard. Until now a SIDA was developed for the quantification of furaneol, diacetyl [16], 2,3-pentandione, 4-

Table 5: Concentrations of some important odorants in the brews of Arabica and Robusta coffees

Compound[a]	Arabica	Robusta
	Concentration[b]	
Diacetyl ($^{13}C_2$)	1.7	1.3
2,3-Pentandione (d_3)	1.3	0.7
Furaneol ($^{13}C_2$)	6.6	1.5
Sotolon[c]	1.0	0.2
Abhexon (d_3)	0.1	<0.03
4-Ethylguaiacol (d_2)	0.06	0.4
4-Vinylguaiacol[d]	1.0	n.d.
3-Mercapto-3-methylbutylformate (d_6)	0.006	0.002
(E)-ß-Damascenone (d_6)	n.d.	0.003

[a] The quantification was performed as stable isotope dilution assay. The labeling of the internal standard with the stable isotope is given in bracketts (^{13}C: carbon-13, d: deuterium).

[b] Concentration: mg/l brew prepared from 54 g roasted coffee powder.

[c] Sotolon was determined using d_3-abhexon as internal standard.

[d] 4-Vinylguaiacol was determined using d_2-4-ethylguaiacol as internal standard.

n.d.: not determined.

Tabel 6. Odor activity values (OAV) of important flavor compounds in the brews of Arabica and Robusta coffees

Compound	Threshold[a]	Arabica OAV	Robusta OAV
Diacetyl	15[b]	113	87
2,3-Pentandione	30[b]	43	24
Furaneol	100[b]	66	15
	30[c]	220	50

[a] µg/kg water.

[b] Odor threshold (nasal perception).

[c] Flavor threshold (retronasal perception).

ethylguaiacol, abhexon, (E)-ß-damascenone and 3-mercapto-3-methylbutylformate in the brews of both, Arabica and Robusta coffees.

As shown in Table 5, the amounts of diacetyl and 2,3-pentandione were higher in Arabica than in Robusta coffee brew indicating the importance of the buttery top-notes for the mild aroma of Arabica coffee. This becomes more clear when comparing the odor activity values (OAV, Table 6). The higher OAV of diacetyl indicated that this dione contributed more significantly to the aroma of the brews than 2,3-pentandione.

Quantitative measurements of furaneol which was used as indicator substance for the caramel-like odorants revealed one reason for the odor difference between Arabica and Robusta. The concentration of furaneol was 4.5 fold higher in the brew of Arabica coffee than in the corresponding sample of the Robusta species (Table 5). Calculation of OAV (Table 6) confirmed the stronger effect of furaneol on the flavor of Arabica coffee compared to the Robusta variety. The concentrations of sotolon and abhexon were lower than those of furaneol, but also these enoloxo compounds prevailed in the Arabica coffee (Table 5).

The OAV of diacetyl in Arabica coffee brew was 2-fold higher than the OAV of furaneol. In contrast, the FD-factor of furaneol was 8-fold higher. This difference indicates the great losses of diacetyl during the isolation procedure compared to the higher boiling compounds like furaneol. Thus, the importance of high volatile compounds was underestimated by the AEDA.

The results in Table 5 show, furthermore, the significantly higher concentration of 4-ethylguaiacol in the Robusta coffee compared to the Arabica coffee. This agreed with the sensory data obtained by the AEDA indicating the importance of phenol-derivatives for the aroma of Robusta coffee.

The predominance of 3-mercapto-3-methylbutylformate in the Arabica coffee was also in agreement with the results of the AEDA.

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Summary

The volatile components of roasted Arabica and Robusta coffees (powder and brew) were analysed by gas chromatography-olfactometry (GC/O) which revealed the odorants having the highest odor-activity values (ratio of concentration to odor threshold). This procedure resulted in 38 odorants of which 32 were identified. The powders of the two coffee varieties differed in the concentration levels of these compounds.

The results indicate that the flavor difference between Arabica and Robusta coffee (powder and brew) are mainly due to the predominance of enoloxo compounds (sotolon, abhexon, furaneol, 3,4-dimethylcyclopentenol-1-one) in the former and of 3,5-dimethyl-2-ethylpyrazine, 2,3-diethyl-5-methylpyrazine, 4-ethylguaiacol and 4-vinylguaiacol in the latter. Preparation of brews enhanced the flavor difference, as the concentration levels of water-soluble odorants (furaneol, sotolon, abhexon) responsible for the "sweet-caramel" flavour note increased more in the Arabica than in the Robusta coffee. On the other hand, the alkylpyrazines and guaiacols were responsible for the "spicy, harsh-earthy" aroma of the Robusta coffee.

Quantification of selected odorants using a stable isotope dilution assay confirmed the differences between the Arabica and Robusta coffees (brew) found by GC/O.

Zusammenfassung

Die flüchtige Fraktion von Arabica- und Robusta-Röstkaffee (Pulver und Getränk) wurde durch Gaschromatographie-Olfaktometrie (GC/O) untersucht. Die Analyse ergab 38 aromaaktive Verbindungen, von denen 32 identifiziert wurden. Die beiden Kaffeesorten zeigten Unterschiede in der Konzentration dieser Aromastoffe.

Das Aroma des Arabica-Kaffees wird hauptsächlich durch Enoloxo-Verbindungen (Sotolon, Abhexon, Furaneol und 3,4-Dimethyl-2-cyclopentenol-1-on) geprägt, während im Robusta-Kaffee 3,5-Dimethyl-2-ethylpyrazin, 2,3-Diethyl-5-methylpyrazin, 4-Ethyl- und 4-Vinylguajacol überwiegen. Die Bedeutung der Enoloxo-Verbindungen nimmt im Arabica-Kaffeegetränk wegen der guten Wasserlöslichkeit zu, so daß sie für das süßlich-karamelartige Aroma verantwortlich sind. Die stechend-erdige Aromanote von Robusta-Kaffee wird dagegen von Alkylpyrazinen und Guajacol-Derivaten geprägt.

Quantitative Daten bestätigten die durch GC/O erhaltenen Ergebnisse in bezug auf die Unterschiede von Arabica- und Robusta-Kaffee (Getränk).

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