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Aroma compound analysis of *Piper nigrum* and *Piper guineense* essential oils from Cameroon using solid-phase microextraction–gas chromatography, solid-phase microextraction–gas chromatography–mass spectrometry and olfactometry

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Abstract

The investigation of aroma compounds of the essential oils of dried fruits of black pepper (*Piper nigrum*) and black and white ‘‘Ashanti pepper’’ (*Piper guineense*) from Cameroon by means of solid-phase microextraction (SPME) was carried out for the first time to identify the odorous target components responsible for the characteristic odor of these valuable spices and food flavoring products. By means of GC–flame ionization detection (FID) and GC–MS (using different polar columns) the main compounds (concentration >3.0%, calculated as % peak area of GC–FID analysis using a non-polar fused-silica open tubular RSL-200 column) of the SPME headspace samples of *P. nigrum* (black) and *P. guineense* (black and white) were found to be: *P. nigrum* (black)—germacrene D (11.01%), limonene (10.26%), β-pinene (10.02%), α-phellandrene (8.56%), β-caryophyllene (7.29%), α-pinene (6.40%) and *cis*-β-ocimene (3.19%); *P. guineense* (black)—β-caryophyllene (57.59%), β-elemene (5.10%), bicyclogermacrene (5.05%) and α-humulene (4.86%); and *P. guineense* (white)—β-caryophyllene (51.75%), *cis*-β-ocimene (6.61%), limonene (5.88%), β-pinene (4.56%), linalool (3.97%) and α-humulene (3.29%). The most intense odor impressions of the essential oils of the various dried pepper fruits were given by professional perfumers as follows: *P. nigrum* (black)—fine, pleasant black pepper note; *P. guineense* (black)—black pepper top-note; and *P. guineense* (white)—pleasant white pepper note. These analytical results for the SPME headspace samples of three different pepper species from Cameroon are in accordance with the olfactoric data of the corresponding essential oils. A GC–sniffing technique was used to correlate the single odor impression of the identified SPME headspace volatiles of the three investigated pepper samples with the following results: the main compounds such as β-caryophyllene, germacrene D, limonene, β-pinene, α-phellandrene and α-humulene, as well as minor constituents such as δ-carene, β-phellandrene, isoborneol, α-guaiene, sarisan, elemicin, calamenene, caryophyllene alcohol, isoelemicin, T-muurolol, cubenol and bulnesol, are of greatest importance for the characteristic pepper odor notes of these three *Piper* samples. Further aroma impressions can be attributed to mono- and sesquiterpenes, hexane, octane and nonane derivatives.

Keywords: *Piper nigrum*; *Piper guineense*; Essential oils; Food analysis; Plant materials; Aroma compounds

1. Introduction

Many essential oils of black pepper (*Piper nigrum*,

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Piperaceae family) seeds and leaves from various geographic origins have been analyzed for their aroma components, and more than 250 volatiles have been identified in this valuable spice and flavoring material for food and perfumery products [1–5]. Using olfactoric evaluations (e.g. aroma extract dilution analysis or the sniffing technique) in combination with gas chromatographic analyses [6–9] the flavor and off-flavor compounds were found to be monoterpenes, minor aldehydes and pyrazines.

In contrast to the well-known composition of *P. nigrum* essential oils, the volatiles of the West African pepper (*Piper guineense* Schum. and Thom., syn. *Piper leonense* C.DC., *Piper famechonii* C.DC.; *Piperaceae* family; so-called ''Ashanti pepper'') have been investigated very rarely [10–14]. Monoterpenes, sesquiterpenes and benzenoids (e.g. dillapiole and myristicin) were identified as the main compounds. In addition to the importance of ''Ashanti pepper'' as a spice and for the flavoring of food products, this *Piper* species is also used in traditional African medicine, because of its various pharmacological effects (e.g. antimicrobial, insecticidal, anticonvulsive, antihypertensive, sedative and tranquillizing activities [15–19]).

An investigation of the aroma compounds of the essential oils of dried fruits of black *P. nigrum* and black and white *P. guineense* from Cameroon by means of solid-phase microextraction (SPME), GC, GC–MS, the GC–sniffing technique and olfactometry (odor evaluations) has not been performed previously. Therefore, the objective of this study was to identify the aroma impact compounds of these SPME samples responsible for the characteristic odor of these natural products using the very effective combination of analytical and olfactoric methods given above [20–28].

2. Experimental

2.1. Plant material

The plant materials (black *Piper nigrum*, black and white *Piper guineense* seeds) were collected from a local market at Douala (Cameroon) in November 1998. The species were identified by a local botanist, and the specimens deposited at the National Herbarium in Yaounde. The seeds were dried naturally at room temperature for 1 month before hydrodistillation.

2.2. Isolation of volatile compounds

The three different pepper seed samples (20 g each) were powdered and subjected to hydrodistillation in a Clevenger-type apparatus for 4 h. The yields (v/w) of the samples were 1.1% (black pepper), 0.9% (black ''Ashanti pepper'') and 1.4% (white ''Ashanti pepper''). The obtained essential oils were dried over anhydrous sodium sulfate and stored at 4 °C until analysis.

2.3. Olfactoric evaluation

The essential oils of the three *Piper* species were evaluated olfactorically by professional perfumers and the aromas of the samples were described as:

(i) fine, pleasant black pepper note with intense pinene- and limonene side-notes (black *P. nigrum*);

(ii) black pepper top-note, nutmeg- and clover-like notes, fruity and herbal/vegetable-like side-notes, in the background fatty-oily (black *P. guineense*); and

(iii) pleasant white pepper note, spicy (direction of clove) and fresh-floral-fruity side-notes (white *P. guineense*).

2.4. Sample preparation

For SPME sampling, each of the three essential oils was placed in a 10 mL glass vial and the vial closed by a septum. The SPME headspace volatiles were collected using a Supelco No. 57348, 2 cm× 50/30 μm divinylbenzene–Carboxen–polydimethylsiloxane Stable-Flex fiber for 4 h. After sampling, the SPME device was placed in the injector of the GC and the GC–MS instruments.

2.5. GC conditions

Gas chromatographic analyses were performed with a Shimadzu GC-14A instrument with flame ionization detection (FID) and a Shimadzu Chromatopac C-R6A integrator, and with a Varian

GC-3700 with FID and a Shimadzu Chromatopac C-R1B integrator (all Shimadzu, Koyoto, Japan). Compounds were separated on 30 m×0.32 mm I.D. fused-silica columns coated either with a 0.25 μm film bonded non-polar fused-silica open tubular RSL-200 (Bio-Rad, Munich, Germany), HP-5MS (Hewlett-Packard, Palo Alto, CA, USA) or with a 0.50 μm film of bonded polar Stabilwax (Restek, Bellefonte, PA, USA). The non-polar column was maintained at 40 °C for 5 min after injection, then programmed at 6 °C min^{-1} to 280 °C, which was maintained for 5 min. Split injection was conducted with a split ratio of 1:20 and 1:50 for the non-polar and polar columns, respectively; hydrogen was used as carrier gas at 2.5, 2.5 and 3.5 kPa. For all columns the injector temperature was 250 °C and the detector temperature 320 °C. Quantification by % peak area calculations was performed using the non-polar FSOT RSL-200 column. Some individual components could be identified by co-injection of pure compounds and comparison of their retention times (as Kovats indices) with published and own data [29–34].

2.6. GC–sniffing technique conditions

Gas chromatography–olfactory analysis (''sniffing technique'') was performed with a Fractovap 2101 GC system equipped with a splitting system, a model 230 LT-Programmer, a model 160 electrometer (Carlo Erba, Milan, Italy), and a Kompensograph-III-Recorder (Siemens, Munich, Germany). Compounds were separated on a 30 m×0.32 mm I.D. fused-silica column coated with a 0.25 μm film of non-polar FSOT RSL-200 (Bio-Rad). The column was maintained 40 °C for 5 min after injection, then programmed at 8 °C min^{-1} to 230 °C, which was maintained for 20 min. Compounds were injected in splitless mode with hydrogen as carrier gas (pressure, 1.8 kPa; column flow, 2 mL min^{-1}). The injector temperature was 250 °C, the detection (FID) temperature was 320 °C, and the sniffing capillary temperature 250 °C. The column eluate sniffing split ratio was 1:50, FID/nose. Peak to odor-impression correlations were performed by two professional perfumers and three fragrance chemists. The olfactoric data obtained were also correlated with aroma descriptions of volatiles published elsewhere [35–41].

2.7. GC–MS conditions

GC–MS was performed with a Shimadzu GC-17 gas chromatograph coupled to a Shimadzu QP5000 mass spectrometer (Compaq-Pro Linea data system, class5k software) and a GC-17A system coupled to a QP5050 system (Pentium-II data system, class5k software). The columns (FSOT RSL-200 and Stabilwax) and temperature programmes used were as for GC analysis. Split injection was performed with helium as carrier gas. For the non-polar column the split ratio was 1:50, the column head pressure 4.9 kPa, and the flow-rate 0.5 mL min^{-1}; for the polar column the head pressure was 105 kPa, and the flow-rate 1.0 mL min^{-1}. Injector, interface, and ion-source temperatures were 250, 300 and 200 °C, respectively. The spectrometers were operated in electron-impact (EI) mode; the scan range was 41–450 u. Compounds were identified by use of National Institute of Standards and Technology (NIST), Wiley, US National Bureau of Standards (NBS) and own mass spectra libraries on-line as well as literature MS data [30,31,42,43] off-line.

3. Results and discussion

The essential oils of black *Piper nigrum* and black and white *Piper guineense* dried seeds were evaluated olfactorically by professional perfumers for their characteristic aroma as follows: fine, pleasant black pepper note with intense pinene and limonene side-notes (*P. nigrum*); black pepper top-note, nutmeg- and clover-like notes, fruity and herbal/vegetable-like side-notes, in the background fatty-oily (*P. guineense*, black); and pleasant white pepper note, spicy (direction of clove) and fresh-floral-fruity side-notes (*P. guineense*, white). The intensity of the odor impressions was highest in the case of black pepper and lowest in the case of black ''Ashanti pepper''.

SPME, a powerful method for the characterisation of such aroma systems [23–28], was used for trapping of the aroma compounds responsible for the significant flavor of the three different *Piper* samples. Analyses of the volatiles were performed by means of GC–FID and GC–MS using different polar columns as well as the GC–sniffing technique to identify the headspace compounds by their retention times (Kovats indices), their mass spectra and their single aroma impressions.

In total, 100 constituents of the three investigated pepper headspace SPME samples could be identified among the more than 130 components detected by GC–FID and GC–MS (Kovats indices and mass spectral correlations; see Table 1 and Fig. 1). As main compounds (concentration >3.0%, calculated as % peak area using a non-polar FSOT RSL-200 column; mean values of all GC–FID analyses using this column) we found: black pepper—germacrene D (11.01%), limonene (10.26%), β-pinene (10.02%), α-phellandrene (8.56%), β-caryophyllene (7.29%), α-pinene (6.40%) and *cis*-β-ocimene (3.19%); black ''Ashanti pepper''—β-caryophyllene (57.59%), β-elemene (5.10%), bicyclogermacrene (5.05%) and α-humulene (4.86%); and white ''Ashanti pepper''—β-caryophyllene (51.75%), *cis*-β-ocimene (6.61%), limonene (5.88%), β-pinene (4.56%), linalool (3.97%) and α-humulene (3.29%).

GC–olfactometry (''GC–sniffing technique'') was used to correlate the single aroma impression from the GC eluate with the gas chromatographic (retention times, Kovats indices) and spectroscopic (mass spectra) data (identical non-polar FSOT RSL-200 column used for GC–FID, GC–MS and the GC–sniffing technique). These combined chromatographic–olfactoric correlations are in significant accordance with the published odor attributes for each identified compound of all three investigated pepper headspace SPME samples (see Table 2) [34–39] and allow the following statement: for the black pepper aroma impression of the black pepper and black ''Ashanti pepper'' as well as partly for the white pepper notes of *P. guineense* (white species) the following volatiles can be said to be responsible: sabinene, β-pinene, α-phellandrene, δ-carene, β-phellandrene, limonene, isoborneol, β-caryophyllene, α-guaiene, α-humulene, sarisan, germacrene D, elemicin, calamenene, caryophyllene alcohol, iso-elemicin, T-muurolol, cubenol and bulnesol. The limonene and pinene notes of *Piper nigrum* are the result of the identified limonene as well as α- and β-pinene in higher concentrations in this headspace SPME sample. Nutmeg and clove odor notes (black and white ''Ashanti pepper'' headspace) can be correlated with terpinen-4-ol, eugenol, methyleugenol and β-caryophyllene, and fruity (black and white *P.* guineense) notes with α-terpinene, limonene, γ-terpinene and *p*-cymen-8-ol. Herbal/vegetable-like odor impressions (black *P. guineense*) were found to correlate with α-thujene, *cis*-β-ocimene, *trans*-β-ocimene, α-cubebene, α-copaene, β-cubebene, dodecanal, γ-elemene, γ-muurolene and spathulenol, while the floral aroma (white ''Ashanti pepper'') can be correlated with linalool, α-terpineol, citronellol, decanol, *trans*-nerolidol, α-bisabolol and hexadecanol. Nonanal, *trans*-2-nonenal, nonanol, octanoic acid and nonanoic acid are responsible for the fatty-oily aroma of the black ''Ashanti pepper'', and *cis*-3-hexen-1-ol, camphene, *p*-cymene, limonene, γ-terpinene, camphor, isoborneol, borneol, *p*-cymen-8-ol, carveol, piperitone and *trans*,*trans*-α-farnesol for the fresh odor of the white ''Ashanti pepper'' sample.

The analytical and olfactoric results for the aroma compound investigation of a *Piper nigrum* essential oil from Cameroon using SPME is somewhat surprising because of the relatively high content of the sesquiterpene hydrocarbon germacrene D (11.01%) with known pepper odor notes and, on the other hand, a medium concentration only of β-caryophyllene, which is normally the main compound with a sesquiterpenic structure in many essential *P. nigrum* oils of various origin [1–5,9]. Additionally, the lack of butane derivatives with an intense black pepper aroma [6–8] with little influence on the pleasant peppery note of this essential oil sample from Cameroon, demonstrated by olfactoric evaluations and GC–sniffing analyses, is remarkable.

In contrast, such a high content of β-caryophyllene in the headspace SPME of black and white *Piper guineense* essential oils from Cameroon has not been found in any ''Ashanti pepper'' essential oil until now [12,14,15]. Therefore, the typical pepper aroma of both samples can be attributed to this sesquiterpene hydrocarbon.

4. Conclusion

In summary, we can report that the significant aroma of essential oils of *Piper nigrum* and of black

Table 1
Headspace SPME volatiles of *P. nigrum* (black) and *P. guneense* (black and white) from Cameroon (concentrations calculated as % peak area of GC–FID using a non-polar FSOT RSL-200 column; mean values of all analyses using this column)

No.	Compound	*I*[a]	Pnb[b]	Pgb[c]	Pgw[d]
01	Acetaldehyde	427	0.01	tr[e]	nd[f]
02	Acetic acid	603	nd	nd	0.01
03	Propanoic acid	668	nd	nd	tr
04	Hexanal	775	nd	tr	nd
05	Butanoic acid	779	tr	tr	tr
06	*trans*-2-Hexenal	830	nd	tr	nd
07	*cis*-3-Hexen-1-ol	849	tr	tr	nd
08	*trans*-2-Hexen-1-ol	853	tr	nd	nd
09	Hexanol	859	nd	tr	nd
10	Heptan-3-ol	877	tr	nd	nd
11	α-Thujene	938	tr	0.02	0.07
12	α-Pinene	943	6.40	1.04	0.31
13	Camphene	955	tr	tr	0.11
14	Sabinene	975	2.46	2.20	0.76
15	β-Pinene	981	10.02	2.55	4.56
16	Myrcene	986	1.38	0.14	0.21
17	α-Phellandrene	998	8.56	2.61	1.18
18	δ-3-Carene	1009	2.78	0.98	1.06
19	α-Terpinene	1012	tr	tr	tr
20	β-Phellandrene	1015	0.41	0.21	0.13
21	*cis*-β-Ocimene	1018	3.19	0.77	6.61
22	*p*-Cymene	1021	2.14	0.35	0.23
23	Limonene	1027	10.26	2.01	5.88
24	*trans*-β-Ocimene	1038	1.33	0.41	0.29
25	γ-Terpinene	1052	tr	0.26	0.09
26	2,5-Dimethyl-3-methoxypyrazine	1065	nd	nd	tr
27	*cis*-Sabinene hydrate	1073	nd	tr	0.10
28	Terpinolene	1078	1.32	0.38	0.42
29	Nonanal	1081	tr	0.45	0.02
30	Linalool	1093	2.51	1.44	3.97
31	*trans*-Sabinene hydrate	1110	tr	tr	tr
32	*trans*-2-Nonenal	1134	1.08	tr	nd
33	Camphor	1139	nd	nd	0.16
34	Nonanol	1154	nd	0.24	tr
35	Isoborneol	1158	tr	tr	0.05
36	Borneol	1162	0.21	0.35	0.13
37	*p*-Cymen-8-ol	1166	0.14	0.17	0.10
38	Octanoic acid	1169	0.21	0.44	tr
39	Terpinen-4-ol	1173	1.61	0.81	0.65
40	α-Terpineol	1179	1.59	0.32	0.39
41	Carveol	1197	0.27	0.23	0.09
42	Cumin aldehyde	1224	0.01	tr	0.42
43	Citronellol	1228	0.11	0.05	tr
44	Carvone	1231	0.16	0.24	0.11
45	Piperitone	1245	tr	tr	0.20
46	Decanol	1263	1.04	0.16	0.32
47	Nonanoic acid	1275	0.06	0.21	tr
48	Safrole	1280	0.52	0.29	2.98
49	Cuminyl alcohol	1284	0.07	0.02	0.21

Table 1. Continued

No.	Compound	I [a]	Pnb [b]	Pgb [c]	Pgw [d]
50	δ-Elemene	1340	tr	0.41	0.16
51	Eugenol	1348	0.07	0.09	0.06
52	α-Cubebene	1351	0.13	0.31	0.58
53	α-Copaene	1377	1.41	1.08	1.45
54	β-Cubebene	1388	tr	tr	nd
55	β-Elemene	1391	2.55	5.10	1.72
56	Dodecanal	1396	0.18	0.24	tr
57	Methyleugenol	1407	0.92	0.11	0.22
58	α-Gurjunene	1412	0.32	0.28	0.24
59	β-Caryophyllene	1418	7.29	57.59	51.75
60	γ-Elemene	1425	0.06	0.14	0.39
61	*trans*-α-Bergamotene	1431	0.14	0.20	0.08
62	*trans*-β-Farnesene	1445	0.41	0.46	0.37
63	α-Guaiene	1453	0.25	0.27	0.81
64	α-Humulene	1467	0.58	4.86	3.29
65	γ-Muurolene	1475	nd	tr	nd
66	Sarisan	1479	0.36	0.49	0.56
67	β-Guaiene	1483	0.17	0.09	0.32
68	Germacrene D	1487	11.01	0.14	1.34
69	α-Zingiberene	1494	tr	tr	tr
70	β-Bisabolene	1498	0.77	0.26	0.18
71	*trans*,*trans*-α-Farnesene	1500	tr	tr	tr
72	Myristicin	1502	nd	0.38	0.61
73	Elemicin	1514	tr	0.27	0.35
74	Bicyclogermacrene	1517	nd	5.05	0.89
75	δ-Cadinene	1519	0.13	0.16	0.24
76	Calamenene	1523	0.52	0.22	0.18
77	Cadina-1,4-diene	1527	0.22	0.17	0.31
78	Elemol	1541	0.16	0.08	0.02
79	*trans*-Nerolidol	1550	1.62	0.25	0.12
80	Caryophyllene alcohol	1556	1.23	0.16	0.33
81	Germacrene B	1562	nd	tr	tr
82	Caryophyllene oxide	1572	0.88	tr	tr
83	Dodecanol	1577	2.21	tr	tr
84	Guaiol	1589	nd	0.12	tr
85	Isoelemicin	1596	nd	tr	nd
86	Dill apiole	1608	nd	nd	tr
87	Spathulenol	1619	1.05	0.24	0.17
88	β-Eudesmol	1630	1.36	tr	tr
89	T-Muurolol	1635	nd	nd	tr
90	Cubenol	1645	nd	tr	nd
91	Bulnesol	1651	nd	0.16	tr
92	Cadina-1,4-dien-3-ol	1658	nd	tr	tr
93	α-Bisabolol	1662	tr	tr	tr
94	β-Bisabolol	1666	1.26	0.18	0.25
95	α-Cadinol	1676	nd	tr	0.31
96	*cis*,*trans*-α-Farnesol	1685	1.09	tr	nd
97	*trans*,*trans*-α-Farnesol	1696	0.44	0.10	tr
98	Benzyl benzoate	1723	tr	0.09	0.25
99	Phenylethyl benzoate	1841	tr	tr	nd
100	Hexadecanol	1870	0.12	tr	tr

[a] Kovats indices using a non-polar FSOT RSL-200 column.
[b] *P. nigrum* (black).
[c] *P. guineense* (black).
[d] *P. guineense* (white).
[e] Trace compound (concentration $<$0.01%).
[f] Compound not detected.

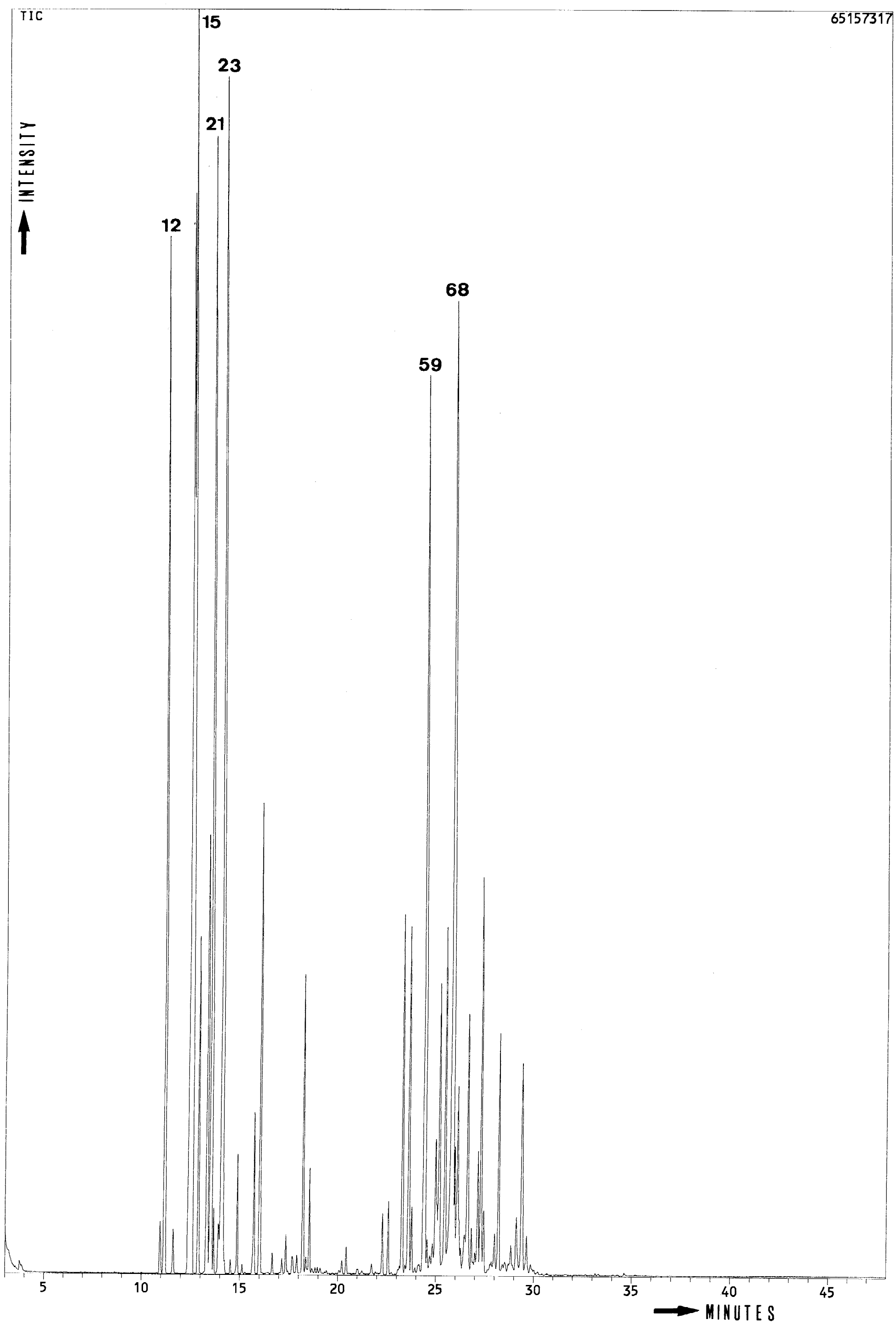

Fig. 1. SPME–GC–MS (total ion current, TIC) of *Piper nigrum* essential oil from Cameroon with detected and identified main compounds (12=α-pinene, 15=β-pinene, 21=*cis*-β-ocimene, 23=limonene, 59=β-caryophyllene, 68=germacrene D) using a non-polar FSOT RSL-200 fused-silica column.

Table 2
Olfactoric data correlations of the GC–sniffing technique (''GC–ST'') of identified volatiles of the three different *Piper* headspace SPME samples with published aroma notes [34–39] (''Literature'')

No.	GC–ST	Literature
01	Pungent	Pungent, etheral, unpleasant
02	Acetic acid	Sour, strong acetic acid
03	Unpleasant, rancid	Pungent, rancid
04	Green	Fatty, green, grassy
05	Buttery	Sharp, cheesy, rancid, putrid, sour, sweaty
06	Green	Green, almond-like, herbal
07	Green	Green, grassy, fresh
08	Green	Green, leafy
09	Sweet, solvent-like	Sweet, herbal, mild-woody
10	Herbal, solvent-like	Herbal
11	Herbal	Green, herbal, woody
12	Pine-needle-like	Pine-like, sharp, woody, turpentine-like
13	Camphoraceous	Camphoraceous, mild-oily
14	Pepper-like	Warm, oily-peppery, woody-herbaceous, spicy
15	Terpene-like	Dry-woody, pine-like, resinous-terpene-like, spicy
16	Balsamic	Mild, sweet, balsamic, plastic-note
17	Spicy	Herbaceous, minty, peppery-woody, fresh, citrus
18	Resinous	Sweet, refined-limonene-like, spicy
19	Citrus-like	Refreshing, lemon-citrus-like
20	Pepper-like	Peppery, minty, refreshing, citrus-like
21	Herbal	Warm-herbaceous, sweet-floral, neroli-oil-like
22	Fresh	Weak citrus-like, lemon- and bergamot-notes
23	Citrus-like	Fresh, citrus-like, mild lemon- and orange-notes
24	Herbal	Herbaceous, weak floral
25	Weak citrus-like	Fresh-herbaceous, citrus-like
26	Earthy	Earthy, cocoa-like
27	Weak balsamic	Mild, pleasant, warm, woody-balsamic
28	Plastic-like	Sweet-piney, oily, petroleum-like
29	Fatty	Floral fatty, waxy
30	Floral	Fresh, floral, clean, sweet, lemon-notes
31	Weak woody	Warm, balsamic-woody, mild
32	Weak fatty	Fatty, waxy
33	Camphor	Fresh, warm-minty, etheral
34	Fatty	Fatty, green
35	Camphoraceous	Camphoraceous, weak peppery and woody
36	Camphoraceous	Woody-camphoraceous, dry-minty
37	Weak citrus	Weak citrus-like
38	Fatty	Fatty, cheesy
39	Spicy	Nutmeg-like, spicy, woody-earthy, liliac-like
40	Floral	Floral, liliac-like
41	Fresh	Caraway- and spearmint-like
42	Pungent	Sharp, acid, pungent, woody, oily
43	Floral	Floral, rose-like
44	Spicy	Caraway- and spearmint-like
45	Fresh	Fresh, minty, camphoraceous
46	Weak floral	Floral, fatty
47	Fatty	Fatty, cheesy
48	Spicy	Sweet, warm-spicy, woody-floral
49	Spicy	Floral, oily-spicy, dillseed- and caraway-like

Table 2. Continued

No.	GC–ST	Literature
50	Weak woody	Lime-oil-like, woody
51	Spicy	Clove- and cinnamon-like, spicy
52	Herbal	Spicy, herbal
53	Vegetable-like	Hops-like, woody
54	Herbal	Herbal, woody
55	Weak fresh	Fresh, warm, weak woody
56	Herbal	Sweet, herbal, waxy, floral
57	Spicy	Clove-like, spicy
58	Woody	Balsamic, woody
59	Spicy	Woody, spicy, terpene-notes
60	Weak woody	Mild, woody
61	Weak woody	Warm, tea-leaf-like
62	Weak sweet	Warm, mild, sweet
63	Weak woody	Sweet-woody, balsamic, peppery
64	Weak woody	Weak woody, weak spicy, herbal
65	Weak woody	Weak spicy, weak herbal, woody
66	Spicy	Spicy, pepper-like
67	Weak woody	Balsamic, woody, spicy
68	Weak spicy	Weak fruity, apple-like, weak dry-woody
69	Spicy	Spicy, fresh, sharp
70	Weak balsamic	Sweet, warm, balsamic, woody, spicy
71	Weak sweet	Mild, warm, sweet
72	Balsamic	Warm, mild, balsamic, woody
73	Spicy	Sweet, woody-floral, spicy
74	Weak woody	Spicy, mushroom-notes, dry-woody
75	Woody	Dry-woody, weak medicinal
76	Spicy	Weak floral, weak spicy
77	Weak spicy	Fruity, mango-like, spicy, woody
78	Weak woody	Sweet-woody, mild, weak floral
79	Floral	Woody-floral, weak green
80	Weak spicy	Earthy-mossy, spicy, weak woody
81	Earthy	Mushroom-like
82	Weak spicy	Weak woody, warm, mild, weak spicy
83	Fatty	Fatty, waxy, coconut-like
84	Weak balsamic	Woody-balsamic, mild, sweet, weak floral
85	Weak spicy	Sweet, spicy, floral
86	Weak woody	Warm-woody, weak spicy
87	Weak herbal	Weak fruity, weak herbal
88	Weak woody	Woody
89	Weak spicy	Weak woody, weak spicy, mild
90	Weak spicy	Spicy, herbal, green-tea-notes
91	Weak spicy	Weak woody, weak spicy
92	Weak woody	Weak fruity, weak woody, spicy
93	Weak floral	Sweet, mild floral
94	Weak sweet	Mild, sweet, weak herbal
95	Weak woody	Weak woody, medicinal, dry
96	Weak floral	Mild, sweet, oily, floral, fresh-green
97	Weak fresh	Fresh-green, weak floral
98	Weak balsamic	Faint, sweet-balsamic, floral
99	Weak floral	Floral, rose-like, honey-notes
100	Weak floral	Waxy, mild, floral

and white *Piper guineense* from Cameroon is the result of the odor impression of various identified headspace SPME constituents of the three corresponding oil samples. In particular, mono- and sesquiterpenes were found to be of essential importance for the fine, pleasant pepper aroma (all essential oils), with pinene and limonene (black pepper oil), clove and nutmeg, herbal/vegetable-like and fatty (black ''Ashanti pepper'') as well as fruity (black and white *P. guineense*), fresh and floral (white ''Ashanti pepper'') side-notes. Headspace SPME trapping of essential oil volatiles and the combined use of GC–FID, GC–MS, the GC–sniffing technique and olfactometry made it possible to identify the target components responsible for the characteristic aroma of these three different Cameroonian *Piper* species for the first time.

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