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Characterization of volatile components of Kangra orthodox black tea by gas chromatography-mass spectrometry

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Abstract

To characterize Kangra tea flavour, volatile components of Kangra orthodox black tea (*Camellia sinensis* (L.) O. Kuntze) were isolated by two different methods viz. simultaneous distillation extraction (SDE) and hydrodistillation, and analysed by GC–MS. The composition of the volatile components extracted by the two methods differed considerably. A total of 101 compounds were tentatively identified in Kangra tea by matching EI mass spectra and retention indices with the literature data. Fifty compounds, constituting the major part of the volatiles (98.4 ± 0.4%) were detected in all the three replicates of made tea obtained by SDE. Major volatile components identified in Kangra tea were (*E*)-2-hexenal, 1-pentene-3-ol, and (*Z*)-3-hexenol among non-terpenoids and linalool, linalool oxides (furanoid), geraniol, methyl salicylate, 3,7-dimethyl-1,5,7-octatrien-3-ol, and epoxylinalol among terpenoids. The total volatile oil yield was about 0.02% by Clevenger type apparatus. SDE was found to be a more efficient technique than the other distillation processes for characterizing volatile components in terms of sample size and components extracted, and the extracted flavour was also closer to Kangra made tea.

Keywords: *Camellia sinensis*; Kangra orthodox black tea; Volatile components; SDE; Hydrodistillation; Clevenger type apparatus; Mini distillation apparatus; Hydrosol

1. Introduction

Tea is the most widely consumed non-alcoholic beverage in the world. Consumer acceptability of the beverage is mainly dependent on the flavour of the finished product. Flavour comprises principally taste and aroma. Non-volatile components are generally responsible for the taste, while volatile components give the aroma. In tea, volatile organic components (VOCs) are present in very minute quantities, i.e. 0.01% of the total dry weight, but these have high impact on the flavour of the products due to their low threshold value and resulting high odour units. Moreover, the olfaction is better developed in humans for flavour perception compared to gestation. VOCs of tea are classified into two groups, Group I consisting of mainly non-terpenoids, which imparts fresh green flavour, e.g. hexenols, and Group II of terpenoids, which imparts sweet flowery aroma to tea, viz, monoterpene alcohols such as linalool and geraniol. The presence of group II compounds is highly desirable. The ratio of terpenoid (Group II) to non-terpenoid (Group I) has been used to classify black teas for flavour quality (Yamanishi, Kobayashi, & Nakamura, 1968a; Yamanishi, Wickremasinghe, & Perera, 1968b).

Kangra valley mainly produces orthodox black teas. The composition and concentration of VOCs play an important role in the valuation, as flavoury teas fetch a price 4–5 times higher. According to tea taster's evaluation, Kangra tea has a distinct flavour, differing from Darjeeling, Keeman, and Uva teas. Growth flush of tea in Kangra valley is divided into three flushes: early (April–May), mains (July–August), and backend (mid September–mid November). Kangra tea shows high seasonal variations. High quality first flush tea sells at a premium price. The first flush

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quality shows qualitative and quantitative decline in the flavour components and infusion characteristics during the mains flush (Gulati & Ravindranath, 1996). Very little work has been done on characterization of Kangra tea flavour. Some of the major volatile flavour components have been used to identify the regio-specific characteristics of important teas of India including Kangra tea (Borse, Rao, Nagalakshmi, & Krishnamurthy, 2002). In the present study, we have determined VOCs obtained by different extraction methods and tried to use them to characterize Kangra orthodox black tea.

2. Experimental

2.1. Materials

Orthodox black tea samples were obtained from the Institute's Experimental Tea Factory. The tea was manufactured from first flush shoots (two and a bud) grown in the Institute's Tea Experimental Farm at Banuri (altitude 1200 m above, mean sea level).

Dichloromethane used was of HPLC grade (Merck, Mumbai, India). Anhydrous sodium sulphate and sucrose were of AR grade.

2.2. Isolation procedures

2.2.1. Extraction of VOCs by simultaneous distillation extraction (SDE)

The SDE apparatus used was a Likens and Nickerson's apparatus (Yamanishi et al., 1968b) modified with an extended condenser and an air vent connected to a vacuum pump maintained at 200 mm Hg. The coolant of the condenser was maintained at 5 °C with the help of a circulatory water bath. One hundred grams of orthodox black tea was placed in a round-bottom flask of 1 l capacity along with 1 g of sucrose dissolved in 700 ml of distilled water. The solvent flask was charged with 20 ml of HPLC-grade dichloromethane. The heating mantles of the solvent and sample flasks were maintained at 60–70 and 75–80 °C, respectively. Once the contents of both flasks were boiling, the process was continued for 20 min to allow the VOCs to collect in dichloromethane. The solution was dried over anhydrous sodium sulfate.

2.2.2. Flavour enrichment by SDE

VOCs extracted from three batches by SDE as mentioned above were pooled, dried over anhydrous sodium sulfate and concentrated following the method of Yamanishi et al. (1968b). The concentrate was used for GC–MS analysis. A dichloromethane blank was also run in GC–MS.

2.2.3. Hydrodistillation

2.2.3.1. Extraction of VOCs using mini distillation apparatus. Five kilograms of orthodox black tea was charged into a mini essential oil distillation apparatus (Babu, Ahuja, Kaul, & Singh, 2005; Babu, Kaul, & Ahuja, 2002) comprising of a distillation vessel capacity 50 l, a compatible cohobation column, condenser, and receiver. Before charging the apparatus the false bottom/perforated grid was adjusted to the lowest point to allow hydrodistillation of the material yet avoid charring it. About 30 g of sucrose were dissolved in 18 l of distilled water and charged along with the material. It took 20 min to bring the contents in the distillation tank to the boil. The VOCs carried by the steam were condensed in a shell and tube condenser and the distillate collected in the receiver. The distillate is termed as 'tea hydrosol'. The process was continued for 3 h to collect 5 l of tea hydrosol. The experiment was repeated with three more batches (5 kg made tea each). The tea hydrosol collected from the four batches was pooled and extracted thrice with 3 l of dichloromethane. The dichloromethane extract (9 l) was dried over anhydrous sodium sulfate and filtered. The filtrate was concentrated under vacuum in a pilot scale Laborota 20S (Heidolph, Germany) rotary evaporator to 30 ml. This concentrate was used for GC–MS analysis.

2.2.3.2. Extraction of VOCs using Clevenger type apparatus. Three grams of sucrose were dissolved in 2.5 l of distilled water and placed in a round-bottom flask of 5 l capacity along with 500 g of orthodox black tea. The material was soaked for about 16 h before it was subjected to hydrodistillation in a Clevenger type apparatus (Clevenger, 1928; Maisonneuve & Metz, 1983). The contents were distilled for 3 h to collect the volatile oil. The experiment was repeated thrice. The mean recovery of volatile oil was about 0.02%. The oil separated was dissolved in dichloromethane (10 ml), filtered, dried over anhydrous sodium sulfate, and used for GC–MS analysis.

2.3. Gas chromatography–mass spectrometry (GC–MS) analysis

The concentrated extracts from the different extraction procedures above were analysed on a Shimadzu QP2010 GC–MS system with 2010 GC. A BP-20 SGE column (30 m × 0.25 mm id, film thickness 0.25 μm) was used with helium as a carrier gas. The injector temperature was 220 °C with split ratio 1:50. The GC oven temperature was programmed to hold at 70 °C for 4 min and then to increase to 220 at 4 °C/min, finally holding at 220 °C for 5 min. Column flow rate was 1.10 ml/min. Ion source temperature was 200 °C and the interface temperature was set at 220°C. The MS was scanned at 70 eV over 40–600 a.m.u. Sample injection volume was 2 μl. Relative percentages were calculated from the TIC from the automated integrator.

2.3.1. Identification of compounds

Retention indices (RI) of the compounds relative to a mixture of *n*-alkanes (C_{11}–C_{27}) were determined. Identification of compounds was first attempted using mass spectral libraries Wiley 7 and NIST 02 (McLafferty, 2000; Stein, 1990). Corroboration of the identification was then sought by matching the mass spectra of compounds with those present in our own library built up from using some authentic flavour compounds and in the literature (Adams, 1989; Jennings & Shibamoto, 1980; McLafferty & Stauffer, 1989) and finally by matching the RI of the compounds reported on equivalent column (Kawakami, Ganguly, Banerjee, & Kobayashi, 1995; Pino, Almora, & Marbol, 2003).

3. Results and discussion

In black tea, volatile flavour components constitute only 0.01–0.02% of the total dry weight, unlike most aromatic crops, which contain higher percentages of volatile constituents. Two different extraction procedures, i.e. simultaneous distillation extraction and hydrodistillation using Clevenger type and mini-distillation apparatus, were employed to extract volatile flavour components in order to characterize Kangra orthodox black tea flavour. GC–MS profile of the extracted flavour shows the presence of a wide range of compounds, including terpenoids, alcohols, acids, aldehydes, ketones, esters, and aromatic, and long-chain hydrocarbons. Qualitative and quantitative differences were observed in terpenoid and non-terpenoid flavour components following different extraction procedures. Solvent blank was run as a control, no additional peaks were detected in the blank except the dichloromethane peak.

The VOC profile of tea obtained by simultaneous distillation extraction was dominated by terpenoids (Table 1). Linalool oxide-II (furanoid) constituted the highest amount (19.06%), followed by geraniol (16.21%) and linalool oxide-I (furanoid) (7.64%). Among the non-terpenoids, (*Z*)-3-hexenol was present in a relatively high amount (7.11%). (*E*)-2-hexenal (3.37%) and alcohols like 1-penten-3-ol (4.68%) and 1-pentanol (3.36%) were also present in comparable amounts. Other compounds like methyl salicylates (3.03%), 3,7-dimethyl-1,5,7-octatrien-3-ol (2.48%) and epoxy linalool (2.28%) were detected in somewhat lower amounts.

An enriched sample prepared by combining and concentrating three SDE extracts, showed the greatest number of volatile compounds in the GC–MS profile (Table 2). Qualitative and quantitative differences were observed between the profiles of the enriched sample and a straight SDE one. The unreplicated minor compounds which constituted 1.60% of the total composition in SDE (Table 1), along with the major components were identified in the enriched SDE sample. The profile of enriched SDE showed monoterpenoids which were not detected in the profile of single SDE due to their very minute undetectable quantities, but attained detectable levels due to increased concentration in combined SDE extraction. The enriched SDE extract was also dominated by terpenoids. The major compounds in enriched SDE were linalool oxide-II (furanoid) (10.66%), geraniol (5.74%), linalool oxide-I (furanoid) (4.51%), linalool (4.05%) in the terpenoid region and (*E*)-2-hexenal (16.71%) and (*Z*)-3-hexenol (6.87%), 1-penten-3-ol (4.15%) in the non-terpenoid region.

Since, sample size is a limitation in SDE, hydrodistillation using Clevenger type apparatus and mini distillation apparatus were also employed to extract VOCs from tea. A yield of 0.02% oil was recovered from 500 g of Kangra orthodox black tea by hydrodistillation using Clevenger type apparatus and was dominated by oxygenated monoterpenes, which comprised 47.64% of the total composition, geraniol (12.32%) and β-ionone (12.15%) being present in the highest amounts, followed by nerolidol (9.29%), hexahydrofarnesylacetone (8.34%), linalool (5.46%), α-damascone (3.70%), and α-irone (1.49%). Linalool oxides I and II, α-terpeneol, epoxylinalol were present in comparatively low percentages. Phytol, the only diterpene detected was also present in high amounts (14.37%). *n*-Hexanoic acid (7.79%) was the major non-terpenoid detected and 2,6,6-trimethyl-2-hydroxycyclohexanone (2.75%) was detected in relatively higher amount.

The GC–MS profile of VOCs, extracted as hydrosol by the mini-distillation apparatus followed by partitioning with dicholromethane, was dominated by aromatic hydrocarbons (59.43%), comprising mainly of ethylbenzene (40.84%), while *p*-xylene (5.29%), *m*-xylene (8.92%), and *o*-xylene (4.01%) were present in relatively high amounts, as well as linalool oxide II (4.81%).

A total of 103 compounds were detected in Kangra orthodox black tea, out of which 101 compounds have been identified and are listed in Table 2. Unidentified compounds are also included in the Table 2 and their mass spectral patterns are given in Table 3. Most of the compounds have previously been reported from tea either on polar or nonpolar GC columns (Robinson & Owuor, 1992). The sesquiterpenes detected in Kangra orthodox black tea were farnesol and its derivatives. Maximum numbers of compounds were extracted by SDE enrichment. Three compounds are being reported here for the first time from tea viz. *cis*-α-irone (dimethyl-α-ionone), 1,3-dioxolane and 2,7-epoxy-megastigma-4,8-diene. The latter compound along with similar megastigmadienes have been reported in the aroma of *Osmanthus* (Kaiser & Lamparsky, 2004). 2,7-Epoxy-megastigma-4,8-diene was detected only in hydrodistillate using the Clevenger type apparatus and could be an artifact formed by heat combination of α-ionone/α-irone and an aliphatic alcohol. A compound of similar structure, 1-methyl-7-oxa-bicyclo-2,2,1-heptane-2-one, has been reported as a chiral building block for terpenoids (Guan & Li, 2005). 1,3-Dioxolane has also been reported from coffee flavour (Tateo, Bononi, Lubian, & Martello, 1998). Theaspirone, jasmine lactone, methyl jasmonate and dihydroactinidiolid which are reported from Japanese tea were not found in Kangra orthodox black tea, by any of the extraction procedures, though 4-butanolide, which is also a lactone, is present. Absence of these compounds can be attributed to geographical variations (altitude and climatic conditions that prevail in Kangra valley in India)

Table 1
Tentatively identified volatile flavour components and their percentage composition in Kangra orthodox black tea processed by SDE

S. No.	Components	RI[a]	Identification	% Composition[b]
1	2-Methyl-2-butanol	1162	RI, MS	0.189 ± 0.0006
2	1-Penten-3-ol	1167	RI, MS	4.684 ± 0.5790
3	4,5-Dihydrofuran-3-carbaldehyde	1172	RI, MS	0.091 ± 0.0211
4	2-Heptanone	1192	RI, MS	0.283 ± 0.0218
5	*n*-Heptanal	1194	RI, MS	0.352 ± 0.1305
6	Isoamyl acohol	1215	RI, MS	0.451 ± 0.0192
7	(*E*)-2-Hexenal	1230	RI, MS	3.375 ± 0.0459
8	2-Pentylfuran	1239	RI, MS	0.251 ± 0.1339
9	1-Pentanol	1259	RI, MS	3.357 ± 0.2838
10	2-Methylpyrazine	1276	RI, MS	0.882 ± 0.0236
11	2,2,6-Trimethylcyclohexanone	1323	RI, MS	0.206 ± 0.0132
12	(*Z*)-2-Pentenol	1331	RI, MS	2.970 ± 0.1357
13	2,3-Octadione	1336	RI, MS	0.270 ± 0.0217
14	Ethylpyrazine	1342	RI, MS	0.448 ± 0.0098
15	6-Methyl-5-hepten-2-one	1347	RI, MS	0.229 ± 0.0465
16	1-Hexanol	1363	RI, MS	1.657 ± 0.0317
17	1-Methyl-2-cyano-2-piperidine	1389	RI, MS	0.145 ± 0.0036
18	(*Z*)-3-Hexenol	1393	RI, MS	7.113 ± 0.2630
19	2,5-Dimethylpyrazine	1408	RI, MS	0.128 ± 0.0244
20	(*E*)- 2-Hexenol	1416	RI, MS	1.314 ± 0.0164
21	Linalool oxide-I (furanoid)	1449	RI, MS	7.641 ± 0.3932
22	1-Octen-3-ol	1461	RI, MS	0.270 ± 0.0300
23	1-Heptanol	1464	RI, MS	0.273 ± 0.0300
24	Linalool oxide-II (furanoid)	1477	RI, MS	19.060 ± 1.1561
25	2-Acetylfuran	1512	RI, MS	0.250 ± 0.0091
26	Benzaldehyde	1528	RI, MS	1.221 ± 0.0450
27	Linalool	1559	RI, MS	7.289 ± 1.0998
28	1-Octanol	1567	RI, MS	0.223 ± 0.0838
29	5-Methyl-2-furfural	1581	RI, MS	0.057 ± 0.0289
30	6-Methyl-3,5-heptadien-2-one	1596	RI, MS	0.054 ± 0.0085
31	2,6,6-Trimethyl-2-hydroxycyclohexanone	1601	RI, MS	0.384 ± 0.0055
32	1,2-Dimethylpyridone	1611	RI, MS	1.275 ± 0.1456
33	3,7- Dimethyl-1,5,7-octatrien-3-ol	1620	RI, MS	2.483 ± 0.2622
34	α-Terpineol	1700	RI, MS	0.222 ± 0.0007
35	(−)-2,6,6-Trimethyl-2-vinyl-4-hydroxy-tetrahydropyran	1746	RI, MS	0.755 ± 0.0234
36	Valeric acid	1756	RI, MS	0.047 ± 0.0417
37	Epoxylinalol	1772	RI, MS	2.280 ± 0.0329
38	Methyl salicylate	1775	RI, MS	3.035 ± 0.7045
39	Nerol	1808	RI, MS	0.125 ± 0.0128
40	Geraniol	1857	RI, MS	16.211 ± 0.3163
41	*n*-Hexanoic acid	1864	RI, MS	3.566 ± 0.2167
42	1-Phenylpropane-1,2-diol	1884	RI, MS	0.821 ± 0.1402
43	2-Phenylethanol	1918	RI, MS	1.015 ± 0.0660
44	β-Ionone	1937	RI, MS	0.185 ± 0.0006
45	Heptanoic acid	1970	RI, MS	0.105 ± 0.0252
46	5,6-Epoxy-β-ionone	1989	RI, MS	0.145 ± 0.0119
47	Nerolidol	2049	RI, MS	0.148 ± 0.0037
48	Octanoic acid	2075	RI, MS	0.104 ± 0.0419
49	(*E*,*E*)-Farnesylacetone	2382	RI, MS	0.575 ± 0.0729
50	Phytol	2619	RI, MS	0.184 ± 0.0330
Total % composition				98.40 ± 0.3881
SD on total %				0.672

[a] Relative retention indices on BP20 column calculated against *n*-alkanes (C_{11}–C_{27}).

[b] Percentage composition is the mean of three replicates.

which give Kangra orthodox black tea a distinct aroma (Takeo, 1983a; Takeo, 1983b; Takeo & Mahanta, 1983; Wickremasinghe, 1974; Yamanishi et al., 1968a).

The SDE method of extraction was able to extract more volatile components from Kangra orthodox tea compared to the two steam distillation extraction techniques and the aroma extracted was found to be more representative and closer to the Kangra tea aroma (Table 4). The aroma extracted by hydrodistillation had a slightly burnt note, while that of Clevenger type possessed a pleasant flowery note (Table 4). The aromatic hydrocarbons detected in relatively high concentrations in tea hydrosol were not detected in other extracts was may be due to the fact that the liquid–liquid extraction of VOCs between hydrosol

Table 2
A comparative of volatile flavour components tentatively identified by different extraction procedures

S. No.	Components	RI	Hydrodistillation		SDE[b]
			Clevenger type	MDA[a]	Enriched
1	Isobutyl alcohol	1103	–	0.37	–
2	*n*-Undecane	1109	–	0.62	–
3	3-Pentanol	1116	–	0.21	–
4	Ethylbenzene	1142	–	40.84	–
5	*p*-Xylene	1149	–	5.30	–
6	*m*-Xylene	1156	–	8.92	–
7	2-Methyl-2-butanol	1162	–	–	0.09
8	1-Penten-3-ol	1167	–	0.21	4.15
9	Isopropylbenzene	1185	–	0.23	–
10	2-Heptanone	1192	–	–	0.23
11	*n*-Heptanal	1195	–	–	0.46
12	*o*-Xylene	1197	–	4.01	–
13	*n*-Tetradecane	1203	–	0.43	–
14	3-Methyl-1-hexene	1213	–	–	1.13
15	Isoamyl alcohol	1215	–	2.80	–
16	(*E*)-2-Hexenal	1230	–	–	16.71
17	1-Pentanol	1259	–	–	2.70
18	2-Methylpyrazine	1276	–	–	0.06
19	Benzene	1290	–	0.14	–
20	3-Hydroxy-2-butanone	1298	1.59	–	–
21	2,2,6-Trimethylcyclohexanone	1323	–	–	0.24
22	(*Z*)-2-Pentenol	1331	–	0.48	2.41
23	6-Methyl-5-hepten-2-one	1347	–	0.27	0.66
24	1-Hexanol	1363	0.12	–	2.25
25	(*Z*)-3-Hexenol	1393	0.90	1.66	6.87
26	Vinylmethylether	1395	0.17	–	–
27	*n*-Nonanal	1398	–	–	0.20
28	2,5,6,6-Tetramethylcyclohexe-2-en-1-one	1405	–	–	0.06
29	(*E*)-2-Hexenol	1416	0.05	–	1.31
30	Linalool oxide-I (furanoid)	1449	0.77	2.02	4.51
31	1-Octen-3-ol	1461	–	–	0.36
32	1-Heptanol	1464	–	–	0.26
33	*n*-Heptyl acetate	1469	–	–	0.05
34	2-Propylfuran	1473	0.10	–	0.13
35	Linalool oxide-II (furanoid)	1477	0.90	4.81	10.66
36	2-Ethylhexan-1-ol	1494	–	–	0.04
37	(*E*,*E*)-2,4-Heptadienal	1497	–	–	0.90
38	2-Acetylfuran	1512	0.25	–	0.10
39	Benzaldehyde	1528	–	–	2.96
40	Linalool	1559	5.46	2.14	4.05
41	1-Octanol	1567	0.24	–	0.43
42	3,5-Octadien-2-one	1575	–	–	0.32
43	6-Methyl-3,5-heptadien-2-one	1596	–	0.14	0.31
44	2,6,6-Trimethyl-2-hydroxycyclohexanone	1601	2.75	1.28	1.78
45	1,2-Dimethylpyridone	1611	–	–	0.13
46	β-Cyclocitral	1619	–	–	0.11
47	3,7- Dimethyl-1,5,7-octatrien-3-ol	1620	–	–	0.15
48	4-Butanolide	1634	–	0.26	–
49	2-Methoxyphenylethanol	1645	0.11	–	0.05
50	Phenylacetaldehyde	1649	–	–	1.62
51	Acetophenone	1654	0.14	0.88	0.32
52	Methyl tetradeca-10,11-dienoate	1663	0.20	–	–
53	3-Methyl-2,4-nonanedione/4-hydroxybutyl hexanoate	1666	–	–	0.05
54	1-Nonanol	1669	1.62	–	0.16
55	Isovaleric acid	1687	–	1.00	0.63
56	2(*S*)-hydroxy-γ-butyrolactone	1692	0.24	–	–
57	α-Terpineol	1700	0.29	0.43	0.18
58	Unidentified	1736	0.39	–	–
59	(−)-2,6,6-Trimethyl-2-vinyl-4-hydroxy- tetrahydropyran	1746	–	0.88	0.42
60	Valeric acid	1756	–	0.86	0.32
61	(*Z*)-3-(Hydroxymethyl)-7-methylocta-2,6-dien-1-yl acetate	1764	0.27	–	–

(*continued on next page*)

Table 2 (*continued*)

S. No.	Components	RI	Hydrodistillation		SDE[b]
			Clevenger type	MDA[a]	Enriched
62	Epoxylinalol	1772	0.31	2.21	1.39
63	Methyl salicylate	1775	2.63	0.46	1.30
64	2-Butene-2-ol	1802	0.21	–	0.05
65	Nerol	1807	0.35	–	0.53
66	α-Damascone	1812	3.70	–	0.06
67	1-Phenylethanol	1822	0.27	0.12	–
68	(*E*)-2-Decenyl acetate	1830	1.40	–	–
69	α-Irone	1850	1.49	–	0.16
70	Geraniol	1857	12.32	0.39	5.74
71	*n*-Hexanoic acid	1864	7.79	5.20	5.58
72	Acetonyl decyl ether	1904	0.12	–	–
73	2-Phenylethanol	1918	–	5.93	1.29
74	β-Ionone	1937	12.15	–	1.26
75	Heptanoic acid	1968	–	–	0.35
76	3-Hexenoic acid	1977	–	1.62	–
77	2-Hexenoic acid	1984	–	1.41	–
78	5,6-Epoxy-β-ionone	1989	–	–	0.48
79	3-Butenen-2-one	1990	0.39	0.12	–
80	2-Methylvaleraldehyde	2024	1.29	–	–
81	Nerolidol	2049	9.29	–	0.47
82	Octanoic acid	2075	–	–	0.38
83	(*Z*)-3-Hexenyl benzoate	2127	0.50	–	–
84	Hexahydrofarnesylacetone	2130	8.34	–	0.63
85	Methyl cyclopropyl ketone	2139	0.35	–	–
86	3,6-Dimethyl-2*H*-pyran -2-one	2152	0.50	–	0.17
87	Nonanoic acid	2181	0.72	–	0.61
88	2,7-Epoxy-megastigma-4,8-diene	2187	0.61	–	–
89	Docosane	2202	–	–	0.21
90	Methyl nonanoate	2232	0.87	–	0.23
91	Tridecanoic acid	2316	1.31	–	0.24
92	Tricosane	2328	0.31	–	0.48
93	2(4*H*)-Benzofuranone	2352	–	0.73	0.15
94	(*E*,*Z*)-Farnesol	2365	–	0.70	0.11
95	(*E*,*E*)-Farnesylacetone	2382	1.04	–	0.21
96	Tetracosane	2402	–	–	0.79
97	Pentacosane	2483	–	–	1.13
98	Dicyclohexyl phthalate	2529	0.20	–	0.23
99	Hexacosane	2602	–	–	1.28
100	Phytol	2619	14.37	–	1.69
101	1,3-Dioxolane	2653	–	–	0.17
102	1-Propene-1-thiol	2635	–	–	0.05
103	Unidentified	*N*	–	–	2.06
	Total % composition		99.40	100	100

N: RI not calculated.

[a] MDA: Mini distillation apparatus.

[b] SDE: Simultaneous distillation extraction.

Table 3
Mass spectral fragmentation patterns of the unidentified compounds

S. No.	RI	Fragmentation pattern in the order of decreasing intensity of peak
58	1736	108,91,41,69,44
103	Not calculated	57,71,43,85,99, 41,55,113,69,97,127,56,70,83,141,155,111,43

Table 4
Sensory evaluation of extract prepared from Kangra orthodox black tea following different methods

Extract	Flowery note	Roasted note	Kangra note	Overall assessment
SDE	++	++	++	Closer to Kangra tea infusion
Clevenger type	+++	+	+	Flowery
MDA	+	+++	+	Roast

(water phase) and dichloromethane was carried out in liquid phase at ambient temperature (22–25 °C) and pressure, while in SDE it took place both in the vapor phase as well as after condensation under vacuum. Chemical and physical properties (diffusion and partition coefficients) of solutes and solvents vary with phases and their physical conditions (temperature, pressure/vacuum) in which they are partitioned. On the other hand, the highly volatile components (RI 1103–1297) were not recorded in GC–MS profile of Kangra tea extracted by Clevenger type apparatus.

In SDE, one-step isolation/concentration of VOCs saved time over the other separating operations, i.e. Clevenger and hydrodistillation using mini-distillation apparatus. SDE is better in terms of sample size, amount of solvent and time consumed to extract the volatile components. In SDE it took 100 g of sample, 20 ml dicholoromethane and 35 min for complete extraction while in hydrodistillation using mini distillation apparatus with a total of 15 kg of tea sample and 9 l dicholoromethane was required. The process took another 3 h to complete. In Clevenger type apparatus 500 g of tea sample which was soaked in water for 16 h and the process took another 3 h for complete extraction.

Moreover, SDE seems to be a better method for characterization of aroma components from crops like tea where VOCs concentration is low, and require fast extractions. The percent compositions of VOCs may vary from sample to sample, season to season as orthodox black tea manufacture is very sensitive to environment and process conditions during manufacture, but the major compounds present will always remain same.

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