

Supplementary Materials

Influence of Citrus Flavor Addition in Brewing Process: Characterization of the Volatile and Non-Volatile Profile to Prevent Frauds and Adulterations

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Table S1. Less prevalent volatile compounds contained in the samples analysed, expressed in area% (\pm SD) as average of three measurements by GC-FID analysis.

n.	Compound	LRI _{ex}	LRI _{rif}	Negative control			Orange					Lemon			Bergamot		Mandarin		
				1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
6	Isovaleric aldehyde	670	676	-	-	0.06 \pm 0.02	0.17 \pm 0.08	0.21 \pm 0.05	-	-	0.25 \pm 0.06	tr	tr	-	-	-	0.36 \pm 0.09	-	-
8	<i>n</i> -Heptane	695	700	-	0.06 \pm 0.02	-	-	tr	-	-	0.10 \pm 0.03	tr	tr	-	-	-	tr	-	-
9	Propyl methyl ketone	698	695	-	-	-	-	0.2 \pm 0.05	-	-	0.27 \pm 0.07	-	-	-	tr	-	0.05 \pm 0.01	-	-
10	2,3-Pentanedione	701	695	-	-	-	-	tr	-	-	0.08 \pm 0.02	-	-	-	-	tr	tr	-	-
11	Pentanal	703	706	-	-	-	-	tr	-	-	0.05 \pm 0.01	-	-	-	-	tr	tr	-	-
12	2,5-Dimethylfuran	704	703	-	0.05 \pm 0.01	-	-	0.05 \pm 0.01	-	-	tr	tr	-	-	-	-	tr	-	-
13	Ethyl propanoate	706	708	0.26 \pm 0.07	tr	tr	-	-	tr	tr	-	tr	tr	-	tr	tr	tr	tr	-
14	<i>n</i> -Propyl acetate	714	708	-	tr	-	-	tr	-	tr	0.11 \pm 0.03	tr	-	-	tr	-	tr	-	-
15	Diethyl acetal	726	721	tr	tr	-	tr	tr	0.05 \pm 0.01	0.05 \pm 0.01	0.07 \pm 0.02	-	-	tr	0.08 \pm 0.02	0.05 \pm 0.01	tr	0.06 \pm 0.02	-
18	Ethyl isobutyrate	749	752	0.05 \pm 0.05	0.18 \pm 0.05	0.07 \pm 0.02	0.06 \pm 0.03	0.46 \pm 0.12	-	tr	0.15 \pm 0.04	tr	tr	-	-	tr	tr	0.08 \pm 0.02	0.05 \pm 0.01
19	Pentyl alcohol	753	752	-	-	-	-	0.32 \pm 0.08	-	-	0.09 \pm 0.02	tr	tr	-	-	-	tr	-	-
20	Toluene	757	763	-	-	-	-	-	-	-	0.19 \pm 0.05	tr	tr	-	-	-	-	-	-
21	Isobutyl acetate	764	768	tr	0.16 \pm 0.04	tr	-	0.12 \pm 0.03	tr	0.07 \pm 0.02	0.17 \pm 0.04	tr	tr	tr	0.05 \pm 0.01	0.06 \pm 0.02	0.06 \pm 0.02	tr	tr
22	Ethyl butyrate	796	803	0.19 \pm 0.05	0.10 \pm 0.03	0.25 \pm 0.06	0.16 \pm 0.08	0.05 \pm 0.01	0.13 \pm 0.03	0.21 \pm 0.05	0.43 \pm 0.11	0.05 \pm 0.01	tr	tr	0.13 \pm 0.03	0.32 \pm 0.08	0.19 \pm 0.05	0.15 \pm 0.04	0.15 \pm 0.04
23	<i>n</i> -Hexanal	803	801	-	0.17 \pm 0.04	0.23 \pm 0.06	-	0.05 \pm 0.01	-	-	0.12 \pm 0.03	-	tr	-	-	-	0.06 \pm 0.02	-	-
25	1-Ethoxy-3-methyl-2-butene	821	817	-	0.13 \pm 0.03	-	-	0.1 \pm 0.03	-	-	0.09 \pm 0.02	0.05 \pm 0.01	tr	-	-	-	tr	tr	tr

28	Ethyl 2-methylbutyrate	846	842	tr	-	0.12 ± 0.03	-	-	-	-	-	-	-	-	-	-	-	tr	0.05 ± 0.01
29	Ethyl isovalerate	848	850	tr	tr	0.09 ± 0.02	tr	0.06 ± 0.02	tr	-	0.16 ± 0.04	tr	-	-	-	-	tr	0.12 ± 0.03	0.07 ± 0.02
30	(2E)-Hexenal	852	850	-	-	-	-	-	-	-	0.06 ± 0.02	-	-	-	-	-	-	-	-
31	(3Z)-Hexenol	856	853	-	-	-	-	-	-	-	0.26 ± 0.07	-	-	-	-	-	-	-	-
32	Furfuryl alcohol	857	849	-	-	-	-	0.07 ± 0.02	-	-	0.14 ± 0.04	tr	-	-	-	-	-	-	-
33	(2E)-Hexenol	865	864	-	-	-	-	-	-	-	0.06 ± 0.02	tr	-	-	-	-	tr	-	-
34	<i>n</i> -Hexanol	867	872	0.06 ± 0.02	tr	0.10 ± 0.03	tr	-	tr	0.06 ± 0.02	tr	-	-	-	tr	tr	tr	0.12 ± 0.03	0.05 ± 0.01
37	5-Methyl-5-hexen-2-one	881	873	-	-	-	-	tr	-	-	0.27 ± 0.07	0.05 ± 0.01	tr	-	-	-	-	-	-
38	Butyl methyl ketone	885	887	-	-	-	-	-	-	-	0.28 ± 0.07	0.09 ± 0.02	-	-	-	-	-	-	-
40	Furfuryl ethyl ether	896		tr	0.21 ± 0.05	0.05 ± 0.01	-	0.12 ± 0.03	tr	-	-	tr	-	-	0.09 ±	-	-	tr	tr
41	Ethyl valerate	897	899	tr	tr	-	-	0.14 ± 0.04	tr	-	0.11 ± 0.03	-	-	tr	-	-	tr	tr	-
43	Isobuty isobutyrate	913	913	0.38 ± 0.10	-	-	0.05 ± 0.02	-	0.13 ± 0.03	tr	-	-	-	-	tr	0.09 ± 0.02	-	-	-
44	Pentyl acetate	915	915	tr	0.05 ± 0.01	-	tr	0.16 ± 0.04	tr	-	0.06 ± 0.02	tr	-	-	0.07 ± 0.02	tr	-	-	-
45	Prenyl acetate	920	920	-	tr	-	-	0.14 ± 0.04	-	-	tr	-	-	-	-	-	-	-	-
46	3-Methylapopinene	921	927	-	-	-	-	0.20 ± 0.05	-	-	0.09 ± 0.02	tr	-	-	-	-	-	-	-
47	α -Thujene	925	927	tr	-	-	-	-	-	-	-	0.07 ± 0.02	-	-	-	-	tr	-	-
48	α -Pinene	926	933	-	tr	-	tr	tr	-	-	0.10 ± 0.03	tr	tr	-	-	-	tr	-	-
49	Methyl thioisovalerate	940	939	tr	-	-	-	-	-	-	-	-	-	-	-	-	-	tr	-
50	α -Fenchene	945	950	-	-	-	-	-	-	-	-	tr	0.12 ± 0.03	0.06 ± 0.02	-	-	-	-	0.09 ± 0.02
51	Camphene	948	953	tr	-	-	-	tr	tr	-	tr	tr	tr	0.09 ± 0.02	0.06 ± 0.02	-	tr	-	-

52	1,1-Diethoxy-3-methylbutane	949	946	-	-	-	-	-	-	-	-	-	tr	-	-	-	-	-	-
53	Butyl isobutyrate	951	953	tr	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
54	Thuja-2,4(10)-diene	953	953	-	tr	-	-	tr	-	-	tr	tr	tr	-	-	-	-	-	-
55	(2E)-Heptenal	959	956	-	-	-	-	-	-	-	-	0.06 ± 0.02	tr	-	-	-	-	-	-
56	Methyl 4-methylpentanoate	963	969	-	tr	-	-	tr	-	tr	-	0.09 ± 0.02	tr	-	-	-	tr	0.05 ± 0.01	-
57	Benzaldehyde	964	960	-	-	-	-	-	tr	-	tr	-	tr	-	-	-	-	-	-
58	Bois de Rose oxide	967	968	-	-	-	tr	0.07 ± 0.02	-	-	-	tr	tr	0.08 ± 0.02	-	0.10 ± 0.03	tr	-	-
59	Isopentyl propanoate	969	966	0.05 ± 0.01	tr	-	-	tr	-	-	0.07 ± 0.02	tr	tr	-	-	-	tr	-	-
60	2-Methylbutyl propanoate	970	968	0.18 ± 0.05	-	-	-	-	-	-	-	-	-	-	-	tr	-	tr	-
61	Sabinene	972	972	-	-	-	-	tr	-	-	-	-	-	-	-	-	-	-	-
62	n-Heptanol	973	970	-	tr	-	tr	tr	-	-	-	-	tr	-	tr	-	tr	tr	-
63	β-Pinene	976	978	-	0.23 ± 0.06	-	-	0.10 ± 0.03	-	-	0.15 ± 0.04	0.13 ± 0.03	tr	-	-	-	tr	-	-
64	2-Ethylbutyl acetate	979	972	-	0.07 ± 0.02	-	-	tr	-	-	-	0.12 ± 0.03	tr	-	-	-	tr	-	-
65	3-Methylpentyl acetate	980	987	-	0.09 ± 0.02	-	-	tr	-	-	-	-	tr	-	-	-	-	-	-
66	Vinyl amyl carbinol	982	978	-	0.31 ± 0.08	-	-	-	-	-	0.06 ± 0.02	0.09 ± 0.02	tr	-	-	-	tr	-	-
67	Hexanoic acid	980	997	tr	-	0.05 ± 0.01	-	-	-	tr	-	0.13 ± 0.03	-	-	-	-	-	0.18 ± 0.05	0.07 ± 0.02
68	6-Methyl-5-Hepten-2-one	981	986	0.07 ± 0.02	0.21 ± 0.05	0.25 ± 0.06	0.05 ± 0.02	0.27 ± 0.07	tr	0.10 ± 0.03	0.10 ± 0.03	0.06 ± 0.02	tr	0.08 ± 0.02	-	0.08 ± 0.02	tr	0.21 ± 0.05	-
70	6-Methyl-5-Hepten-2-ol	992	995	tr	-	-	-	0.08 ± 0.02	tr	-	0.11 ± 0.03	0.44 ± 0.11	-	tr	-	0.06 ± 0.02	0.27 ± 0.07	-	-
71	Butyl butanoate	994	999	tr	0.08 ± 0.02	-	-	0.18 ± 0.05	-	tr	0.11 ± 0.03	0.10 ± 0.03	tr	tr	-	-	-	-	-
73	Isobutyl 2-methylbutyrate	998	1002	0.12 ± 0.03	-	0.10 ± 0.03	tr	-	-	-	-	-	-	-	-	-	-	0.13 ± 0.03	-
76	Isobutyl isovalerate	1000	1000	0.18 ± 0.05	-	-	-	-	-	tr	-	-	-	-	-	0.13 ± 0.03	-	0.08 ± 0.02	-
77	α-Phellandrene	1004	1007	-	-	-	-	-	-	-	0.06 ± 0.02	0.15 ± 0.04	0.15 ± 0.04	0.06 ± 0.02	-	-	-	0.23 ± 0.06	-

78	δ -3-Carene	1006	1009	-	-	-	-	tr	-	-	-	0.28 \pm 0.07	0.33 \pm 0.08	-	-	-	-	0.15 \pm 0.04	-
79	Ethyl-(3E)-hexenoate	1007	1008	-	0.06 \pm 0.02	tr	-	tr	-	-	0.10 \pm 0.03	-	-	-	-	-	tr	-	-
80	Ethyl 2-methyl-2-pentenoate	1010	1020	-	-	tr	-	-	tr	-	-	-	-	tr	-	-	-	-	-
81	Hexyl acetate	1011	1012	-	0.40 \pm 0.10	-	-	0.07 \pm 0.02	0.17 \pm 0.04	0.18 \pm 0.05	0.11 \pm 0.03	0.08 \pm 0.02	0.07 \pm 0.02	tr	0.06 \pm 0.02	0.20 \pm 0.05	0.27 \pm 0.07	-	tr
88	<i>n</i> -Hexanoic acid	1019	1015	-	-	-	-	-	-	-	0.07 \pm 0.02	-	0.06 \pm 0.02	-	-	-	0.16 \pm 0.04	-	-
95	Phenylacetaldehyde	1042	1045	-	-	-	-	tr	-	-	-	0.12 \pm 0.03	-	-	-	tr	-	-	-
96	Tetrahydro-2,2-dimethyl-5-(1-methyl-1-propen-1-yl)furan	1043	1045	-	0.16 \pm 0.04	-	-	-	-	-	-	0.34 \pm 0.09	tr	0.05 \pm 0.01	-	-	-	-	-
98	2,6-Dimethyl-5-heptenal	1055	1053	0.06 \pm 0.02	-	-	-	tr	-	-	-	0.05 \pm 0.01	-	-	-	tr	-	-	-
99	Isopentyl butyrate	1056	1054	0.07 \pm 0.02	-	tr	-	-	-	-	-	-	tr	-	-	-	tr	tr	tr
101	Ethyl 5-methylhexanoate	1062	1072	0.08 \pm 0.02	tr	tr	tr	tr	tr	tr	-	0.18 \pm 0.05	-	-	-	tr	tr	tr	-
102	<i>cis</i> -Sabinene hydrate	1069	1069	tr	-	0.14 \pm 0.04	-	tr	-	-	-	-	-	-	-	-	-	-	0.07 \pm 0.02
103	<i>p</i> -Mentha-3,8-diene	1071	1073	0.11 \pm 0.03	-	0.05 \pm 0.01	tr	-	-	-	0.10 \pm 0.03	tr	-	-	-	-	tr	0.08 \pm 0.02	tr
105	<i>n</i> -Hexyl vinyl carbinol	1074	1079	tr	-	tr	-	-	-	0.05 \pm 0.01	-	0.05 \pm 0.01	tr	0.05 \pm 0.01	-	-	tr	-	-
106	2-Methyl-1-octanol	1084		tr	-	-	-	-	-	-	tr	-	-	-	-	-	-	-	-
107	Allyl hexanoate	1086	1081	tr	-	-	-	-	-	-	-	0.07 \pm 0.02	-	-	-	-	-	-	-
109	Fenchone	1088	1090	-	-	-	-	-	-	-	-	-	-	-	-	-	tr	-	-
110	Hexanal, diethyl acetal	1089	1088	-	-	-	-	-	-	-	-	0.19 \pm 0.05	-	-	-	tr	-	-	-
112	2-Nonanone	1092	1093	0.50 \pm 0.13	-	-	0.05 \pm 0.02	-	tr	0.05 \pm 0.01	-	-	-	-	tr	tr	-	0.07 \pm 0.02	-
117	Solusterol	1104	1106	0.33 \pm 0.08	-	-	-	-	-	-	-	-	-	tr	-	-	-	0.26 \pm 0.07	-
118	<i>cis</i> -Rose oxide	1107	1110	-	-	-	-	tr	-	-	-	-	tr	-	-	-	tr	-	-
119	<i>p</i> -1,3,8-Menthatriene	1108	1106	-	-	-	-	tr	-	-	-	tr	-	-	-	-	-	-	-

120	2-Methylbutyl isovalerate	1109	1109	0.73 ± 0.18	-	-	0.10 ± 0.04	-	0.11 ± 0.03	-	-	-	-	-	-	tr	-	-	-
121	Heptyl acetate	1110	1114	0.09 ± 0.02	0.10 ± 0.03	tr	tr	tr	tr	tr	-	-	tr	-	tr	tr	tr	tr	-
124	<i>trans-p</i> -Mentha-2,8-dien-1-ol	1121	1122	-	-	-	-	-	-	-	-	tr	tr	-	-	-	-	tr	-
125	Myrcenol	1122	1121	0.16 ± 0.04	-	-	-	-	tr	tr	-	-	-	-	-	-	-	-	-
126	2,5-Dimethyl-4-ethoxy-3(2H)-furanone	1123	1123	-	0.07 ± 0.02	-	-	0.07 ± 0.02	-	-	tr	0.08 ± 0.02	tr	-	-	-	-	0.09 ± 0.02	-
127	Methyl octanoate	1124	1125	-	-	tr	-	-	0.06 ± 0.02	tr	-	-	-	-	-	-	-	-	tr
128	(<i>Z</i>)- <i>p</i> -Menth-2-en-1-ol	1126	1124	-	-	-	-	-	-	-	-	-	tr	tr	-	-	-	tr	-
129	(4 <i>E</i> ,6 <i>Z</i>)-Allocimene	1129	1128	-	-	-	-	-	-	-	-	tr	-	-	-	-	-	tr	-
130	<i>trans</i> -Sabinol	1131	1140	-	-	-	-	-	-	-	-	tr	-	-	-	-	-	0.23 ± 0.06	-
131	Dihydrocitronellal	1132	1125	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
132	Limona ketone	1133	1131	-	0.06 ± 0.02	-	-	tr	-	-	-	-	-	0.10 ± 0.03	-	-	-	-	-
133	2-Vinylanisole	1136	1135	0.05 ± 0.01	-	-	tr	-	-	-	-	0.07 ± 0.02	tr	-	-	-	-	0.14 ± 0.04	-
135	<i>cis-p</i> -Mentha-2,8-dien-1-ol	1139	1138	-	-	-	-	-	-	-	0.18 ± 0.05	-	tr	0.05 ± 0.01	-	-	-	-	tr
136	<i>neo</i> -allo-Ocimene	1142	1145	-	-	-	-	-	-	-	-	0.15 ± 0.04	-	tr	-	-	-	0.14 ± 0.04	-
137	<i>exo</i> -Isocitral	1146	1144	-	-	-	-	-	-	-	-	-	-	-	-	-	-	tr	-
138	<i>trans</i> -Pinocarveol	1142	1141	-	-	-	-	-	-	-	-	tr	-	-	-	-	-	-	-
139	<i>trans-p</i> -Mentha-2-en-1-ol	1144	1139	-	tr	-	-	-	-	-	-	tr	tr	-	-	-	-	tr	-
140	Camphor	1145	1149	-	-	-	-	0.2 ± 0.05	-	-	-	tr	tr	-	-	0.28 ± 0.07	0.35 ± 0.09	-	-
141	Isopulegol	1149	1149	-	-	-	-	0.1 ± 0.03	-	-	-	-	-	-	-	-	-	0.23 ± 0.06	-
142	Citronellal	1151	1152	-	-	-	-	-	-	-	-	tr	tr	-	-	-	-	-	-
144	Camphene hydrate	1156	1158	-	-	-	-	-	-	-	-	-	-	0.19 ± 0.05	-	-	-	-	-
146	Pinocarvone	1161	1164	tr	0.06 ± 0.02	0.08 ± 0.02	-	tr	-	-	-	tr	tr	-	0.12 ± 0.03	tr	tr	0.17 ± 0.04	-

149	Linalool ethyl ether	1167	1166	tr	tr	-	-	tr	-	-	-	tr	tr	-	tr	0.21 ± 0.05	0.05 ± 0.01	tr	-
150	(2E)-Nonenol	1169	1170	-	-	-	-	tr	-	-	-	-	tr	-	-	-	-	-	-
152	trans-β-Terpineol	1171	1169	-	-	-	-	-	-	-	-	-	-	0.24 ± 0.06	-	-	-	-	-
154	Nonanol	1175	1176	-	-	-	-	tr	0.09 ± 0.02	-	-	-	-	-	-	-	tr	0.07 ± 0.02	-
156	cis-Pinocamphone	1177	1176	0.08 ± 0.02	-	-	-	-	-	-	-	tr	-	-	-	-	-	-	-
158	Isogeranial	1179	1185	tr	0.08 ± 0.02	-	-	-	-	-	-	-	-	-	-	-	-	-	-
161	4'-Methyl acetophenone	1184	1188	-	0.32 ± 0.08	-	tr	0.13 ± 0.03	-	0.05 ± 0.01	-	tr	tr	tr	-	-	tr	0.05 ± 0.01	-
162	Butyl hexanoate	1189	1193	-	0.3 ± 0.08	-	-	-	-	-	-	-	-	-	-	-	-	-	-
163	2-Decanone	1192	1196	0.22 ± 0.06	-	-	-	-	tr	-	-	-	-	0.12 ± 0.03	-	-	-	tr	tr
166	(Z)-Dihydrocarvone	1197	1198	-	-	-	-	tr	-	-	-	-	-	-	-	-	-	-	-
168	γ-Terpineol	1200	1200	-	-	-	-	-	-	-	-	0.15 ± 0.04	tr	-	-	-	-	-	-
169	cis-Piperitol	1201	1203	-	-	-	-	-	-	0.06 ± 0.02	-	-	-	-	tr	-	-	-	-
174	trans-Piperitol	1212	1209	-	-	tr	-	-	tr	-	-	tr	-	tr	-	-	-	-	-
175	trans-Pulegol	1214	1212	-	tr	-	-	-	-	-	-	-	tr	0.06 ± 0.02	-	-	-	-	-
176	Verbenone	1216	1208	-	tr	-	-	tr	-	-	-	-	tr	-	-	-	tr	-	-
177	(2Z)-(3,3-dimethyl-cyclohexylidene)	1221	1227	-	tr	-	-	0.06 ± 0.02	-	-	-	tr	tr	-	-	-	tr	-	-
178	trans-Carveol	1223	1223	-	-	-	-	tr	-	0.07 ± 0.02	-	tr	tr	-	-	tr	tr	-	-
179	Nerol	1224	1228	tr	-	-	0.07 ± 0.03	tr	0.08 ± 0.02	0.10 ± 0.03	-	tr	tr	-	-	0.16 ± 0.04	0.17 ± 0.04	-	-
181	3,7-Dimethyl-7-octen-1-ol	1236	1240	-	-	-	-	tr	-	0.07 ± 0.02	-	0.07 ± 0.02	tr	tr	tr	tr	tr	-	0.10 ± 0.03
182	Hexyl 2-methylbutanoate	1237	1239	0.12 ± 0.03	-	-	-	tr	-	0.06 ± 0.02	-	-	tr	0.18 ± 0.05	tr	-	0.06 ± 0.02	-	-
183	cis-Carveol	1239	1232	-	0.06 ± 0.02	-	-	tr	-	-	-	0.06 ± 0.02	tr	-	-	-	tr	-	-

184	Neral	1240	1238	-	tr	-	-	-	-	-	0.05 ± 0.01	tr	-	tr	-	-	tr	-	-
185	Ethyl phenylacetate	1242	1246	0.06 ± 0.02	0.06 ± 0.02	0.19 ± 0.05	0.07 ± 0.03	0.09 ± 0.02	-	0.06 ± 0.02	-	tr	-	tr	tr	tr	tr	0.06 ± 0.02	0.17 ± 0.04
186	Carvone	1248	1246	-	0.05 ± 0.01	-	-	tr	-	tr	-	0.07 ± 0.02	tr	0.10 ± 0.03	-	-	tr	-	-
187	Isopentyl hexanoate	1249	1252	-	-	0.08 ± 0.02	tr	tr	-	tr	-	-	-	-	-	-	tr	-	-
188	Linalyl acetate	1250	1250	-	tr	-	-	tr	-	-	0.07 ± 0.02	tr	-	-	-	-	-	-	-
191	<i>trans</i> -Myrtanol	1262	1270	tr	tr	-	-	tr	-	-	-	0.06 ± 0.02	tr	0.10 ± 0.03	-	0.07 ± 0.02	0.06 ± 0.02	0.06 ± 0.02	-
192	(2 <i>E</i>)-Decenal	1263	1265	0.08 ± 0.02	-	tr	tr	tr	-	-	-	tr	tr	-	-	-	tr	0.06 ± 0.02	-
193	<i>cis</i> -Verbenyl acetate	1268	1278	tr	0.13 ± 0.03	-	0.10 ± 0.05	tr	-	-	-	tr	-	-	-	-	-	0.12 ± 0.03	-
194	Geranial	1270	1268	tr	tr	-	-	tr	-	-	-	-	tr	-	-	-	0.06 ± 0.02	0.11 ± 0.03	-
195	Decyl alcohol	1273	1278	tr	0.21 ± 0.05	-	0.14 ± 0.07	0.28 ± 0.07	-	-	tr	0.06 ± 0.02	0.07 ± 0.02	tr	tr	tr	tr	0.10 ± 0.03	0.08 ± 0.02
197	Methyl nerate	1275	1276	0.19 ± 0.05	-	-	0.05 ± 0.02	-	tr	-	-	-	-	-	-	-	-	-	0.10 ± 0.03
198	(2 <i>Z</i>)-Decenal	1276	1265	-	0.11 ± 0.03	0.05 ± 0.01	-	-	-	-	-	-	-	-	-	-	tr	-	-
199	Perillaldehyde	1277	1278	-	-	-	-	-	-	-	0.10 ± 0.03	0.13 ± 0.03	0.09 ± 0.02	0.11 ± 0.03	-	-	tr	-	-
200	Citronellyl formate	1278	1275	-	0.06 ± 0.02	-	-	-	-	-	0.07 ± 0.02	tr	tr	-	-	-	tr	-	-
201	Lavandulyl acetate	1284	1284	-	tr	-	-	0.07 ± 0.02	-	-	-	0.07 ± 0.02	tr	0.08 ± 0.02	-	-	0.15 ± 0.04	-	-
203	Thymol	1288	1293	0.16 ± 0.04	0.06 ± 0.02	tr	-	-	-	-	-	-	-	-	-	-	-	-	0.06 ± 0.02
204	Limonen-10-ol	1289	1290	tr	0.07 ± 0.02	-	-	tr	-	-	-	-	-	-	-	-	tr	-	-
206	Propyl octanoate	1292	1293	tr	-	-	-	tr	-	-	-	-	tr	0.24 ± 0.06	-	-	tr	-	-
210	<i>n</i> -Undecanal	1306	1309	-	-	-	-	tr	tr	-	0.07 ± 0.02	0.16 ± 0.04	0.10 ± 0.03	0.09 ± 0.02	-	-	tr	0.05 ± 0.01	0.07 ± 0.02

211	Nonyl acetate	1311	1313	-	0.11 ± 0.03	-	-	0.43 ± 0.11	-	-	0.06 ± 0.02	tr	tr	tr	-	-	-	-	-
212	4-Vinyl guaiacol	1313	1309	-	-	-	0.21 ± 0.10	-	tr	-	-	-	-	-	-	tr	-	-	-
213	(2E,4E)-Decadienal	1316	1322	-	-	-	-	-	-	-	tr	0.07 ± 0.02	-	tr	-	-	-	tr	-
215	Linalyl propionate	1331	1333	tr	-	-	-	-	tr	-	-	0.06 ± 0.02	-	-	-	-	-	-	-
216	trans-Carvyl acetate	1335	1332	-	-	-	-	-	-	-	-	tr	tr	-	-	-	tr	-	-
217	1,2-Diacetylglycerol	1344	1348	-	-	-	-	-	-	-	-	0.05 ± 0.01	-	-	-	-	-	-	-
218	Isobutyl octanoate	1347	1347	tr	-	tr	tr	-	tr	-	-	-	-	-	tr	-	-	-	-
219	α-Terpinyl acetate	1349	1349	-	-	-	-	tr	-	-	-	-	tr	-	-	-	tr	-	-
223	α-Ylangene	1373	1371	-	-	-	-	-	tr	-	-	-	-	-	0.08 ± 0.02	-	-	-	-
224	n-Decanoic acid	1378	1398	-	0.11 ± 0.03	-	-	-	-	-	-	-	0.17 ± 0.04	tr	-	-	-	-	-
226	(E)-β-Damascenone	1380	1379	-	0.06 ± 0.02	-	-	0.09 ± 0.02	-	-	0.10 ± 0.03	tr	tr	tr	-	0.07 ± 0.02	0.05 ± 0.01	-	-
233	n-Tetradecane	1400	1400	0.07 ± 0.02	tr	-	-	tr	-	-	-	-	-	tr	-	-	tr	-	-
234	Methyl eugenol	1404	1402	-	tr	-	-	-	tr	tr	-	-	tr	tr	-	-	tr	-	-
235	n-Dodecanal	1410	1410	tr	tr	tr	0.05 ± 0.02	0.06 ± 0.02	-	-	0.14 ± 0.04	0.21 ± 0.02	0.42 ± 0.11	tr	-	-	tr	-	-
237	cis-α-Bergamotene	1413	1416	-	tr	-	-	-	-	-	-	tr	tr	0.33 ± 0.08	-	-	tr	-	-
238	α-Cedrene	1419	1414	-	-	-	-	-	-	-	-	tr	tr	0.14 ± 0.04	-	-	-	-	-
240	p-Menth-1-en-9-ol acetate	1421	1426	-	-	-	-	-	-	-	-	-	-	-	-	-	tr	-	-
241	(E)-α-Ionone	1424	1421	-	-	-	-	-	-	-	-	tr	-	-	-	-	-	-	-
243	γ-Elemene	1431	1432	tr	-	-	0.06 ± 0.03	-	-	tr	-	0.06 ± 0.02	tr	-	tr	-	tr	-	0.08 ± 0.02
244	β-Copaene	1432	1433	-	-	-	-	-	-	-	-	-	0.14 ± 0.04	-	-	-	-	-	-
245	2-Undecanol, acetate	1433	1433	-	-	-	-	-	0.08 ± 0.02	-	-	-	-	-	-	-	-	-	-
247	β-Duprezianene	1435	1427	-	0.08 ± 0.02	-	-	-	-	-	-	tr	-	0.19 ± 0.05	-	-	-	-	-

248	Phenylethyl butyrate	1439	1443	-	tr	-	tr	-	-	-	0.07 ± 0.02	-	-	-	-	-	-	-	-
249	Isopentyl octanoate	1447	1446	0.06 ± 0.02	0.06 ± 0.02	0.2 0± 0.05	0.40 ± 0.19	0.14 ± 0.04	0.08 ± 0.02	0.05 ± 0.01	-	0.07 ± 0.02	tr	-	0.17 ± 0.04	tr	0.05 ± 0.01	tr	-
250	2-Methylbutyl octanoate	1448	1449	tr	-	0.1 0± 0.03	0.12 ± 0.06	0.05 ± 0.01	tr	tr	-	0.07 ± 0.02	tr	-	0.06 ± 0.02	-	tr	0.05 ± 0.01	0.07 ± 0.02
251	(E)-β-Farnesene	1453	1448	-	0.06 ± 0.02	-	0.40 ± 0.19	0.05 ± 0.01	-	-	0.08 ± 0.02	0.06 ± 0.02	tr	0.28 ± 0.07	tr	-	tr	-	-
253	β-Santalene	1460	1459	-	0.05 ± 0.01	-	-	tr	-	-	0.06 ± 0.02	0.08 ± 0.02	0.06 ± 0.02	0.19 ± 0.05	-	-	tr	-	tr
254	Geranyl propanoate	1470	1471	tr	-	-	-	-	tr	tr	0.09 ± 0.02	0.08 ± 0.02	tr	tr	-	-	tr	-	-
255	γ-Gurjunene	1472	1476	-	-	-	-	-	-	-	-	-	tr	-	-	-	0.05 ± 0.01	-	-
256	Selina-4,11-diene	1473	1476	-	-	-	-	-	-	-	-	0.10 ± 0.03	tr	-	-	-	-	-	-
257	γ-Curcumene	1475	1482	-	-	-	-	0.18 ± 0.05	-	-	-	0.06 ± 0.02	tr	0.12 ± 0.03	-	-	-	0.07 ± 0.02	-
258	Germacrene D	1479	1480	0.09 ± 0.02	-	-	0.14 ± 0.07	-	-	-	-	-	-	0.05 ± 0.01	-	-	-	-	-
259	α-Curcumene	1481	1480	tr	-	-	-	-	-	-	-	-	-	-	-	-	tr	0.22 ± 0.06	-
260	γ-Muurolene	1483	1478	-	-	-	-	-	0.49 ± 0.12	-	-	-	-	-	-	-	-	-	-
261	α-Amorphene	1484	1482	-	-	-	-	-	0.09 ± 0.02	-	-	-	-	-	-	-	-	-	-
262	trans-β-Bergamotene	1485	1482	-	-	-	0.05 ± 0.02	tr	0.05 ± 0.01	-	-	0.11 ± 0.03	tr	0.05 ± 0.01	0.07 ± 0.02	-	-	-	-
264	β-Selinene	1494	1492	-	-	-	0.14 ± 0.07	-	-	-	-	-	-	-	-	-	-	0.18 ± 0.05	-
265	α-Zingiberene	1497	1496	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.26 ± 0.07	-
266	Bicyclogermacrene	1497	1497	-	-	-	-	tr	-	-	-	0.07 ± 0.02	0.07 ± 0.02	0.12 ± 0.03	-	-	tr	-	-
267	n-Pentadecane	1499	1500	-	-	-	-	-	0.31 ± 0.08	tr	-	0.10 ± 0.03	-	-	-	-	tr	-	-
270	α-Selinene	1502	1501	0.14 ± 0.04	-	-	0.14 ± 0.07	-	0.22 ± 0.06	-	-	-	-	-	-	-	-	0.22 ± 0.06	-

296	β -Atlantol	1610	1611	-	0.05 \pm 0.01	-	-	tr	-	-	-	-	tr	-	-	-	tr	-	-	
297	Humulol	1613	1611	-	-	-	-	-	0.14 \pm 0.04	-	-	-	-	-	-	-	-	-	-	
298	Humulene epoxide II	1618	1613	-	-	-	-	-	tr	-	-	-	-	-	-	-	-	-	-	
299	Junenol	1622	1619	tr	-	-	-	tr	-	-	-	0.11 \pm 0.03	tr	-	-	-	tr	-	-	
300	Epicubenol	1632	1631	tr	0.07 \pm 0.02	-	0.06 \pm 0.03	tr	0.08 \pm 0.02	-	0.13 \pm 0.03	0.06 \pm 0.02	tr	-	tr	-	tr	-	-	
301	γ -Eudesmol	1633	1632	tr	tr	-	-	tr	-	-	tr	0.06 \pm 0.02	tr	-	-	-	tr	0.05 \pm 0.01	-	
302	Isoamyl decanoate	1644	1644	0.07 \pm 0.02	tr	tr	0.12 \pm 0.06	0.07 \pm 0.02	tr	tr	-	0.06 \pm 0.02	-	-	0.10 \pm 0.03	-	tr	tr	0.05 \pm 0.01	
303	Cadin-4-en-10-ol	1649	1650	-	-	-	-	tr	0.07 \pm 0.02	tr	0.17 \pm 0.04	-	tr	-	tr	-	tr	tr	-	
304	<i>epi</i> - β -Bisabolol	1672	1675	-	-	-	-	-	-	-	0.06 \pm 0.02	-	tr	-	-	-	tr	tr	-	
305	β -Sinensal	1697	1701	-	-	-	-	-	-	-	-	-	tr	-	-	-	-	0.08 \pm 0.02	-	
306	<i>n</i> -Heptadecane	1700	1700	-	-	-	-	-	-	-	-	0.08 \pm 0.02	tr	-	-	-	-	tr	-	
307	α -Sinensal	1753	1749	-	-	-	tr	-	-	-	-	-	tr	-	tr	-	-	-	-	
308	Ethyl tetradecanoate	1795	1794	tr	tr	0.06 \pm 0.02	0.08 \pm 0.04	0.05 \pm 0.01	-	0.06 \pm 0.02	-	-	-	-	0.15 \pm 0.04	-	tr	0.11 \pm 0.03	0.17 \pm 0.04	
309	Farnesyl acetate	1824	1832	-	0.1 \pm 0.03	-	-	tr	-	-	tr	-	-	-	-	-	-	-	-	
310	(5 <i>E</i> ,9 <i>E</i>)-Farnesyl acetone	1912	1915	-	-	-	-	0.07 \pm 0.02	-	-	-	-	-	-	-	-	-	-	-	
311	Ethyl 9-hexadecenoate	1961		-	-	-	-	0.08 \pm 0.02	-	-	-	-	-	-	-	-	-	-	-	
312	Ethyl palmitate	1984	1993	0.15 \pm 0.04	-	0.14 \pm 0.04	0.12 \pm 0.06	-	-	0.10 \pm 0.03	-	-	tr	-	0.11 \pm 0.03	-	0.05 \pm 0.01	0.07 \pm 0.02	0.07 \pm 0.02	
313	Ethyl linoleate	2160	2164	-	-	-	-	tr	-	-	-	tr	-	-	tr	-	-	tr	-	
314	Ethyl linolenate	2166	2165	-	-	-	-	tr	-	-	-	-	-	tr	-	-	tr	-	-	
Tot					5.40 \pm	5.60 \pm	2.46 \pm	3.16 \pm	5.63 \pm	4.04 \pm	1.45 \pm	6.47 \pm	6.39 \pm	1.90 \pm	4.44 \pm	1.47 \pm	1.88 \pm	3.63 \pm	4.42 \pm	1.62 \pm
					1.36	1.40	0.62	1.53	1.42	1.02	0.36	1.61	1.61	0.48	1.10	0.37	0.47	0.84	1.11	0.41

The compounds number is reported in order of elution, considering the total number of compounds eluted. For the identification of the other compounds not reported see Table 1.

Table S2. Major volatile constituents of citrus essential oils, expressed in area% as average of three measurements.

n.	Compound	LRL _{lib}	Sicilian Blond orange	Sicilian Red orange	Calabrian Bergamot	Sicilian Lemon	Sicilian yellow mandarin	Sicilian green mandarin
1	α -Thujene	927			0.71 \pm 0.06	1.28 \pm 0.12	2.24 \pm 0.20	2.16 \pm 0.19
2	α -Pinene	933	2.01 \pm 0.18	2.03 \pm 0.18	2.02 \pm 0.18	3.29 \pm 0.30	4.45 \pm 0.40	4.27 \pm 0.39
3	Sabinene	972	1.89 \pm 0.17	1.66 \pm 0.15	2.19 \pm 0.20	4.53 \pm 0.41		0.85 \pm 0.08
4	β -Pinene	978			5.84 \pm 0.53	11.04 \pm 1.00	3.69 \pm 0.33	3.64 \pm 0.33
5	Myrcene	991	6.15 \pm 0.55	6.24 \pm 0.56	2.18 \pm 0.20	3.44 \pm 0.31	4.56 \pm 0.41	4.39 \pm 0.40
6	<i>n</i> -Octanal	1006	2.11 \pm 0.19	0.60 \pm 0.05				
7	δ -3-Carene	1009	0.86 \pm 0.08	1.3 \pm 0.12				
8	α -Terpinene	1018				0.90 \pm 0.08	1.59 \pm 0.14	0.97 \pm 0.09
9	<i>p</i> -Cymene	1025			0.75 \pm 0.07			
10	Limonene	1030	76.37 \pm 6.89	78.13 \pm 7.05	23.56 \pm 2.12	39.7 \pm 3.58	50.41 \pm 4.55	47.74 \pm 4.31
11	(<i>E</i>)- β -Ocimene	1046			0.71 \pm 0.06	0.65 \pm 0.06		
12	γ -Terpinene	1058			7.90 \pm 0.71	10.79 \pm 0.97	19.14 \pm 1.73	19.29 \pm 1.74
13	<i>n</i> -Octanol	1073	0.34 \pm 0.03					
14	Terpinolene	1086			1.02 \pm 0.09	1.44 \pm 0.13	2.64 \pm 0.24	2.70 \pm 0.24
15	Linalool	1101	1.94 \pm 0.17	1.57 \pm 0.14	11.44 \pm 1.03			
16	α -Terpineol	1196	0.37 \pm 0.03		0.67 \pm 0.06	0.79 \pm 0.07		1.02 \pm 0.09
17	<i>n</i> -Decanal	1208	1.78 \pm 0.16	0.81 \pm 0.07				
18	Neral	1238				2.16 \pm 0.19		
19	Linalyl acetate	1250			24.04 \pm 2.17			
20	Geranial	1268	0.5 \pm 0.05		0.98 \pm 0.09	3.10 \pm 0.28		
21	α -Terpinyl acetate	1349			0.98 \pm 0.09			
22	Neryl acetate	1361			1.85 \pm 0.17	1.78 \pm 0.16		
23	Geranyl acetate	1380			1.49 \pm 0.13	1.77 \pm 0.16		
24	Methyl, N-methyl-anthranilate	1410					1.63 \pm 0.15	2.09 \pm 0.19
25	(<i>E</i>)-Caryophyllene	1424			1.44 \pm 0.13	0.95 \pm 0.09		
26	<i>trans</i> - α -Bergamotene	1432			1.36 \pm 0.12	1.48 \pm 0.13		
27	Valencene	1492	0.36 \pm 0.03	0.88 \pm 0.08				
28	(<i>E,E</i>)- α -Farnesene	1504						0.73 \pm 0.07
29	β -Bisabolene	1508			1.95 \pm 0.18	2.08 \pm 0.19		
30	α -Sinensal	1749					1.32 \pm 0.12	1.39 \pm 0.13
Tot			94.68 \pm 8.54	93.22 \pm 8.41	93.08 \pm 8.39	91.17 \pm 8.22	91.67 \pm 8.27	91.24 \pm 8.23

Table S3. Major volatile constituents of citrus juice and citrus non alcoholic beverages, expressed in area% as average of three measurements.

n.	Compound	LRL _{lib}	Sicilian Blond orange	Sicilian Red orange	Calabrian Bergamot	Sicilian Lemon	Sicilian mandarin	Mandarin non alcoholic beverage	Bergamot non alcoholic beverage
1	α -Thujene	927					1.37 \pm 0.12		
2	α -Pinene	933	0.90 \pm 0.08	0.70 \pm 0.06	1.59 \pm 0.14		2.81 \pm 0.25		
3	β -Pinene	978	0.92 \pm 0.08	2.03 \pm 0.18	0.77 \pm 0.07		3.36 \pm 0.30		
4	Myrcene	991	6.66 \pm 0.60	6.60 \pm 0.60	4.96 \pm 0.45	6.54 \pm 0.59	5.12 \pm 0.46	3.82 \pm 0.34	2.47 \pm 0.22
5	<i>n</i> -Octanal	1006						0.83 \pm 0.07	
6	α -Terpinene	1018					2.05 \pm 0.18	0.68 \pm 0.06	
7	<i>p</i> -Cymene	1025			1.33 \pm 0.12	4.42 \pm 0.40	0.79 \pm 0.07	2.08 \pm 0.19	2.37 \pm 0.21
8	Limonene	1030	78.65 \pm 7.09	54.35 \pm 4.90	42.14 \pm 3.80	40.04 \pm 3.61	52.69 \pm 4.75	41.61 \pm 3.75	22.03 \pm 1.99
9	(<i>Z</i>)- β -Ocimene	1035							1.90 \pm 0.17
10	(<i>E</i>)- β -Ocimene	1046		2.44 \pm 0.22	1.64 \pm 0.15		1.06 \pm 0.10		3.15 \pm 0.28
11	γ -Terpinene	1058	3.02 \pm 0.27	6.53 \pm 0.59	9.72 \pm 0.88	10.91 \pm 0.98	19.97 \pm 1.80	23.18 \pm 2.09	8.56 \pm 0.77
12	Terpinolene	1086	0.73 \pm 0.07	0.72 \pm 0.06	1.09 \pm 0.10	1.41 \pm 0.13	3.34 \pm 0.30	3.50 \pm 0.32	1.70 \pm 0.15
13	Linalool	1101	1.08 \pm 0.10	4.29 \pm 0.39	5.22 \pm 0.47	2.58 \pm 0.23			8.20 \pm 0.74
14	<i>n</i> -Nonanal	1107				1.43 \pm 0.13			
15	Fenchyl alcohol	1123							0.96 \pm 0.09
16	<i>cis</i> -Ocimenol								0.94 \pm 0.08
17	Terpinen-4-ol	1184				3.77 \pm 0.34		1.36 \pm 0.12	1.87 \pm 0.17
18	α -Terpineol	1196			2.51 \pm 0.23	6.84 \pm 0.62	0.72 \pm 0.06	8.69 \pm 0.78	16.64 \pm 1.50
19	Safranal	1206				1.40 \pm 0.13			
20	<i>n</i> -Decanal	1208					0.72 \pm 0.06	0.97 \pm 0.09	
21	Octyl acetate	1214			2.70 \pm 0.24				
22	Linalyl acetate	1250		8.40 \pm 0.76	2.95 \pm 0.27				0.38 \pm 0.03
23	Geraniol	1255			0.83 \pm 0.07				1.40 \pm 0.13
24	α -Terpinyl acetate	1349			0.72 \pm 0.06				1.81 \pm 0.16
25	Neryl acetate	1361		0.99 \pm 0.09	1.91 \pm 0.17	2.02 \pm 0.18			4.07 \pm 0.37
26	Geranyl acetate	1380		1.69 \pm 0.15	2.28 \pm 0.21	1.16 \pm 0.10			5.28 \pm 0.48
27	Methyl, N-methyl-anthranilate	1410					1.19 \pm 0.11	2.41 \pm 0.22	
28	(<i>E</i>)-Caryophyllene	1424			2.92 \pm 0.26	1.74 \pm 0.16		1.50 \pm 0.14	2.41 \pm 0.22
29	<i>trans</i> - α -Bergamotene	1432			3.97 \pm 0.36	3.02 \pm 0.27			2.16 \pm 0.19
30	Valencene	1492	1.53 \pm 0.14	1.71 \pm 0.15		1.18 \pm 0.11			
31	(<i>Z</i>)- α -Bisabolene	1503			0.82 \pm 0.07				
32	(<i>E,E</i>)- α -Farnesene	1504						1.54 \pm 0.14	
33	β -Bisabolene	1508			4.67 \pm 0.42	3.94 \pm 0.36			3.55 \pm 0.32
Tot			93.49 \pm 8.43	90.45 \pm 8.16	94.74 \pm 8.54	92.40 \pm 8.33	95.19 \pm 8.58	92.17 \pm 8.31	91.85 \pm 8.28

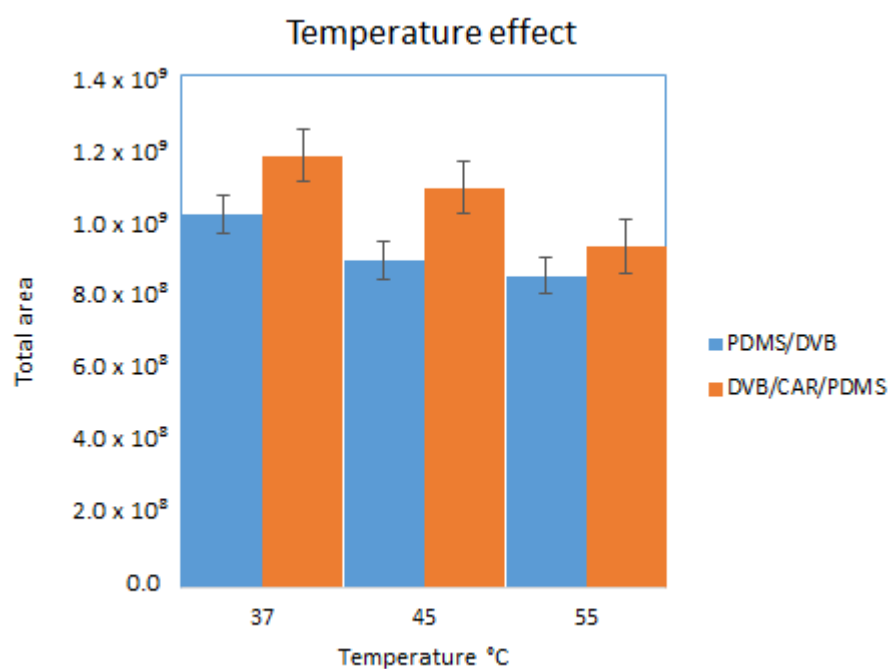


Figure S1. Evaluation of the temperature influence on SPME method extraction optimization \pm SD.

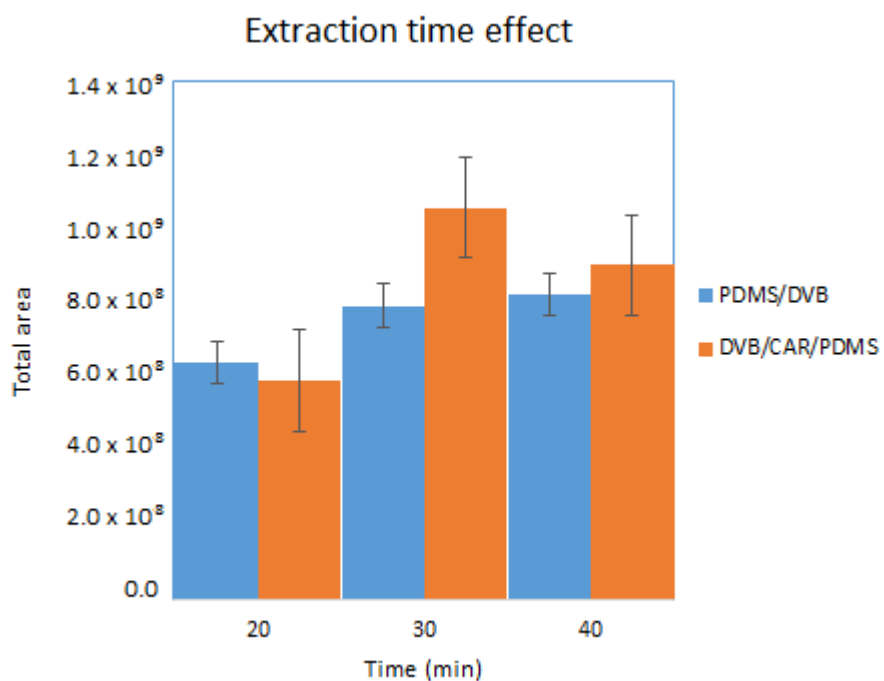


Figure S2. Evaluation of time influence on SPME method extraction optimization \pm SD.

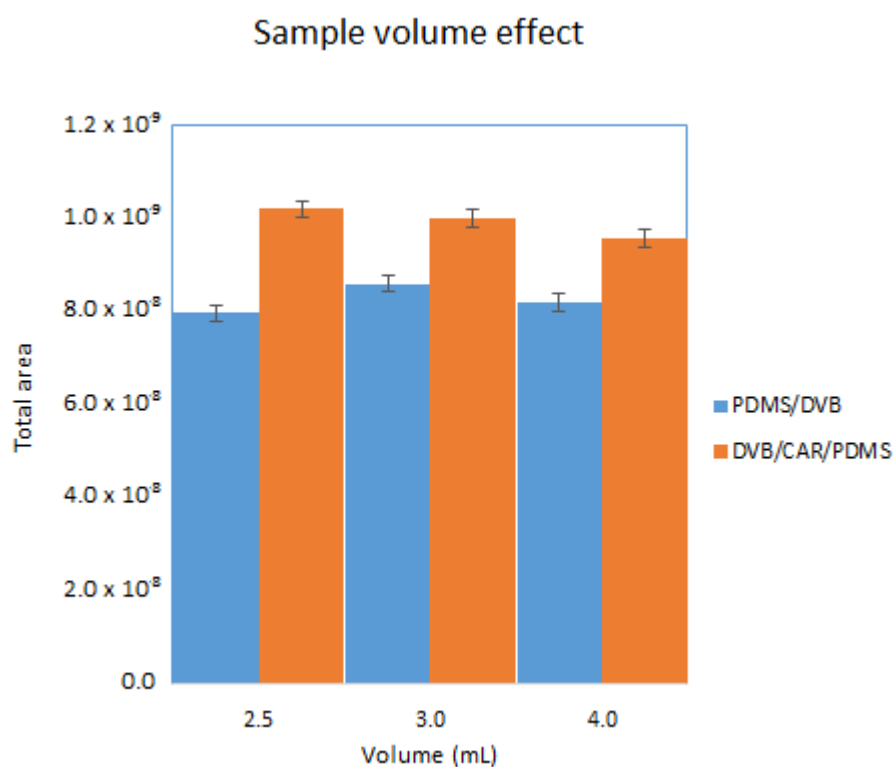


Figure S3. Evaluation of sample volume influence on SPME method extraction optimization \pm SD.

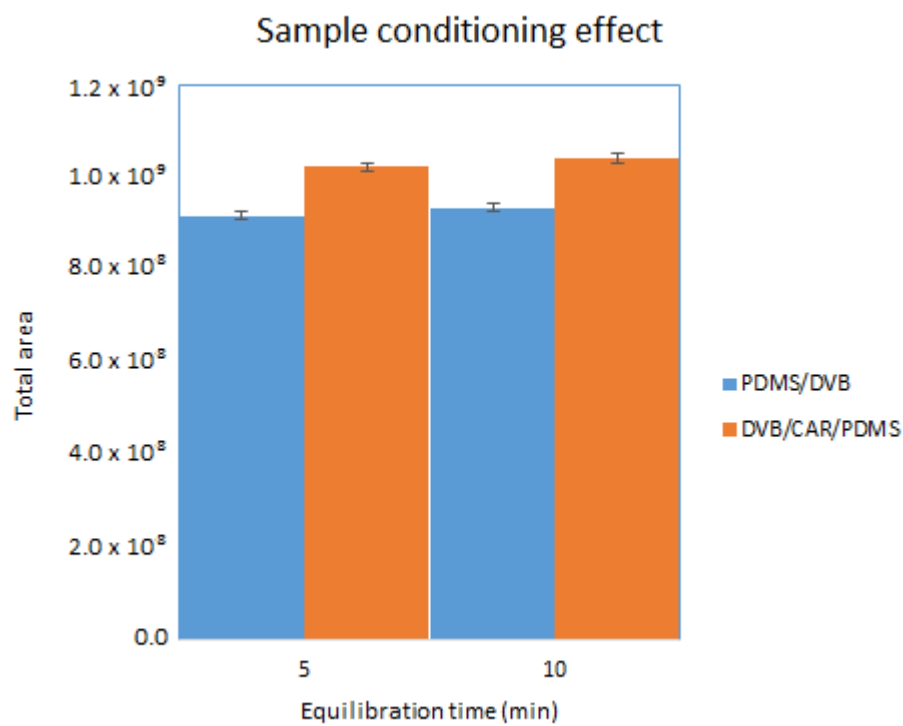


Figure 4. Evaluation of sample time conditioning influence on SPME method extraction optimization \pm SD.

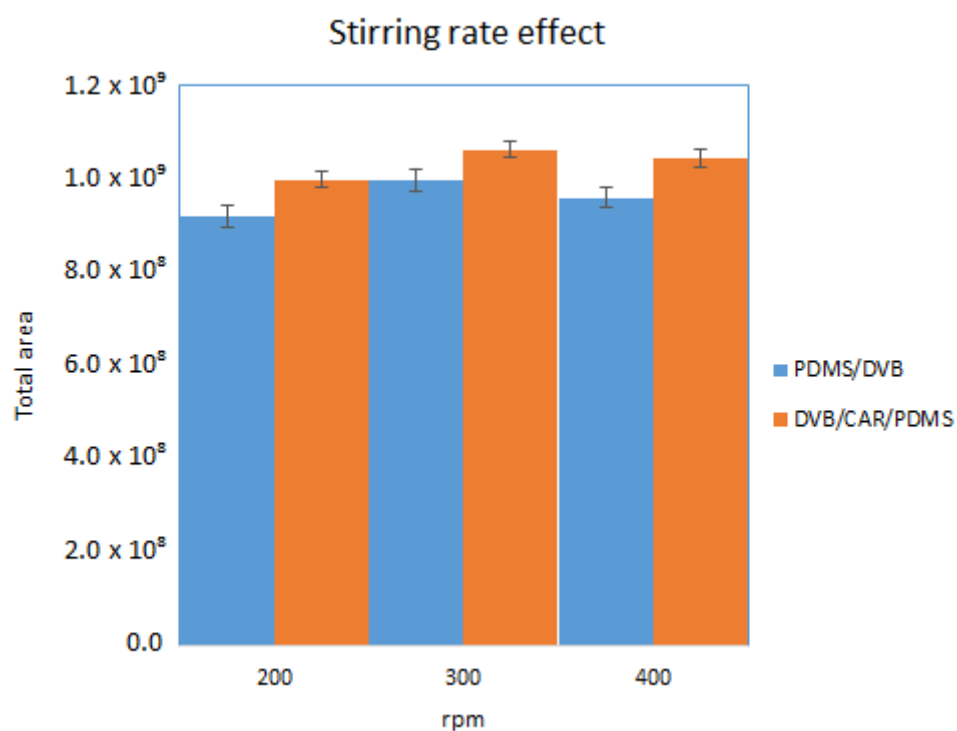


Figure S5. Evaluation of stirring rate influence on SPME method extraction optimization \pm SD.

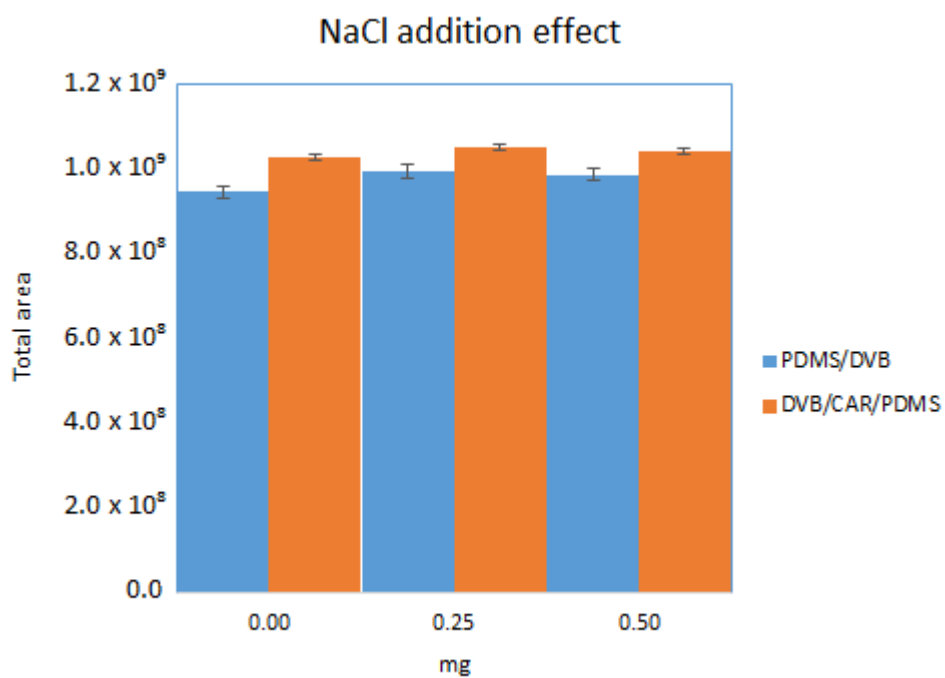


Figure S6. Evaluation of NaCl addition influence on SPME method extraction optimization \pm SD.

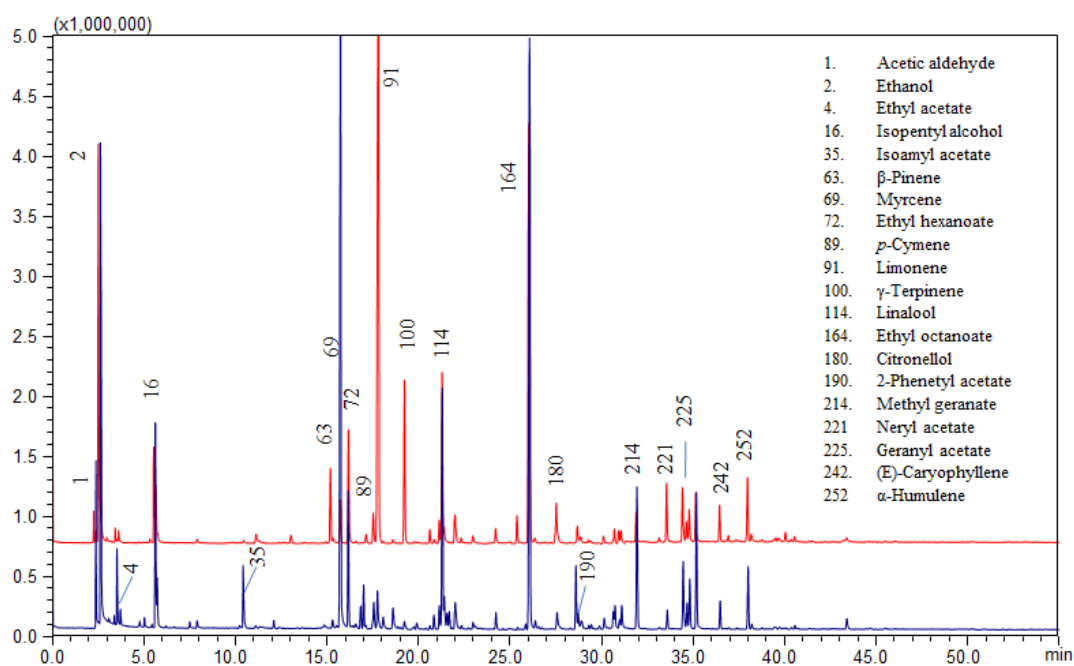


Figure S7. Comparison between volatile profile of sample 1 (in blue) and sample 1 spiked with 1.8 % lemon peel (in red).

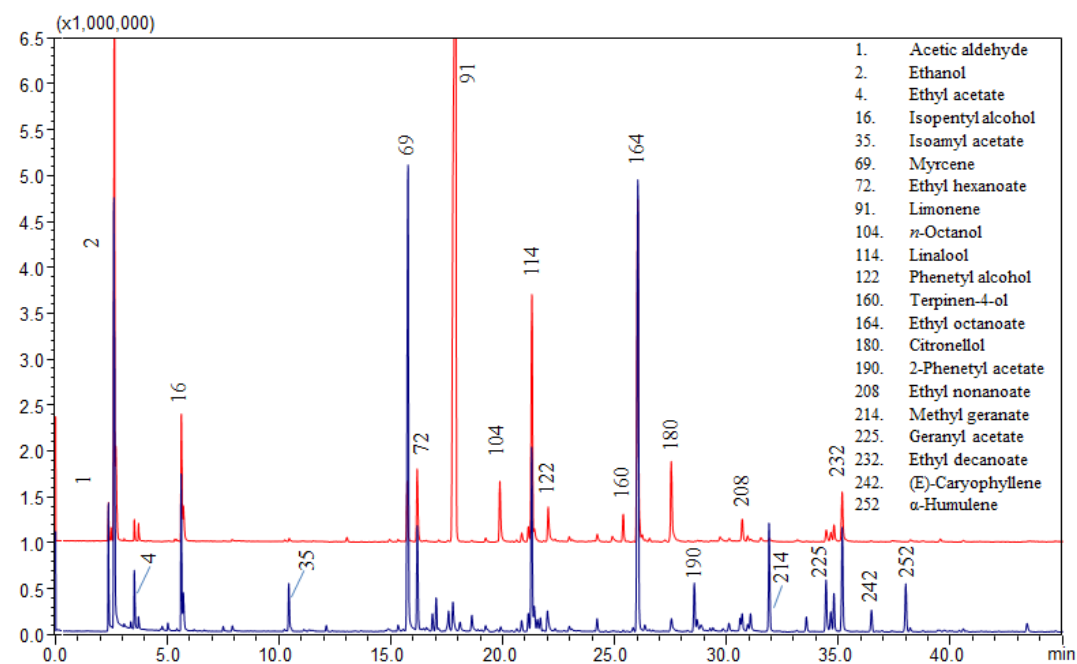


Figure S8. Comparison between volatile profile of sample 1 (in blue) and sample 1 spiked with 1.5 % orange peel (in red).

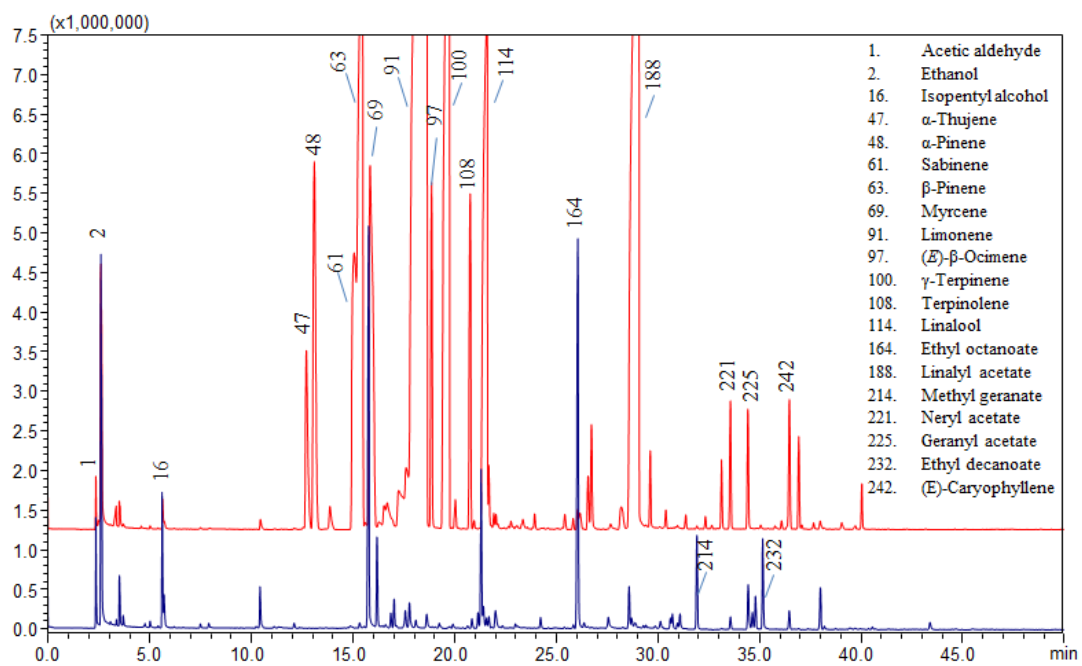


Figure S9. Comparison between volatile profile of sample 1 (in blue) and sample 1 spiked with 0.3 % bergamot E.O. (in red).