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ORIGINAL ARTICLE

Assessment of volatile compounds and sensory characteristics of Mexican hibiscus (*Hibiscus sabdariffa* L.) calyces hot beverages

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Abstract This study was conducted to assess and correlate the sensory characteristics and volatile compounds of hot beverages from the calyces of four Mexican varieties of hibiscus (4Q4, Puebla Precoz, UAN 16-1, and Sudan). A panel of 10 judges, detected six flavour descriptors in all samples. Sensory studies revealed highly characteristic flavour profiles of these varieties. In order to obtain the extracts and further characterize the odour-active volatiles of the studied beverages, a simultaneous steam distillation and solvent extraction procedure followed by a GC–MS analysis was employed. A total of 104 volatile compounds were identified in all samples. By determining the odour activity values (OAVs) it was possible to identify compounds with high odor-activity in the beverages, such as: 2-furfural, 5-methyl-2-furfural, hexanal, (E)-2-hexenal, (Z)-3-hexen-1-ol, 1-octen-3-one, 1-octen-3-ol, 5-methyl-2(3H)-furanone, phenylacetaldehyde, nonanal, (E)-2-nonenal, geranylacetone, α-ionone and β-ionone. Moreover, on the basis of their OAVs, the differences in odour profiles of beverages were predominately due to these odorants.

Keywords Jamaica · *Hibiscus sabdariffa* · Volatiles · Gas chromatography-mass spectrometry · Odour activity value

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Introduction

The hibiscus plant (*Hibiscus sabdariffa* L.), an annual crop bush, also known as 'absina', 'Jamaica' or 'roselle', pertains to the family Malvaceae (Diaz et al. 2009) and has been widely utilized in various countries for culinary and medicinal purposes (Da-Costa-Rocha et al. 2014; Patel 2014). The calyces, due to its high concentration of acids, vitamin C and anthocyanins are the main part utilized of this plant. In Mexico, fresh and dried hibiscus calyces are used to prepare both cold and hot brilliantly red beverages (Cisse et al. 2009). Due to its attractive red colour and slightly tart flavour, the hibiscus beverage has great potential for the food industry, such as a delicious beverage with health benefits. Among its medicinal properties, the hibiscus has demonstrated both hypotensive activity and the ability to reduce inflammation as occurs in chronic inflammatory diseases (Da-Costa-Rocha et al. 2014; Patel 2014).

On the other hand, flavour plays a very important role in the sensory evaluation of foods and therefore is considered an important parameter of quality for consumers. Hibiscus flavour is a combination of both sweet and tart, similar to cranberry (Wong et al. 2003; Pino et al. 2006; Ramírez et al. 2010).

There are several works pertaining to the health benefits of hibiscus calyces, but few studies have investigated volatile compounds in hibiscus hot beverages (Chen et al. 1998; Pino et al. 2006; Ramírez-Rodrigues et al. 2012). Ramírez et al. (2010) found 2-furfural and 5-methyl-2-furfural in both hot and cold beverages prepared from dried hibiscus, while those prepared using fresh hibiscus were rich in linalool and 2-ethylhexan-1-ol. However, these results are potentially incomplete since the isolation method employed was solid-phase microextraction with

only one fibre type, which suggest that isolated volatiles might not necessarily be the most odour-active compounds (Pino 2013). Those sensory screening strategies require complementation by means of quantitative data and by the estimation of threshold values in order to calculate odour activity values (OAVs), aiming at obtaining a more realistic ranking of the odorants potentially more relevant in the beverage.

The aim of this work has been to characterize the volatile compounds and its relation to the sensory characteristics of hot beverages from four varieties (4Q4, Puebla Precoz, UAN 16-1, and Sudan) of hibiscus cultivated in Mexico.

Materials and methods

Samples

Air-dried (70 °C) hibiscus calyces (Hahm et al. 2011) were obtained from the experimental field of the Universidad Autónoma de Nayarit, Mexico during the 2015 winter season (November–December). Four varieties were evaluated: 4Q4, Puebla Precoz, UAN 16-1, and Sudan. Samples were stored in airtight opaque containers at 5 °C.

Sensory analysis

Dried hibiscus calyces were mixed with distilled water in a ratio of 1:4 w/w and extracted at 98 °C for 30 min without stirring in a beaker covered with a watch glass. After extraction, the beverages were immediately decanted and served hot for sensory analysis. The aroma profile of hibiscus calyces hot beverages was evaluated by ten panellists and sensory lexicon was arrived at by consensus (Lawless and Heymann 2010). Sensory attributes were discussed by judges during these sessions in order to accomplish a consensus of standardized assessment procedure and to select appropriate reference stimuli. Each beverage was orthonasally evaluated by quantitative descriptive analysis. Panellists rated the six descriptive sensory attributes in the overall aroma of the beverage on a continuous scale from 0 (not detectable) to 15 (intensely detectable).

Simultaneous distillation-solvent extraction

Simultaneous distillation-solvent extraction with a Likens–Nickerson apparatus procedure was utilized to mimic the preparation of hibiscus hot water infusion (Pino et al. 2006). After addition of an internal standard (methyl nonanoate, 5 mg), dried calyces (200 g) were blended with distilled water (600 mL) and simultaneously distilled and extracted for 1 h with 40 mL of dichloromethane. The volatile concentrate was dried over anhydrous sodium sulfate and concentrated to 0.6 mL on a Kuderna–Danish evaporator with a 12-cm Vigreux column and further evaporated to 0.2 mL with a gentle nitrogen stream. Extracts were stored in sealed amber vials at 4 °C until analysis.

GC–FID and GC–MS analyses

GC–FID analysis was performed on a Perkin Elmer Autosystem XL (Shelton, CT, USA) gas chromatograph with a flame ionization detector. Injection was on split mode (ratio 1:50) at 250 °C. Separation was carried out on AT-5 ms (30 m × 0.25 mm, 0.5 µm; Alltech, Waukegan. IL, USA) or DB-Wax column (30 m × 0.25 mm, 0.25 µm; J&W Scientific, Folsom, CA, USA) columns. Initial oven temperature was 50 °C (2 min) and then increased (4 °C/min) to 250 °C (10 min). The carrier gas utilized was helium at a flow-rate of 1 mL/min. The retention times of a series of *n*-alkanes (C_8–C_{32}) was used to calculate the retention indices for all identified compounds and for reference standards. Concentrations were expressed as mg methyl nonanoate equivalents kg^{-1} of dry weight, response factors being taken as 1.0 for all compounds with reference to the internal standard and a recovery factor of 70% at least. All analyses were replicated twice.

GC–MS analyses were performed utilizing a Perkin Elmer Clarus 500 (Shelton, CT, USA) gas chromatograph coupled to a Perkin Elmer Clarus 500 MSD (Shelton, CT, USA). The chromatographic conditions were the same as in GC–FID. Mass spectrometer parameters were as follows: electron impact mode at 70 eV; acquisition range, m/z 30–400 u; interface and ion source temperatures were 250 °C. Identification of volatile compounds were performed by comparing their linear temperature retention indices (LRIs) and mass spectra with authentic standards from Sigma-Aldrich (St. Louis, MO, USA), Fluka (Buchs, Switzerland), and others were supplied by Dallant (Barcelona, Spain). Tentative identification of compounds for which it was not possible to locate reference compounds was achieved by comparison of their mass spectra with spectral data from commercial libraries (NIST 02, Wiley 275, Palisade 600) and our specific library for volatile compounds (Flavorlib). Experimental LRIs were also compared with those reported in the literature (Adams 2001) and with standards when possible.

Odour thresholds

Odour thresholds were determined by a panel of 20–25 trained panellists recruited from the Food Industry Research Institute, Havana, Cuba. The ASTM procedure

for the determination of odour and thresholds by a forced-choice ascending concentration series method was used (ASTM E679-04 2004). Odour activity value (OAV) was calculated by dividing the concentration with the threshold value of the compound in water.

Statistical analysis

Selection of judges was conducted by means of sequential analysis of test results from each candidate. An unstructured scale method was applied for evaluating every attribute in each studied variety. A variance analysis with a statistical design of randomized complete blocks was utilized.

Results and discussion

Hot beverages for each of the four Mexican hibiscus varieties were evaluated by trained panelists utilizing quantitative descriptive analysis (Table 1). Results demonstrated distinct differences between the varieties. The flavor of Sudan beverage had the highest acid note ($p < 0.05$), followed by Puebla Precoz > UAN 16-1 > 4Q4. The flavor of Sudan beverage also possessed significantly higher astringent notes ($p < 0.05$), followed by Puebla Precoz and UAN 16-1, and 4Q4 with the lowest astringency. Puebla Precoz variety produces a hot beverage with a similar floral note to those produced by 4Q4 and UAN 16-1, but significantly higher ($p < 0.05$) than Sudan beverage. The sensory profile of UAN 16-1 beverage presented notably higher herbal and caramel notes ($p < 0.05$). The floral note was significantly higher in the beverages produced by UAN 16-1, Puebla Precoz and 4Q4, followed by Sudan. The flavor of 4Q4 beverage was found as having a balance for all sensory attributes, but with the strongest red berry note ($p < 0.05$) of all varieties examined.

A total of 104 volatile compounds were detected in hibiscus calyces beverages; 88 of them were positively identified (Table 2). Positive identification was achieved by comparison of LRIs and mass spectra with those of authentic standard compounds analysed under identical experimental conditions, while tentative identification was established on matching LRIs and mass spectra of unknowns against those reported in commercial libraries. In general, the composition of beverages included aldehydes (23), acids (14), terpenes (13), ketones (12), furans (11), esters (7), alcohols (6), phenols (5), and miscellaneous compounds (13).

The most representative compounds in the beverage for each variety were 2-furfural and 5-methyl-2-furfural. Differences in concentrations for all varieties were found for both aldehydes: UAN 16-1 > Puebla Precoz > Sudan > 4Q4. Additionally these compounds have been reported in previous works (Chen et al. 1998; Pino et al. 2006). It has been noted that these aldehydes might originate from degradation of sugars (Ramírez et al. 2010). They have been described as recognized sweet and caramel-like odorants (Burdock 2010) and the calculated OAVs (Table 3) show that both aldehydes contribute to the sweet and caramel notes of hibiscus hot beverage. The comparative analysis of caramel potencies between the four beverages concur with the order in concentrations of both aldehydes.

Other sugar derived volatile compounds were common in all beverages, but in lesser amounts: 2-furanmethanol, 2-ethylfuran, 5-methyl-2(3*H*)-furanone, 2-acetylfuran, methyl 2-furoate, 2,4-dihydroxy-2,5-dimethyl-3(2*H*)-furanone, 2-pentylfuran, and 2-acetyl-5-methylfuran. Of those, 5-methyl-2(3*H*)-furanone and 2-pentylfuran, with their characteristic odour notes described as herbal and caramel-like (Burdock 2010) respectively, are congruent with the sensory data presented herein.

Fatty acid derived volatile compounds constituted the largest number of components (56 compounds). Among them, hexanal, (*E*)-2-hexenal, and (*Z*)-3-hexen-1-ol, with their characteristic odour described as herbal and green (Olías et al. 1993) were found as odour-active compounds. From this group, other important odorants are 1-octen-3-one (mushroom and green notes) and 1-octen-3-ol (sweet and herbaceous notes). Ramírez et al. (2010) reported 1-octen-3-one as the most intense aroma compound in hibiscus hot beverage. Nonanal and (*E*)-2-nonenal are associated with floral notes and has previously been reported to be present in dried hibiscus hot beverage

Table 1 Sensory descriptors of hot beverages of four Mexican hibiscus varieties

Sensory descriptor[a]	Sudan	Puebla precoz	4Q4	UAN 16-1
Acid	9.5 ± 0.6 a	8.0 ± 0.5 b	4.0 ± 0.2 d	6.0 ± 0.2 c
Astringent	9.0 ± 0.7 a	5.8 ± 0.4 b	2.5 ± 0.1 c	6.0 ± 0.4 b
Herbal	1.0 ± 0.5 c	1.5 ± 0.6 bc	2.0 ± 0.5 b	3.0 ± 0.2 a
Caramel	2.0 ± 0.4 c	4.0 ± 0.3 b	1.5 ± 0.5 c	10.0 ± 0.6 a
Floral	1.6 ± 0.7 b	2.0 ± 0.5 a	1.8 ± 0.4 ab	2.5 ± 0.5 a
Red berry	3.2 ± 0.3 b	2.0 ± 0.5 c	4.0 ± 0.4 a	2.8 ± 0.3 bc

[a]Values in the same row with different letters indicate significant difference at $p < 0.05$

Table 2 Volatile compounds from varieties of hibiscus calyces hot beverages

Compound	LRI_A^a	LRI_P^a	Identity[b]	Concentration (mg kg^{-1})[c]			
				Sudan	Puebla precoz	4Q4	UAN16-1
2,3-Butanedione	594	983	A	0.06a	0.08a	0.01b	0.01b
Butanal	597	852	A	tr[d]	tr	tr	nd
2-Butanone	600	907	A	tr	tr	tr	tr
Ethyl acetate	612	606	A	tr	nd	tr	nd
2-Methyl-3-buten-2-ol	620	1035	B	nd[d]	0.01	tr	tr
Acetic acid	645	1426	A	0.03b	0.02b	0.03b	0.05a
2-Butenal	650	1038	A	0.02a	0.01b	0.01b	nd
3-Methylbutanal	654	898	A	0.06a	0.01b	0.01b	0.02b
2-Methylbutanal	658	880	A	tr	tr	tr	tr
1-Penten-3-one	680	1149	A	0.02ab	0.03a	0.01b	0.04a
2,3-Pentanedione	700	1058	A	tr	tr	tr	tr
Pentanal	703	979	A	0.01b	0.01b	0.06a	0.01b
2-Ethylfuran	707	960	A	0.01b	0.01b	0.06a	0.03b
3-Hydroxybutan-2-one	718	983	B	0.01b	0.04a	0.01b	0.01b
Propanoic acid	721	1526	A	tr	tr	tr	tr
3-Methylbutan-1-ol	741	1215	A	nd	tr	0.03	nd
(*E*)-2-Pentenal	758	1130	B	0.03c	0.05b	0.06ab	0.07a
1-Hexen-3-one	775	1096	B	0.02a	0.02a	tr	nd
Hexan-3-one	784	1046	A	tr	tr	tr	tr
Hexanal	802	1064	A	0.04b	0.02c	0.07a	0.08a
4-Methyl-3-penten-2-one	804	1131	B	tr	tr	tr	tr
2-Furanmethanol	813	1666	A	tr	tr	tr	tr
2-Methyltetrahydrofuran-3-one	817	1270	B	tr	tr	tr	tr
Methylpyrazine	826	1250	A	tr	0.03	tr	tr
2-Furfural	836	1441	A	3.76c	4.06b	3.66c	6.23a
(*E*)-2-Hexenal	856	1219	A	0.12b	0.11b	0.14a	0.16a
(*Z*)-3-Hexen-1-ol	859	1387	A	0.03ab	0.01b	0.05a	0.07a
5-Methyl-2(3*H*)-furanone	880	1429	A	0.04b	0.02b	0.06a	0.08a
2-Methylbutanoic acid	885	1652	A	nd	nd	0.01	0.01
Heptan-2-one	892	1185	A	0.03a	0.02a	tr	nd
Heptanal	902	1189	A	0.03a	0.03a	0.02a	0.01a
3-(Methylthio)propanal	905	1469	A	tr	tr	tr	nd
(*E,E*)-2,4-Hexadienal	910	1392	A	0.01a	0.01a	nd	nd
2-Acetylfuran	912	1486	A	0.01a	0.01a	0.02a	0.01a
Pentanoic acid	920	1710	A	tr	tr	tr	tr
2,5-Dimethylpyrazine	923	1306	A	tr	nd	nd	nd
Pentanoic acid	927	1710	A	tr	0.03a	0.02a	0.02a
(*E*)-2-Heptenal	955	1334	A	0.02a	0.03a	0.01a	nd
Benzaldehyde	960	1502	A	tr	tr	nd	tr
5-Methyl-2-furfural	964	1543	A	0.63c	0.76b	0.61c	0.94a
1-Octen-3-one	978	1294	A	0.04b	0.03b	0.05a	0.07a
Methyl 2-furoate	981	1553	A	tr	tr	tr	tr
1-Octen-3-ol	984	1445	A	0.04b	0.03b	0.05ab	0.07a
2,4-Dihydroxy-2,5-dimethyl-3(2*H*)-furanone	989	–	C	tr	tr	tr	tr
2-Pentylfuran	991	1249	A	0.03c	0.05b	0.02c	0.08a
(*E*)-Dehydroxylinalool oxide	993	1209	B	tr	tr	tr	nd
Octanal	999	1287	A	0.09a	0.08a	0.05b	0.02c

Table 2 continued

Compound	LRI_A^a	LRI_P^a	Identity[b]	Concentration (mg kg^{-1})[c]			
				Sudan	Puebla precoz	4Q4	UAN16-1
(*Z*)-Dehydroxylinalool oxide	1007	1237	B	0.05a	0.03b	0.03b	nd
Hexanoic acid	1010	1840	A	0.02a	0.01a	0.01a	0.02a
p-Cymene	1025	1250	A	tr	tr	tr	0.06
Limonene	1029	1190	A	0.03a	0.01b	tr	0.04a
1,8-Cineole	1031	1205	A	0.03a	0.02a	0.01a	nd
(*Z*)-3-Hexenoic acid	1033	1945	A	0.01a	nd	nd	0.01a
2,2,6-Trimethylcyclohexan-1-one	1035	1336	B	0.02a	0.01a	0.03a	0.01a
2-Acetyl-5-methylfuran	1037	1650	A	0.02b	0.09a	0.01b	0.01b
Phenylacetaldehyde	1042	1646	A	0.01b	0.05a	0.03b	0.07a
(*Z*)-2-Octenal	1049	1439	A	0.01a	0.01a	tr	0.02a
(*Z*)-Linalool oxide (furanoid)	1073	1423	A	0.01c	0.05a	0.03b	0.07a
(*E*)-Linalool oxide (furanoid)	1087	1450	A	0.01c	0.04b	0.02c	0.06a
Terpinolene	1089	1287	A	0.01a	nd	0.01a	tr
p-Cymenene	1091	1435	A	tr	tr	tr	tr
Heptanoic acid	1095	1965	A	nd	0.01	nd	nd
Linalool	1097	1546	A	nd	tr	nd	nd
Nonanal	1101	1382	A	0.02b	0.04a	0.03ab	0.05a
(*E*)-6-Methyl-3,5-heptadien-2-one	1105	1587	B	0.01a	0.01a	0.01a	0.02a
2-Phenylethanol	1107	1897	A	0.01b	0.02ab	0.03a	0.04a
cis-Rose oxide	1109	1364	B	tr	tr	tr	tr
Terpinen-1-ol	1120	1628	A	tr	0.01	tr	tr
2-Ethylhexanoic acid	1128	1820	A	nd	0.01b	0.02b	0.04a
Methyl 2-methyloctanoate	1155	1380	A	0.1b	0.04ab	0.02	0.02b
trans-β-Terpineol	1158	1570	A	0.01	tr	tr	tr
Nerol oxide	1160	1468	B	0.01	tr	tr	tr
(*E*)-2-Nonenal	1163	1542	A	0.02b	0.03b	0.02b	0.05a
Nonan-1-ol	1169	1666	A	tr	0.01	tr	0.01
Methyl phenylacetate	1179	1758	A	tr	tr	nd	0.01
Methyl salicylate	1189	1720	A	0.02a	0.03a	0.03a	0.01a
α-Terpineol	1192	1680	A	0.01	tr	tr	tr
Octanoic acid	1197	2025	A	tr	tr	tr	0.01
Decanal	1202	1500	A	tr	tr	0.01	tr
Benzoic acid	1215	2423	A	tr	tr	nd	tr
Benzothiazole	1240	1962	A	0.03b	0.05a	tr	tr
(*E*)-2-Decenal	1264	1642	A	0.02a	0.01a	0.01a	0.02a
(*E*)-Cinnamaldehyde	1270	2025	A	0.01a	0.02a	nd	nd
Thymol	1290	2146	A	0.01a	0.02a	nd	0.02a
Undecanal	1305	1609	A	tr	tr	tr	tr
Carvacrol	1309	2225	A	0.02a	nd	0.02a	tr
2-Methoxy-4-vinylphenol	1314	2156	A	0.01b	0.03a	0.02ab	0.04a
(*E,E*)-2,4-Decadienal	1317	1796	A	0.01b	tr	0.01b	0.04a
Eugenol	1359	2151	A	0.05a	tr	0.04a	0.05a
Decanoic acid	1386	2272	A	0.02a	0.02a	0.03a	0.01a
Dodecanal	1409	1722	A	0.01b	0.04a	0.02b	0.05a
α-Ionone	1426	1863	A	0.03ab	0.02b	0.04a	0.01c
Geranylacetone	1455	1849	B	0.06b	0.06b	0.06b	0.08a
(*E*)-Isoeugenol	1458	2368	A	tr	nd	nd	nd

Table 2 continued

Compound	LRI_A[a]	LRI_P[a]	Identity[b]	Concentration (mg kg^{-1})[c]			
				Sudan	Puebla precoz	4Q4	UAN16-1
β-Ionone	1485	1932	A	0.04ab	0.03b	0.05a	0.01c
Benzophenone	1628	2470	A	0.01a	0.01a	0.02a	0.01a
Benzyl benzoate	1760	2638	A	0.02a	0.01a	0.03a	0.01a
Tetradecanoic acid	1779	2690	A	0.01a	0.01a	0.01a	tr
Hexahydrofarnesyl acetone	1846	2110	B	0.01a	0.03a	nd	nd
Benzyl salicylate	1866	2767	A	0.02a	0.01a	nd	0.02a
(*E,E*)-Farnesyl acetone	1922	2377	B	0.01b	0.01b	0.04a	0.01b
Methyl hexadecanoate	1925	2233	A	tr	tr	tr	tr
(*Z*)-Phytol	1947	2570	A	0.01a	tr	0.01a	tr
Hexadecanoic acid	1960	2899	A	tr	tr	0.01	nd

[a]LRI_A and LRI_P = Linear retention index on AT-5 ms and DB-Wax columns

[b]Identity: A, mass spectrum and LRIs agreed with standards; B, mass spectrum and LRIs agreed with literature data; C, mass spectrum agreed with mass spectral database

[c]Concentrations were expressed as mg methyl nonanoate equivalents kg^{-1} of beverage. Values in the same row with different letters indicate significant difference at $p < 0.05$

[d]tr = trace (< 0.01 mg kg^{-1}); nd = not detected

Table 3 Orthonasal odour thresholds and odour activity values (OAV) of volatile compounds in hibiscus hot beverages

Compound	Odour quality[a]	Odour threshold (μg kg^{-1})	OAV			
			Sudan	Puebla precoz	4Q4	UAN16-1
2-Furfural	Caramel	3000	1	1	1	2
5-Methyl-2-furfural	Caramel	6	105	127	102	157
1-Octen-3-one	Mushroom, green	4	10	8	12	18
1-Octen-3-ol	Sweet, herbaceous	20	2	2	3	4
Hexanal	Herbal	2.4[b]	17	8	29	33
(*E*)-2-Hexenal	Herbal	110[b]	1	1	1	1
(*Z*)-3-Hexen-1-ol	Herbal	3.9[b]	8	3	13	18
5-Methyl-2(3*H*)-furanone	Sweet, herbaceous	7	6	3	9	11
2-Pentylfuran	Caramel	6	5	8	3	13
Phenylacetaldehyde	Floral	4	2	12	8	18
Nonanal	Floral	2.8[b]	7	14	11	18
(*E*)-2-Nonenal	Floral	0.1	200	300	200	500
2-Methoxy-4-vinylphenol	Spicy	5.1[b]	2	6	4	8
Eugenol	Spicy	6	8	< 1	7	8
α-Ionone	Raspberry	0.6	50	33	67	17
Geranylacetone	Floral	60	1	1	1	1
β-Ionone	Raspberry	0.2	200	150	250	50

[a]According to Burdock 2010

[b]Czerny et al. (2008)

(Ramírez et al. 2010). Table 3 revealed that these constituents should be important in the overall aroma of the hibiscus hot beverage. The quantitative differences of these compounds among the beverages are in accordance with the sensory results in Table 1.

In contrast with the results of Ramírez et al. (2010), numerous terpenes were found as odour-active compounds (Table 3). However, these compounds did not contributed to the hibiscus hot beverage aroma. Linalool was found as a highest intensity aroma compound in fresh hibiscus

extracts, but was undetected in dried hibiscus extracts (Ramírez et al. 2010).

Phenylpropanoids and phenols represented another group of volatiles with relatively intense odor-active compounds (Table 3). Phenylacetaldehyde is related to floral notes and has previously been reported to be present in dried hibiscus hot beverage (Ramírez et al. 2010), whereas methyl salicylate has a spicy, sweet, and wintergreen-like odour and has been found in berries (Burdock 2010). In contrast, eugenol and 2-methoxy-4-vinylphenol, with spicy notes and OAVs > 1 were not reflected in the aroma profiles of hibiscus hot beverages. This terpene alcohol was only detected in trace amounts in hot beverage from var. Puebla Precoz.

Three carotenoid degradation products appear to be odour-active compounds in the hibiscus calyces hot beverages. Geranylacetone was described as a green and rosy floral odour and fresh-floral (Burdock 2010). This compound has been found previously in hibiscus extracts (Ramírez et al. 2010) and in calyces (Farag et al. 2015). The other two, α-ionone and β-ionone, have a peculiar raspberry note (Burdock 2010) and β-ionone is known to be an important contributor to the aroma of raspberries (Klesk et al. 2004). These two carotenoid degradation products have not been previously reported in hibiscus. The comparative analysis of red berry potencies between the four beverages are in accord with the order in concentrations of both isomers.

Conclusion

This study has revealed the potent odorants that are responsible for the overall flavour of hot beverages prepared from four Mexican hibiscus varieties. Results of the OAVs and sensory studies demonstrated that significant differences in odour profiles of the different hot beverages were mainly produced by the interaction of caramel (2-furfural and 5-methyl-2-furfural), herbal (hexanal, (*E*)-2-hexenal, (*Z*)-3-hexen-1-ol, 1-octen-3-one, 1-octen-3-ol, and 5-methyl-2(3*H*)-furanone), floral (phenylacetaldehyde, nonanal, (*E*)-2-nonenal, and geranylacetone), with red berry (α-ionone and β-ionone) notes contributing to the complexity of the flavour. However, the definitive role played by the odorants will require final measurement utilizing alternative reconstitution techniques and sensory evaluation.

Compliance with ethical standards

Conflict of interest The authors declare that there is no conflict of interest.

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