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Development of chemical-based reference standards for rooibos and honeybush aroma lexicons

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ABSTRACT

The honeybush sensory wheel was revised, using a large sample set (n = 585) comprising of the major commercial *Cyclopia* species, i.e. *C. intermedia*, *C. subternata* and *C. genistoides*. Only positive and negative aroma attributes were included in the wheel. Chemicals were identified to serve as reference standards for the honeybush (*Cyclopia* spp.) lexicon. Similarly, chemical-based reference standards were identified for the rooibos (*Aspalathus linearis*) lexicon. From a comprehensive literature search and gas chromatography–mass spectrometry analyses, chemicals were screened by an expert panel for their suitability in terms of typicality of the target aroma. Each chemical was evaluated in a 'base tea' and compared to a specific 'reference tea' exhibiting a high intensity of the target aroma. A total of 30 and 44 chemicals for rooibos and honeybush, respectively, were selected for validation by a trained panel. Descriptive sensory analysis was conducted to assign typicality and intensity scores for each chemical representing a target aroma attribute. Several chemicals were identified as suitable reference standards for the following aroma notes: isophorone ('rooibos-woody'), maltyl isobutyrate ('caramel'), *cis*-3-hexenol ('green grass'), 4-oxoisophorone ('seaweed') and 2,4,6-trichloroanisole ('musty/mouldy') for rooibos; and 2-acetyl-5-methylfuran ('woody'), levulinic acid ('fynbos-sweet'), maltyl isobutyrate ('caramel'), and 2-acetylpyrrole ('nutty') for honeybush.

1. Introduction

Sensory lexicons provide standardized, descriptive vocabularies that allow for accurate and objective communication of sensory properties of products among industry role-players (De Pelsmaeker, De Clercq, Gellynck, & Schouteten, 2019; Drake & Civille, 2003; Lawless & Civille, 2013) across all cultures and language barriers (Cherdchu, Chambers, & Suwonsichon, 2013; Monteiro et al., 2017). Generally, descriptive sensory analysis (DSA) is used for the development of a lexicon to describe and quantify the perceived sensory attributes associated with a specific product (Lawless & Heymann, 2010; Muñoz & Civille, 1998). A sensory lexicon consists of a list of sensory attributes with a definition for each attribute, as well as qualitative and/or quantitative reference standards. Qualitative reference standards facilitate concept alignment (Muñoz & Civille, 1998), whereas quantitative reference standards assist in defining intensity limits and panel calibration (Murray & Delahunty, 2000; Rainey, 1986). The use of quantitative references (intensity scale values) isincreasingly evident in the development of sensory lexicons (Chambers et al., 2016; Vázquez-Araújo, Chambers, & Carbonell-Barrachina, 2012).

Reference standards can be chemical- and/or food-based. A disadvantage of commercial food products as reference standards is that they may be reformulated or become unavailable over time. Chemical standards have the advantage of global availability, consistency, convenience and shelf-life stability (Drake & Civille, 2003). For the evaluation and selection of suitable chemical reference standards a neutral base (product matrix) is recommended to assist assessors to understand an attribute as it appears in the product in question (Chambers et al., 2016; Monteiro, Vilela, & Correia, 2014). Gas-chromatography (GC) analysis of the volatile fraction of the product of interest may aid selection of suitable chemical reference standards (Drake & Civille, 2003; Xia, Zhong, Chang, & Li, 2015). The use of chemical-based reference standards may allow for a clearer and grounded lexicon, however, interaction of the compound with the food matrix and different

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thresholds of the compounds could have an impact on the perceived aroma (Drake & Civille, 2003; Friedrich & Acree, 1998; Villamor & Ross, 2013; Zellner, Dugo, Dugo, & Mondello, 2008).

The role of lexicons in defining the specific quality characteristics of indigenous or traditional products that have Protected Designation of Origin (PDO) or Protected Geographical Indication (PGI) status, i.e. to distinguish them from inferior quality products, is evident in literature (Monteiro, Vilela, & Correia, 2014; Pereira, Dionísio, Matos, & Patarata, 2015; Stolzenbach, Byrne, & Bredie, 2011; Vázquez-Araújo, Chambers, & Carbonell-Barrachina, 2012). Rooibos tea, a global commodity, has recently received PGI status to offer protection to the term 'rooibos' (Biénabe & Marie-Vivien, 2017). The current South African regulatory guidelines for export of both rooibos (Anonymous, 2002) and honeybush (Anonymous, 2000) simply state that both herbal tea infusions should have a characteristic taste, aroma and color. Potential misinterpretation of the sensory quality of rooibos and honeybush by industry role-players emphasizes the need for validated product-specific sensory lexicons.

It is important that lexicons include both positive and negative sensory attributes as positive attributes are typical of the product, while negative attributes are usually a result of low quality, most likely caused by poor processing practices. Koch, Muller, Joubert, Van der Rijst, and Næs (2012) showed that poor grade rooibos associated with negative sensory attributes. While no grading system currently exists for honeybush, infusions prepared from 'under-fermented' plant material have, by comparison to optimally 'fermented' plant material, perceptible intensities of negative aroma attributes, in particular 'green grass' (Bergh, Muller, Van der Rijst, & Joubert, 2017; Erasmus, Theron, Muller, Van der Rijst, & Joubert, 2017). The 'smoky' taint is caused by over-heating of plant material during contact with a hot surface, thus a result of poor processing practices (Bergh et al., 2017).

Previous studies described the development of a sensory lexicon and sensory wheel for 'fermented' (oxidized) rooibos, based on 69 samples from plant material originating from 64 plantations, harvested during one production year (Koch et al., 2012). This sensory wheel captured flavor, taste and mouthfeel attributes of the product and was subsequently changed to an aroma wheel following sensory analysis of a larger sample set (n = 208) that included samples of three production years and the two major production areas (Jolley, Van der Rijst, Joubert, & Muller, 2017). The change to an aroma wheel was justified since the same results were obtained for aroma and flavor, except that attribute intensities were higher when aroma was scored. Similarly, the same need for a sensory wheel and lexicon was identified for 'fermented' honeybush tea (Theron et al., 2014), especially since many of the marketing companies handled both rooibos and honeybush as part of their product portfolio. An important difference between rooibos and honeybush is that rooibos is produced from only one species, *Aspalathus linearis*, whereas honeybush tea is produced from several *Cyclopia* species (Joubert, Gelderblom, Louw, & De Beer, 2008). The first generic honeybush sensory wheel was developed, based on the aroma/flavor, taste and mouthfeel attributes of six *Cyclopia* species. The latter species included the primary commercialized *Cyclopia* species, as well as some minor species, with the number of samples per species varying between 7 and 11. Further development focused on species-specific aroma wheels for *C. intermedia* (Bergh et al., 2017), *C. subternata*, *C. genistoides* and *C. maculata* (Robertson et al., 2018), yet a need still exists for revision of the generic honeybush sensory wheel, based on a comprehensive sample set of each of the primary species.

The lexicons for both these herbal teas were previously developed using verbal descriptions and in some instances food-based reference standards, however, there is a need for the identification of chemical-based reference standards to illustrate individual aroma attributes and to improve global understanding of the aroma descriptions. The aim of the present study was therefore to identify, screen and validate chemical reference standards for the revision of rooibos and honeybush lexicons. Given that a large set of honeybush samples was used to validate the attributes for the lexicon, the generic honeybush wheel was also updated.

2. Materials and methods

2.1. Aroma lexicons

Selection of rooibos chemical reference standards was based on 17 aroma attributes and their lexicon descriptions as described by Jolley et al. (2017). For honeybush the selection of chemical reference standards was based on 23 aroma attributes and their lexicon descriptions (Bergh et al., 2017; Erasmus et al., 2017). A new comprehensive data set was compiled for this herbal tea, obtained by sensory analysis of *C. genistoides* (n = 130), *C. subternata* (n = 260) and *C. intermedia* (n = 195) samples. These samples spanned several production years and included samples produced at the optimum 'fermentation' conditions (Bergh et al., 2017; Erasmus et al., 2017; Robertson et al., 2018), as well as commercial samples to represent the entire product category.

2.2. Aroma chemicals

Chemicals were supplied by Sigma-Aldrich (St Louis, MO, USA) and Kerry EMEA (Durban, South Africa). Kerry EMEA supplied chemicals diluted in propylene glycol or triacetin at 0.001, 0.01, 0.1, 1% or 10% (w/v). Other chemicals were also diluted with propylene glycol to 0.1% or 1% (w/v) solutions prior to use. Nano-encapsulated chemicals in plastic capsules, sourced from FlavorActiV^{TM} Ltd (Aston Rowant, UK), and rooibos steam condensate, supplied by Rooibos Ltd (Clanwilliam, South Africa), were added directly to the infusions. The rooibos steam condensate, containing volatile compounds, was collected during the pasteurization step of rooibos plant material (Koch, Muller, De Beer, Næs, & Joubert, 2013).

2.3. Herbal tea samples

Rooibos and honeybush 'base tea' samples were selected to serve as respective matrices when evaluating the aroma chemicals. The criterium for selection of each base tea was the typicality of their overall aroma profiles, yet without overt prominence of any positive aroma notes. The two base teas were thus considered to be 'neutral' in aroma profile, yet each providing the typical matrix of its respective herbal teas. A commercial, 'fermented' rooibos sample and a 1:1:1 (m/m/m) blend of commercial, 'fermented' *Cyclopia* spp. (*C. intermedia*, *C. genistoides* and *C. subternata*) were selected as base teas. Each respective base tea without the addition of chemicals also served as control ('calibration tea') during screening and validation of the aroma chemicals for the specific herbal tea (Fig. 1).

Specific rooibos and honeybush 'reference teas' were selected from our in-house sample library, previously identified to exhibit a high intensity of a specific target aroma (Bergh et al., 2017; Erasmus et al., 2017; Jolley et al., 2017; Robertson et al., 2018). The reference teas were used to familiarize panelists with the respective aroma attributes and to ascertain to what extent the perceived aroma of a chemical was typical of the target aroma attribute illustrated by die reference tea. Infusions of all rooibos and honeybush samples were freshly prepared before serving as described by Jolley et al. (2017) and Erasmus et al. (2017), respectively.

2.4. Identification and screening of potential chemical reference standards

Literature, chemical databases and aroma chemical supplier data were studied to identify chemicals that could potentially serve as reference standards for the aroma lexicon descriptors. Additionally, potential odorant compounds in volatile fractions of rooibos and honeybush infusions, identified by GC–MS analyses (Kerry EMEA) (Supplementary information in Appendix, Table A.1 & A.2), were

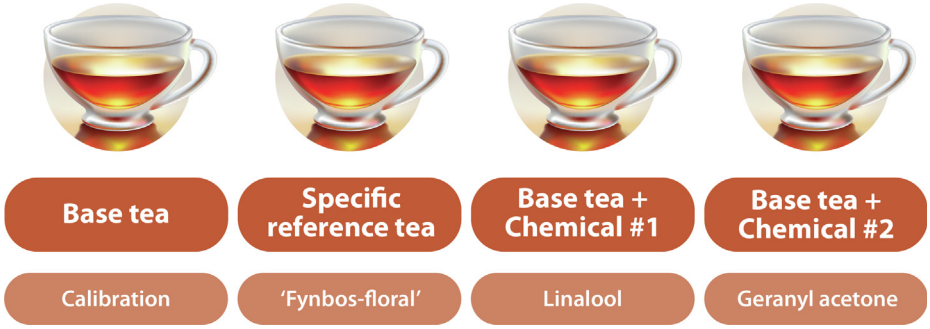

Fig. 1. Assessment of a target aroma descriptor, e.g. 'fynbos-floral' of the honeybush lexicon. 'Base tea' without the addition of a chemical served as calibration sample. The perceived aroma typicality and intensity of each chemical in the 'base tea' was compared to that of the specific 'reference tea' selected for a high intensity of the target aroma, e.g. 'fynbos-floral'.

included for evaluation.

Aroma chemicals were firstly screened by a panel of expert judges (n = 4) during 34 sessions (ca. 1.5 h per session) to eliminate atypical chemicals. This panel had extensive experience with DSA of both rooibos and honeybush. During each screening session different chemicals for two to three rooibos or honeybush lexicon descriptors were evaluated. Descriptors evaluated per session were selected based on their category (e.g. 'honey' and 'caramel' in the sweet-associated category) or whether they complement each other (e.g. 'apple' and 'sweet spice' or 'apricot' and 'fruity-sweet').

A freshly prepared base tea infusion (1000 mL) was spiked with a nano-encapsulated chemical capsule directly before each session. Similary, the diluted chemical was added in 20 μL increments until its aroma was perceived clearly. Subsequently, ca. 100 mL aliquots of the base tea, base tea dosed with chemical and reference tea, representing the target aroma (Fig. 1), were transferred to white porcelain mugs, covered with plastic lids to limit loss of volatiles, and placed in water baths controlled at 65 °C for the duration the evaluation period. The chemicals were assessed in terms of their typicality and intensity. Typicality of a chemical refers to the similarity of the perceived target aroma of the chemical in the dosed base tea compared to that of the aroma attribute of the specific reference tea. Based on the afore-mentioned, the chemical compound was eliminated or selected for validation. The chemical concentration (dose) was amended where applicable.

2.5. Validation of chemical reference standards using DSA

The validation of the chemical reference standards for rooibos and honebush was conducted consecutively over a three month period.

2.5.1. Panel training

Eight female judges, aged 25 to 65, and with extensive experience in DSA of fermented rooibos and honeybush served on the panel. A maximum of two chemicals per aroma descriptor were presented to the panel in 6 and 12 training sessions for rooibos and honeybush, respectively. For each aroma descriptor, the panel was presented with a base tea, base tea dosed with the chemical compounds and corresponding reference tea (Fig. 1). Tea infusions were prepared and presented as for screening.

At the start of each training session, the panel was informed of the target descriptor (e.g. 'fynbos-floral') to be assessed to focus panelists on the relevant descriptor. Panelists were instructed to remove the sample from the water bath, remove the plastic lid and swirl the infusion several times before analysing the aroma. Each panelist evaluated the different infusions individually, followed by a group discussion in which the group reached consensus on the suitability of each chemical as a potential chemical reference standard, based on typicality and intensity. Firstly, panelists assessed and described the base tea to calibrate their sensory perception. This was followed by the assessment of the chemical (e.g. (*R/S*)-linalool) by comparing the base tea dosed with the chemical with the corresponding reference tea (e.g. rooibos or honeybush sample exhibiting a high intensity of 'fynbos-floral'). Descriptions of the perceived target aroma in each sample were noted. The typicality (0 = atypical to 100 = extremely typical) and intensity (0 = not detectable to 100 = extremely high intensity) of the target aroma attribute was scored on unstructured line-scales once consensus was reached.

2.5.2. Analysis of samples

For DSA testing one target aroma was analyzed per session to limit panel fatigue and carry-over effects. Samples were tested in triplicate with a 15 min break between each sample set. Two chemicals were tested per target aroma attribute, apart from 'green grass', 'honey' and 'seaweed' for which only one chemical was tested. Blind testing of samples, labelled with 3-digit codes, was conducted with presentation order randomized per panelist. In addition, a clearly labelled mug with base tea (labelled as 'base') was included to serve as a fixed point to calibrate panelists at the start of each session. The specific reference tea (labelled as such) for the target aroma (e.g. 'fynbos-floral') was also included to sensitize panelists in terms of typicality and intensity. Scores for the perceived typicality and intensity of the target aroma were captured, using Compusense® five software (Compusense, Guelph, Canada). All analyses were conducted in individual tasting booths in a sensory laboratory under standard lighting and controlled temperature (21 °C) conditions.

2.6. Statistical procedures

The experimental design for DSA testing of each herbal tea was completely random with three replicates of each sample served to each panelist in random order. The DSA data were subjected to various statistical techniques to confirm panel reliability (Næs, Brockhoff, & Tomic, 2010) and data normality (Shapiro & Wilk, 1965). Data were subjected to analysis of variance (ANOVA), using the GLM (General Linear Model) procedure of SAS statistical software (Version 9.4, SAS Institute, Cary, NC, USA) according to the model for the study design. When effects were significant, Fisher's least significant difference was calculated to compare the means of typicality and intensity of an aroma chemical to that of the specific reference tea. $P < 0.05$ was considered significant.

2.7. Configuration of honeybush aroma wheel and occurrence frequency bar graphs

For development of the aroma wheel, the average intensity of each attribute (n = 585 samples) was calculated. Similar to a piechart, the percentage of the wheel that each attribute should occupy was obtained by expressing this average for an attribute as a percentage of the sum of the average intensities for all attributes. The occurrence of an attribute in the full sample set was counted when present at an average intensity ≥ 1 on a 100-point scale. This count value was used to calculate occurrence frequency as a percentage of the total number of samples. The data were presented in two bar graphs, displaying the positive and

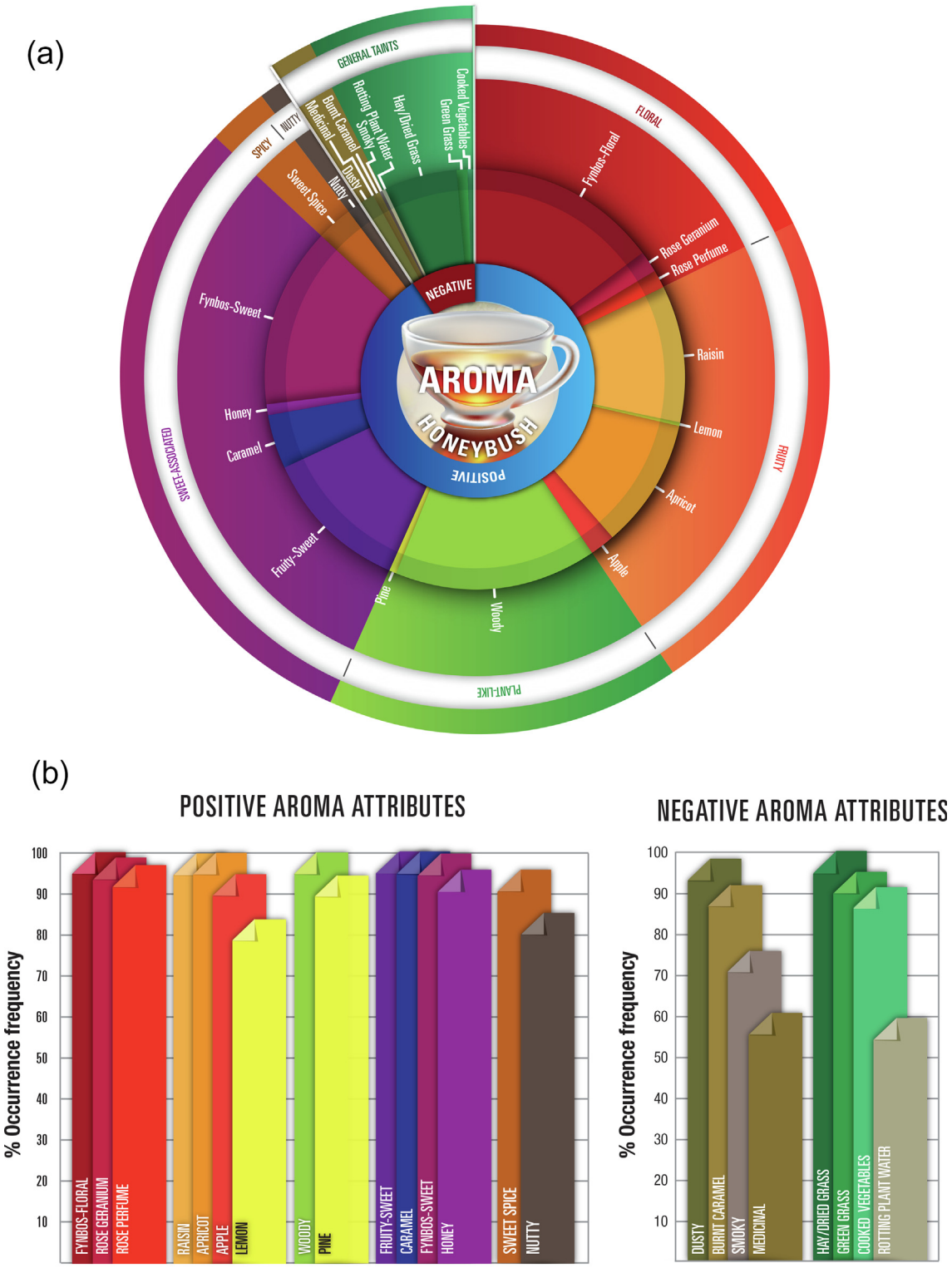

Fig. 2. The revised generic honeybush aroma wheel (a), compiled from sensory data for *C. genistoides*, *C. subternata* and *C. intermedia*, depicts relative intensities (width of a wedge) of 23 aroma attributes. The bar graphs (b) indicate the occurrence frequency (%) of positive and negative aroma attributes in the full sample set (n = 585).

negative aroma attributes, respectively.

3. Results and discussion

In the present study, the development of the chemical-based reference standards for rooibos and honeybush entailed identification, screening and validation of aroma compounds with the aid of expert and trained assessors.

3.1. Revised honeybush aroma wheel

The revised aroma wheel, compiled from *C. intermedia*, *C. subternata*

and *C. genistoides* samples, is depicted in Fig. 2. As for the rooibos aroma wheel (Jolley et al., 2017), the relative intensity of each of the aroma attributes is reflected by the width of a wedge. The major aroma notes are 'woody', 'fynbos-floral', 'fynbos-sweet', and to a lesser extent, 'fruity-sweet', agreeing with the relative intensities indicated for the species-specific aroma wheels of *C. intermedia* (Bergh et al., 2017), *C. genistoides*, *C. subternata* and *C. maculata* (Robertson et al., 2018). The positive aroma attributes, 'orange', 'plant-like' and 'coconut' included in the first generic honeybush sensory wheel (Theron et al., 2014) were removed, 'raisin' was added and 'walnut' and 'cassia/cinnamon' were changed to the more generic terms, 'nutty' and 'sweet spice', respectively. The negative aroma attributes remained the same except for the exclusion of 'yeasty' and addition of 'smoky'. The new honeybush aroma wheel is accompanied by two bar graphs, indicating the occurrence frequency (%) of the positive and negative aroma attributes in the full sample set, respectively. The bar graphs give another dimension to the relative importance of each attribute within the overall sensory profile of honeybush.

3.2. Identification of potential chemical reference standards

Major volatile compounds, identified by GC–MS analysis in freshly brewed rooibos (Supplementary material; Table A.1) were β-damascenone, geranyl acetone, β-ionone, 2,4-heptadienal and guaiacol. Other compounds included 6-methyl-5-hepten-2-one, 6-methyl-3,5-heptadien-2-one, 2-phenylethanol and eugenol. These compounds were previously identified (Habu, Flath, Mon, & Morton, 1985) in a rooibos steam distillate extract. A total of 28 chemical compounds identified from rooibos GC–MS data were sourced for screening.

Furthermore, volatile compounds (n = 33) that may contribute to the perceived aroma attributes of a freshly brewed blend of *Cyclopia* spp. were identified by GC–MS analysis (Supplementary material; Table A.2). Major compounds identified were 6-methyl-5-hepten-2-one, (*R/S*)-linalool, β-ionone, eugenol and several isomers of megastigmadienones and megastigmatrienones (not specified). Other compounds that were present in smaller quantities included hexanal, β-cyclocitral, β-damascenone and geranyl acetone. Le Roux, Cronje, Burger, and Joubert (2012) identified (*E*)-β-damascenone, (*R/S*)-linalool, (*E*)-β-damascone, geraniol, (*E*)-β-ionone, and (7*E*)-megastigma-5,7,9-trien-4-one by GC-olfactomery (GC-O) analysis as the major odor-active volatile compounds in fermented *C. subternata*. In addition, the GC-O assessors perceived the aromas of (6*E*,8*Z*)-megastigma-4,6,8-trien-3-one, (6*E*,8*E*)-megastigma-4,6,8-trien-3-one, (7*E*)-megastigma-5,7,9-trien-4-one, 10-*epi*-γ-eudesmol, *epi*-α-muurolol, and *epi*-α-cadinol as 'typically honeybush-like'. However, only commercially available megastigma-4,6,8-trien-3-one could be sourced for screening. Ntlhokwe, Muller, Joubert, Tredoux, and De Villiers (2018) identified 3-hydroxy-2-methylpyrone (maltol) and (*E*)-cinnamaldehyde in honeybush, volatiles associated with caramel and cinnamon aroma, respectively. For our investigation maltol and (*E*)-cinnamaldehyde could be sourced from a commercial supplier. A total of 25 chemical compounds identified from honeybush GC–MS data were subsequently sourced for screening.

From an extensive literature search and GC–MS data, a total of 90 and 80 potential chemical-based reference standards were identified and screened for rooibos (Table 1) and honeybush (Table 2), respectively. The objective was to test at least two chemicals per aroma attribute. Only chemical compounds assigned with food grade status and/or a FEMA (Flavor and Extract Manufacturers Association) number, i.e. compounds that are 'generally recognized as safe' (GRAS) for their intended use as flavor ingredients (Marnett et al., 2013) were sourced for testing. Only one chemical could be sourced for testing of the 'rooibos-woody' note. For this reason, the rooibos steam condensate, with a predominant 'rooibos-woody' aroma, was included in the final list of reference standards that were tested.

Table 1
Chemicals screened for the 17 rooibos aroma lexicon attributes in order of testing.

General attribute	Specific attribute	Chemical	FEMA[a] no
Floral	Fynbos-floral	geranyl acetone[b]	3542
		(*E*)-β-ionone[b]	2595
		(*R/S*)-linalool	2635
		nerol	2770
		2-nonanone	2785
		α-terpineol	3045
Woody	Rooibos-woody	camphene[b]	2229
		guaiacol[b]	2532
		isophorone[b]	3553
		3,5-octadien-2-one[b]	4008
		2,2,6-trimethylcyclohexanone[b]	3473
		rooibos steam condensate	n/a
Fruity	Apricot	β-cyclocitral[b]	3639
		β-damascenone[b]	3420
		benzophenone	2134
		diethyl succinate	2377
		(*R/S*)-γ-decalactone	2360
		(*R/S*)-γ-dodecalactone	2400
		ethyl phenylacetate	2452
		heptyl acetate	2547
		isoamyl isobutyrate	3507
		megastigma-4,6,8-trien-3-one [b]	4663
		3-methylbutanal	2692
		2-methylbutanal	2691
		neryl acetate	2773
		phenethyl phenylacetate	2866
		γ-undecalactone	3091
	Apple	(*E*)-2-heptenal[b]	3165
		hexanal[b]	2557
		(*E*)-2-hexenal[b]	2560
		ethyl isobutyrate	2428
		hexyl acetate	2565
	Citrus	decanal[b]	2362
		(*E*)-2-octenal[b]	3215
		6-methyl-5-hepten-2-one[b]	2707
		nonanal	2782
		octanal	2797
		(*E*)-2-undecenal	3423
Sweet-associated	Fruity-sweet	3-octen-2-one[b]	3416
		2-pentylfuran[b]	3317
		ethyl propionate	2456
		geranyl isovalerate	2518
		2-heptanone	2544
		propyl propionate	2958
	Honey	phenethyl acetate[b]	2857
		phenylacetaldehyde[b]	2874
		methyl phenylacetate	2733
	Caramel	5-ethyl-3-hydroxy-4-methyl-2(5*H*)-furanone	3153
		ethyl maltol	3487
		2-acetylfuran	3163
		maltol	2656
		maltyl isobutyrate	3462
		5-methylfurfural	2702
Spicy	Sweet spice	eugenol[b]	2467
		(*E,E*)-2,4-heptadienal[b]	3164
		6-methyl-3,5-heptadien-2-one[b]	3363
		4-vinylguaiacol[b]	2675
		(*E*)-cinnamaldehyde	2286
		dihydrocoumarin	2381
		ethyl isobutyrate	2428
		(*E*)-2-undecenal	3423
Vegetative taint	Hay/dried grass	camphene[b]	2229
		(*E*)-2-nonenal[b]	3213
		o-cresol	3480
		dihydrocoumarin	2381
		(*Z*)-4-heptenal	3289
		tonkalide	2556
		nonanal	2782
		3-methyl-2,4-nonanedione	4057

(*continued on next page*)

Table 1 (*continued*)

General attribute	Specific attribute	Chemical	FEMA[a] no
		octanal	2797
	Green grass	(*Z*)-3-hexenol	2563
	Rotting plant water	ethanethiol	4258
		dimethyl sulfide	2746
		dimethyl trisulfide	3275
		methional	2747
		methionol	3415
		methanethiol	2716
	Seaweed	acetaldehyde	2003
		(*E,E*)-2,4-decadienal	3135
		(*E,E*)-2,4-octadienal	3721
		4-oxoisophorone	3421
General taint	Burnt caramel	2-butanone	2170
		3-ethyl-2,5-dimethylpyrazine	3149
		ethyl maltol	3487
		furfural	2489
		furfuryl alcohol	2491
		4-hydroxy-2,5-dimethyl-3(2*H*)-furanone	3174
		levulinic acid	2627
		2-methoxythiophenol	4159
		3-methyl-1,2-cyclopentanedione	2700
	Medicinal/ rubber	*p*-cresol[b]	2337
		methyl salicylate[b]	2745
		p-ethylphenol	3156
	Dusty	L-borneol	2157
		(*R/S*)-geosmin	4682
		2-isopropyl-3-methoxypyrazine	3358
	Musty/ mouldy	2-ethylfuran[b]	3673
		2,4,6-trichloroanisole	*n/a*

[a] Flavor and Extract Manufacturers Association (FEMA) numbers sourced from database that supply information for the flavor industry (www.thegoodscentcompany.com).

[b] Chemicals identified in 'fermented' rooibos by GC–MS, analyses conducted by Kerry EMEA (Durban, South Africa).

3.3. Screening of chemical reference standards by expert panel

Selected chemicals were screened based on perceived typicality of the positive characteristic sensory attributes ascribed to freshly prepared rooibos and honeybush tea infusions, respectively. In addition, some chemicals were screened for typicality of negative aroma attributes (often associated with taints related to poor processing and storage methods) as perceived in the respective reference teas. Some compounds were screened for both rooibos and honeybush lexicon attributes, and for more than one aroma attribute within a lexicon (Tables 1 and 2).

3.3.1. Sensory profile of base tea

Use of a base tea for evaluation of potential chemical reference standards is essential to improve concept alignment among assessors, i.e. to understand each lexicon attribute as it is perceived in the product. The expert panel described the rooibos base tea as 'clean woody and low in floral and sweet-associated aromas with a subtle honey aroma note that was perceived as the tea cooled down'. The honeybush base tea was described by the expert panel as 'woody with subtle floral, fruity and sweet-associated aroma notes'.

3.3.2. Selection of chemical reference standards from literature (aroma description vs. perception)

The aroma character of each chemical was assessed in the base tea

Table 2
Chemicals screened for the 23 generic honeybush (*Cyclopia* spp.) aroma lexicon attributes in order of testing.

General attribute	Specific attribute	Chemical	FEMA[a] no
Floral	Fynbos-floral	geranyl acetone[b]	3542
		(*E*)-β-ionone[b]	2595
		(*R/S*)-linalool[b]	2635
		(*R/S*)-β-damascone[b]	3243
		(*Z*)-jasmone[b]	3196
		geranyl formate	2514
		2-nonanone	2785
	Rose geranium	nerol	2770
		geraniol	2507
	Rose perfume	phenethyl acetate	2857
		phenylacetaldehyde	2874
Plant-like	Woody	isophorone[b]	3553
		3,5-octadien-2-one[b]	4008
		2,2,6-trimethylcyclohexanone[b]	3473
		anhydrolinalool oxide[b]	3759
		guaiacol	2532
		2-acetyl-5-methylfuran	3609
	Pine	camphene[b]	2229
		ocimene quintoxide[b]	3665
		bornyl acetate	2159
		D-camphor	2230
Fruity	Apricot	δ-decalactone[b]	2361
		β-cyclocitral[b]	3639
		β-damascenone[b]	3420
		2-methylbutanal[b]	2691
		geranyl isovalerate	2518
		maltyl isobutyrate	3462
		isoamyl isobutyrate	3507
		heptyl acetate	2547
	Apple	(*E*)-2-hexenal[b]	2560
		ethyl isobutyrate	2428
		(*E*)-2-heptenal	3165
	Raisin	β-cyclocitral[b]	3639
		β-damascenone[b]	3420
		(*R/S*)-β-damascone[b]	3243
		megastigma-4,6,8-trien-3-one[b]	4663
	Lemon	(*E/Z*)-citral[b]	2303
		6-methyl-5-hepten-2-one[b]	2707
		(*R/S*)-ocimene[b]	3539
		myrcene[b]	2762
		α-terpinene	3558
		(*E*)-2-octenal	3215
Sweet-associated	Fynbos-sweet	(*R/S*)-δ-decalactone[b]	2361
		phenethyl alcohol	2858
		levulinic acid	2627
	Fruity-sweet	(*R/S*)-δ-decalactone[b]	2361
		isoamyl isobutyrate	3507
		geranyl isovalerate	2518
		2-nonanone	2785
		2-pentylfuran	3317
		diethyl succinate	2377
		maltyl isobutyrate	3462
		ethyl propionate	2456
		propyl propionate	2958
	Honey	methyl phenylacetate	2733
	Caramel	maltol	2656
		ethyl maltol	3487
		furfuryl alcohol	2491
		levulinic acid	2627
		maltyl isobutyrate	3462
Spicy	Sweet spice	eugenol[b]	2467
		(*E,E*)-2,4-heptadienal[b]	3164
		(*E*)-cinnamaldehyde	2286
		dihydrocoumarin	2381
Nutty	Nutty	2-acetyl-5-methylfuran	3609
		benzaldehyde[b]	2127
		5-methyl-2-hepten-4-one	3761
		2-acetylpyrrole	3202
Vegetative taint	Hay/dried grass	octanal	2797

(*continued on next page*)

Table 2 (*continued*)

General attribute	Specific attribute	Chemical	FEMA[a] no
		(*E*)-2-nonenal[b]	3213
		camphene[b]	2229
		o-cresol	3480
		(*Z*)-4-heptenal	3289
		3-methyl-2,4-nonanedione	4057
		nonanal	2782
	Green grass	(*Z*)-3-hexen-1-ol	2563
	Rotting plant water	2-methoxythiophenol	4159
		ethanethiol	4258
		dimethyl trisulfide	3275
	Cooked vegetables	methional	2747
		methionol	3415
		2-pentylfuran	3317
		benzyl acetate	2135
		S-methylthioacetate	3876
General taint	Burnt caramel	3-ethyl-2,5-dimethylpyrazine	3149
		4-hydroxy-2,5-dimethyl-3(2*H*)-furanone	3174
	Medicinal/ rubber	methyl salicylate	2745
		p-ethylphenol	3156
		p-cresol	2337
	Dusty	L-borneol	2157
		(*R/S*)-geosmin	4682
	Smoky	guaiacol	3532
		3-ethylpyridine	3394
		furfurylmethyl disulphide	3362

[a] Flavor and Extract Manufacturers Association (FEMA) numbers sourced from database that supply information for the flavor industry (www.thegoodscentcompany.com).

[b] Chemicals identified in 1:1:1:1 blend of 'fermented' *Cyclopia genistoides, C. subternata, C. maculata* and *C. longifolia* by GC–MS, analyses conducted by Kerry EMEA (Durban, South Africa).

at an intensity level which provided a perceptible aroma. Noble et al. (1987) suggested that for training purposes the intensity of a reference standard should be representative of the levels at which an aroma characteristic may be encountered. However, to illustrate a specific note, an intensity which provides an obvious aroma perception is recommended.

Lawless and Civille (2013) proposed that an initial literature search could disclose useful references for descriptors from different product categories. However, the aroma description of a chemical given by different literature sources is not consistent, and the aroma description from literature was often found to be misleading. In several instances, the literature description of the aroma associated with a chemical differed from that perceived by our expert panel in the respective base tea infusions. For example, ethyl isobutyrate is categorized in the 'sweet spices' class by Acree and Arn (2004), but described as 'sweet, ethereal, fruity and floral' by Arctander (1969) and 'sweet, ripe/over-ripe/fermented apple' by our expert panel when added to rooibos base tea. The aroma characteristics of chemicals may change as the product matrix and composition changes, as reviewed by Chambers and Koppel (2013) for hexanal and 3-methyl-1-butanol. Interactions of the aroma chemicals with the rooibos and honeybush matrices are unknown and may have influenced their perceived aroma and aroma intensity. Furthermore, the perceived aroma of a chemical may change at different concentration levels. Only limited literature sources and chemical databases (www.thegoodscentscompany.com) specify the aroma description of a chemical with details of concentration and solvent, for example at 1% in propylene glycol vs. 10% in propylene glycol. The aroma of (*E*)-2-hexenal identified for the 'apple' attribute is described by Arctander (1969) as 'powerful green-fruity, pungent vegetable-like' which becomes 'pleasant fruity and fresh-green' at dilutions < 0.1%. Similarly, Hongsoongnern and Chambers (2008) reported notable changes in the aroma character of certain chemicals at different concentrations during the development of a 'green' aroma lexicon. These findings illustrated that it is critical to specify the concentration and solvent when a chemical is used as a reference standard. Recently published lexicons indicate the preparation methods for specific reference standards (Chambers et al., 2016; López-López, Sánchez-Gómez, Montaño, Cortés-Delgado, & Garrido-Fernández, 2018; Monteiro et al., 2017; Pereira et al., 2015; Pujchakarn, Suwonsichon, & Suwonsichon, 2016; Xia et al., 2015).

3.3.3. Selecting reference standards from literature (character-impact compounds)

Volatile chemicals that contribute to the aroma of the principal sensory identity of a product are commonly referred to as character-impact compounds (Molnár, 2009). These compounds can be classified into four groups: 1) the characteristic aroma conclusively determined by a single character-impact compound; 2) the characteristic aroma due to a combination of a small number of compounds; 3) the characteristic aroma replicated, using a large number of compounds; and 4) no character-impact compounds have been identified and therefore the aroma cannot be reproduced adequately (Molnár, 2009). Groups 3 and 4 are relevant in the case of thermally processed foods (coffee and bread) and fermented foods (red wine, beer, cocoa and tea), and pose a challenge in selecting an appropriate chemical as reference standard. Processing of honeybush tea entails 'fermentation', which is a high temperature oxidation process (Bergh et al., 2017), while rooibos 'fermentation', also an oxidation process (Joubert & De Beer, 2011), takes place at temperatures where microorganisms can proliferate (Gouws, Hartel, & Van Wyk, 2014) and potentially add to the development of its aroma profile. Rooibos 'fermented' in the laboratory mostly lack the full aroma profile of rooibos produced by industry according to the typical open-air process (unpublished data).

A few single character-impact compounds identified from literature or from GC–MS data were screened for lexicon attributes. (*E*)-Cinnamaldehyde, the character-impact compound for cinnamon (Fischett, 2010) and perceived as 'apple-cinnamon' by the expert panel, was selected for the 'sweet spice' attribute of the rooibos lexicon. Contrastingly, γ-decalactone and δ-decalactone identified as odor impact compounds for apricot in sweet Fiano wine (Genovese, Gambuti, Piombino, & Moio, 2007), were perceived as 'artificial peach-like' in the rooibos base tea and 'peach, butter, coconut' in honeybush base tea, respectively.

The selection of chemicals for the fruity aroma attributes, 'apricot' and 'cooked apple' represented challenges as a character-impact aroma is often elicited by a synergistic blend of several aroma chemicals. The aroma of *n*-hexanal (component of natural apple flavor) is reminiscent of 'green, painty, rancid oil', however, in combination with character-impact compounds, ethyl 2-methyl butyrate and 2-hexenal, the characteristic aroma note of 'apple' is perceived (Flath, Black, Guadagni, McFadden, & Schultz, 1967). Similarly, in the present study *n*-hexanal identified in rooibos was perceived as 'vegetable, green grass, woody'. Both (*E*)-2-heptenal (perceived as 'apple, spice, almond') and (*E*)-2-hexenal (perceived as 'fresh, green apple') identified in rooibos were selected for further validation as chemicals for the 'apple' attribute in the rooibos lexicon. Contrastingly, β-damascenone, also considered as a character-impact compound for apple (Cunningham, Acree, Barnard, Butts, & Braell, 1986) and present in both rooibos and honeybush, was perceived as 'woody, sweet', or 'peach', and rejected for further validation as reference standard for 'apple'.

The floral-like compound, (*E*)-β-ionone, was a major compound in rooibos, but was perceived as 'musk-like' by the expert panel. For the 'citrus' attribute of rooibos, 6-methyl-5-hepten-2-one (perceived as 'citrus') and (*E*)-2-undecenal (perceived as 'sweet orange/orange peel') were selected. GC–MS results indicated the presence of 6-methyl-5-hepten-2-one at high quantities even though the intensity and

Table 3
Descriptive sensory analysis (DSA) results for potential chemical reference standards evaluated for 'fermented' rooibos aroma attributes (chemicals that did not differ significantly from the target aroma in the specific reference tea[a] for typicality are highlighted in **bold**).

General attributes	Specific attributes	Sample	Typicality	Intensity
Floral	Fynbos-floral	base tea (control)	87.38a ± 7.42	46.71c ± 6.04
		floral reference tea	88.56a ± 2.73	51.65c ± 1.39
		(*R/S*)-linalool	53.69b ± 7.29	74.73a ± 1.95
		geranyl acetone[b]	63.29b ± 5.41	60.81b ± 6.13
Woody	Rooibos-woody	base tea (control)	90.98a ± 0.89	58.30b ± 1.00
		rooibos-woody reference tea	**93.31a ± 3.50**	62.75a ± 2.89
		rooibos steam condensate	**94.81a ± 1.48**	58.72b ± 0.42
		isophorone[b]	**92.75a ± 2.01**	61.21ab ± 1.64
Fruity	Apricot	base tea (control)	75.21ab ± 4.91	47.57b ± 5.09
		apricot reference tea	92.60a ± 4.02	65.71a ± 3.87
		β-cyclocitral[b]	49.22c ± 15.25	36.83b ± 7.99
		2-methylbutanal	65.33bc ± 8.60	42.36b ± 8.88
	Apple	base tea (control)	26.85d ± 5.24	11.70d ± 0.25
		apple reference tea	84.48a ± 4.94	40.63c ± 2.06
		(*E*)-2-heptenal[b]	49.19c ± 2.25	53.79b ± 0.54
		(*E*)-2-hexen-1-al[b]	63.63b ± 0.80	61.68a ± 2.78
	Citrus	base tea (control)	67.27b ± 9.41	14.65d ± 3.04
		citrus reference tea	93.90a ± 5.52	22.01c ± 4.47
		(*E*)-2-undecenal	27.71d ± 3.59	74.23a ± 1.67
		6-methyl-5-hepten-2-one[b]	49.13c ± 5.96	62.83b ± 4.67
Sweet-associated	Fruity-sweet	base tea (control)	55.35b ± 3.94	34.67c ± 1.48
		fruity-sweet reference tea	93.10a ± 0.92	77.94a ± 1.65
		geranyl isovalerate	43.83b ± 5.64	43.98b ± 6.24
		propyl propionate	53.35b ± 12.29	42.33bc ± 5.13
	Honey	base tea (control)	96.31a ± 3.93	27.94c ± 4.07
		honey reference tea	95.94a ± 1.13	51.33b ± 2.15
		methyl phenylacetate	48.93b ± 1.83	67.12a ± 0.91
	Caramel	base tea (control)	88.98a ± 2.59	26.13b ± 2.91
		caramel reference tea	**84.25a ± 3.04**	71.49a ± 3.59
		maltyl isobutyrate	**88.59a ± 1.13**	20.92b ± 3.22
		ethyl maltol	68.53b ± 4.16	73.36a ± 6.77
Spicy	Sweet spice	base tea (control)	71.96b ± 11.34	30.35b ± 4.54
		sweet spice reference tea	89.29a ± 0.51	46.89a ± 5.56
		(*E*)-cinnamaldehyde	45.25c ± 7.19	51.62a ± 3.56
		(*E,E*)-2,4-heptadienal[b]	72.61b ± 11.53	42.99a ± 8.25
Vegetative taint	Hay/dried grass	base tea (control)	90.95a ± 1.34	48.15d ± 1.59
		hay/dried grass reference tea	95.04a ± 5.80	68.80a ± 0.11
		o-cresol	65.77b ± 3.26	55.75c ± 0.74
		nonanal	63.49b ± 1.13	58.06b ± 1.31
	Green grass	base tea (control)	27.87b ± 7.35	21.60b ± 8.51
		green grass reference tea	**89.89a ± 8.98**	64.26a ± 2.43
		(*Z*)-3-hexen-1-ol	**80.38a ± 4.11**	66.71a ± 5.84
	Rotting plant water	base tea (control)	4.21d ± 2.49	5.23c ± 1.70
		rotting plant water reference tea	78.67a ± 2.13	37.25b ± 1.93
		methionol	49.15b ± 6.71	33.25b ± 4.06
		ethanethiol	21.89c ± 3.90	79.23a ± 1.51
	Seaweed	base tea (control)	66.04a ± 13.01	13.08a ± 0.94
		seaweed reference tea	**78.16a ± 5.65**	14.43a ± 1.55
		4-oxoisophorone	**62.68a ± 7.28**	10.61a ± 3.28
General taint	Burnt caramel	base tea (control)	21.91c ± 3.33	11.33c ± 3.26
		burnt caramel reference tea	78.84a ± 4.58	66.44b ± 1.91
		4-hydroxy-2,5-dimethyl-3(*H*)-furanone	54.13b ± 4.01	65.88b ± 2.75
		3-ethyl-2,5-dimethylpyrazine	31.75c ± 8.22	73.86a ± 2.07
	Medicinal/rubber	base tea (control)	14.24c ± 5.57	10.35d ± 3.04
		medicinal/rubber reference tea	93.66a ± 0.93	48.06c ± 0.33
		p-ethylphenol	75.55b ± 3.44	76.00a ± 2.83
		p-cresol[b]	70.00b ± 2.93	62.27b ± 1.29
	Dusty	base tea (control)	29.48c ± 2.81	23.06c ± 2.37
		dusty reference tea	82.46a ± 0.74	53.92b ± 3.22
		L-borneol	56.19b ± 0.72	83.04a ± 2.53
		(*R/S*)-geosmin	56.00b ± 2.85	53.88b ± 3.32
	Musty/mouldy	base tea (control)	38.98b ± 18.94	14.46c ± 2.64
		musty/mouldy reference tea	**83.82a ± 4.43**	45.45b ± 5.08
		2,4,6-trichloroanisole	**79.60a ± 2.80**	59.00a ± 5.26
		2-ethylfuran[b]	49.14b ± 23.51	14.48c ± 3.23

[a] Rooibos tea previously identified to exhibit a high intensity of specific target aroma attribute.
[b] Chemicals identified in fermented rooibos by GC–MS.

occurrence frequency of the 'citrus' attribute are normally extremely low (Jolley et al., 2017).

Screening of chemicals for the negative rooibos and honeybush aroma attributes is particularly important since their presence is detrimental to quality and ultimately consumer acceptance of the herbal tea. For the 'seaweed' attribute, present in rooibos infusions, only 4-oxoisophorone was selected for further validation. Interestingly, 4-oxoisophorone is formed on oxidation of isophorone (identified in

Table 4
Descriptive sensory analysis (DSA) results for potential chemical reference standards evaluated for 'fermented' honeybush aroma attributes (chemicals that did not differ significantly from the target aroma in the specific reference tea[a] for typicality are highlighted in **bold**).

General attributes	Specific attributes	Sample	Typicality	Intensity
Floral	Fynbos-floral	base tea (control)	92.67a ± 1.15	64.88ab ± 0.92
		fynbos-floral reference tea	89.21a ± 2.72	64.11b ± 3.56
		(*R/S*)-linalool[b]	67.24c ± 0.35	68.10a ± 1.59
		geranyl acetone[b]	73.67b ± 2.92	52.90c ± 0.38
	Rose geranium	base tea (control)	94.45ab ± 1.71	48.16b ± 2.21
		rose geranium reference tea	98.06a ± 1.76	51.25ab ± 1.85
		nerol	90.76b ± 0.35	50.13b ± 0.33
		geraniol	90.89b ± 6.59	54.23a ± 1.61
	Rose perfume	base tea (control)	94.35a ± 0.69	29.76d ± 2.46
		rose perfume reference tea	95.88a ± 1.48	41.76c ± 1.68
		phenethyl acetate	48.44c ± 2.48	75.38b ± 1.87
		phenylacetaldehyde	61.44b ± 1.87	82.49a ± 0.55
Plant-like	Woody	base tea (control)	76.47b ± 11.14	51.10c ± 1.13
		woody reference tea	**96.31a ± 0.16**	68.31b ± 0.63
		guaiacol	47.92c ± 3.63	75.18a ± 2.69
		2-acetyl-5-methylfuran	**85.44ab ± 9.83**	52.29c ± 1.63
	Pine	base tea (control)	93.11a ± 0.72	20.18d ± 2.71
		pine reference tea	93.85a ± 1.59	66.94b ± 0.77
		bornyl acetate	67.71b ± 1.36	75.83a ± 0.91
		camphene[b]	51.36c ± 4.49	50.13c ± 1.90
Fruity	Apricot	base tea (control)	83.25bc ± 1.07	50.75c ± 1.73
		apricot/apricot jam reference tea	95.11a ± 0.25	77.16a ± 2.62
		maltyl isobutyrate	75.84c ± 0.53	55.02b ± 2.79
		2-methylbutanal[b]	87.17b ± 7.78	43.94d ± 1.47
	Apple	base tea (control)	97.60a ± 2.08	23.66c ± 2.58
		cooked apple reference tea	93.92b ± 1.84	48.83b ± 3.43
		(*E*)-2-hexen-1-al[b]	72.31c ± 0.45	78.22a ± 0.49
		ethyl isobutyrate	52.59d ± 0.82	77.85a ± 2.68
	Raisin	base tea (control)	96.18a ± 2.57	27.97c ± 1.88
		raisin reference tea	96.87a ± 1.62	45.47a ± 1.21
		β-cyclocitral[b]	89.30b ± 2.15	32.23b ± 2.81
		β-damascenone[b]	59.43c ± 1.17	28.60c ± 0.69
	Lemon/lemon grass	base tea (control)	19.11c ± 3.50	10.49c ± 2.44
		lemon/lemon grass reference tea	88.39a ± 5.19	54.10b ± 0.99
		6-methyl-5-hepten-2-one[b]	63.69b ± 5.31	68.81a ± 1.66
		(*R/S*)-ocimene[b]	62.05b ± 5.08	65.70a ± 5.92
Sweet-associated	Fynbos-sweet	base tea (control)	95.45a ± 0.86	48.71b ± 2.78
		fynbos-sweet reference tea	**91.99b ± 2.60**	52.06b ± 1.84
		phenethyl alcohol	64.03c ± 0.87	62.27a ± 1.38
		levulinic acid	**90.29b ± 2.21**	49.44b ± 2.93
	Fruity-sweet	base tea (control)	90.20a ± 3.85	37.89d ± 3.19
		fruity-sweet reference tea	95.85a ± 3.59	75.91a ± 2.55
		geranyl isovalerate	71.41c ± 1.36	49.23b ± 0.19
		propyl propionate	81.57b ± 2.56	44.35c ± 0.78
	Honey	base tea (control)	96.01a ± 3.45	20.56c ± 1.13
		honey reference tea	97.71a ± 2.06	37.25b ± 0.47
		methyl phenylacetate	65.76b ± 2.19	86.37a ± 1.01
	Caramel	base tea (control)	94.27a ± 0.70	35.27c ± 5.26
		caramel reference tea	**91.28a ± 2.63**	43.22b ± 1.53
		ethyl maltol	78.15b ± 1.95	73.75a ± 5.58
		maltyl isobutyrate	**94.19a ± 1.52**	42.01bc ± 2.77
Spicy	Sweet spice	base tea (control)	69.68b ± 10.75	17.93d ± 1.81
		sweet spice reference tea	95.20a ± 3.03	83.65a ± 2.89
		dihydrocoumarin	67.75b ± 2.03	69.26b ± 1.84
		(*E,E*)-2,4-heptadienal[b]	67.91b ± 2.48	54.72c ± 6.41
Nutty	Nutty	base tea (control)	91.50a ± 5.79	22.68c ± 2.07
		nutty reference tea	**99.03a ± 0.91**	35.74a ± 3.09
		benzaldehyde[b]	72.22b ± 2.12	28.79b ± 1.63
		2-acetylpyrrole	**90.93a ± 6.37**	23.86c ± 1.68
Vegetative taint	Hay/dried grass	base tea (control)	94.75a ± 0.92	21.20c ± 2.45
		hay/dried grass reference tea	94.61a ± 0.97	42.33b ± 0.99
		o-cresol	70.40b ± 2.54	44.04b ± 2.17
		nonanal	58.82c ± 2.76	49.31a ± 1.69
	Green grass	base tea (control)	2.08c ± 3.61	0.00c ± 0.00
		green grass reference tea	95.77a ± 3.66	51.56b ± 0.22
		(*Z*)-3-hexen-1-ol	79.71b ± 0.13	78.13a ± 0.22
	Rotting plant water	base tea (control)	0.00c ± 0.00	0.00c ± 0.00
		rotting plant water reference tea	96.88a ± 2.71	53.02b ± 0.75
		dimethyl trisulfide	44.26b ± 1.81	93.65a ± 0.09
		ethanethiol	46.23b ± 3.48	95.44a ± 3.27
	Cooked vegetables	base tea (control)	0.00d ± 0.00	0.00d ± 0.00
		cooked vegetables reference tea	98.19a ± 1.58	79.65a ± 0.67
		methional	52.38c ± 2.54	74.00b ± 1.15

(*continued on next page*)

Table 4 (*continued*)

General attributes	Specific attributes	Sample	Typicality	Intensity
		methionol	60.94b ± 0.98	43.09c ± 1.60
General taint	Burnt caramel	base tea (control)	13.32d ± 2.59	10.76d ± 1.23
		burnt caramel reference tea	94.99a ± 0.21	38.41c ± 1.07
		4-hydroxy-2,5-dimethyl-3(*H*)-furanone	53.48b ± 3.20	56.89b ± 2.58
		3-ethyl-2,5-dimethylpyrazine	29.16c ± 6.76	76.73a ± 3.63
	Medicinal/rubber	base tea (control)	6.46d ± 0.83	6.27c ± 0.04
		medicina/rubber reference tea	96.71a ± 0.53	69.80b ± 0.12
		p-ethylphenol	69.81c ± 1.08	82.19a ± 4.02
		p-cresol	74.75b ± 0.33	72.13b ± 4.06
	Dusty	base tea (control)	95.42a ± 3.97	16.89d ± 1.33
		dusty reference tea	94.88a ± 4.45	35.24c ± 1.28
		L-borneol	57.81b ± 1.69	84.88a ± 0.84
		(*R/S*)-geosmin	59.72b ± 1.19	58.96b ± 1.44
	Smoky	base tea (control)	8.32d ± 3.50	0.92d ± 1.04
		smoky reference tea	99.96a ± 0.03	59.90a ± 0.85
		guaiacol	40.22c ± 5.67	29.95b ± 0.62
		3-ethylpyridine	71.23b ± 16.79	11.96c ± 2.19

[a] Honeybush tea previously identified to exhibit a high intensity of the specific target aroma attribute.
[b] Chemicals identified in fermented honeybush by GC–MS.

rooibos) and imparts an aroma reminiscent of 'tobacco, hay, tea and seaweed (nori)' (N. Wiltshire, Kerry EMEA, Durban, South Africa, 2015, personal communication). However, further analysis is required to verify whether 4-oxoisophorone is responsible for the 'seaweed' aroma present in a tainted rooibos product batch.

The 'hay/dried grass' attribute is generally associated with under-fermented rooibos (Joubert, 1998) and is classified as a taint when present at high intensities (Koch et al., 2012). Nonanal is used in certain commercial aroma kits as a standard for 'hay' character. Its aroma has been described as 'hay, like dried grass or cucumber skin' according to the AROXA™ flavor ingredients range (www.aroxa.com) and as 'dry hay or straw' according to FlavorActiV™ (www.flavoractiv.com). In the present study, both nonanal and *o*-cresol were perceived as 'dry grass' during screening and therefore selected for further validation. Similarly, 'green grass' is associated with low grade rooibos (Koch et al., 2012) and (*Z*)-3-hexenol was selected for further validation as standard for both herbal teas.

Several chemicals associated with a 'rotten' or 'sulfurous/vegetable' character were screened for the 'rotting plant water' attribute. Thiols, in particular, impart off-flavors to beer (Vermeulen, Gijs, & Collin, 2005; Walker, 1995) and wine (Swiegers, Bartowsky, Henschke, & Pretorius, 2005). Ethanethiol was selected for both rooibos and honeybush for further validation as it is regarded as the chemical responsible for the 'putrefaction' taint typically found in beer (Baxter & Hughes, 2001).

Chemicals associated with 'burnt' may impart an undesired 'tobacco' or 'smoky' aroma. These aroma notes are not typical of the 'burnt caramel' attribute associated with rooibos and honeybush. The character-impact compound for inter alia burnt sugar aroma, 4-hydroxy-2,5-dimethyl-3(2*H*)-furanone, imparts a sweet caramel, burnt-sugar flavor with noticeable fruitiness to beer, Arabica coffee and white bread crust (Acree & Arn, 2004; Hodge, Mills, & Fisher, 1972). This compound was therefore selected for further validation of the 'burnt caramel' attribute of rooibos and honeybush. Methyl salicylate, the character-impact compound for wintergreen (Fischett, 2010), and also identified in rooibos, was screened for the negative attribute, 'medicinal/rubber' ('Band-aid®'). However, this chemical was rejected for its prominent 'bubblegum' aroma and low intensity of the 'Band-aid®' character. Furthermore, the off-odor impact volatile for 'musty' found in corked wine, 2,4,6-trichloroanisole (Parker, Elmore, & Methven, 2015), was selected for the 'musty/mouldy' taint of the rooibos lexicon. (*R/S*)-Geosmin is an off-odor-impact compound for 'earthy-musty', imparting an undesirable 'earthy' taint to drinking water (Parker, Elmore, & Methven, 2015). (*R/S*)-Geosmin is available as reference standard in commercial aroma kits (www.aroxa.com; www.flavoractiv.com) and was selected for the 'dusty' attribute of rooibos and honeybush lexicons. Interestingly, a total of 9 compounds identified from rooibos and 11 from honeybush GC–MS data, were selected as potential chemical reference standards for further validation (Tables 1 and 2).

3.4. Validation of chemical reference standards for rooibos and honeybush lexicons

DSA was conducted to determine how representative the aroma chemicals were of the target aromas in the respective reference teas. Given the complexity of rooibos and honeybush aroma profiles, we focused on typicality as it forms the basis of selecting a suitable chemical standard. It was important that the intensity of the respective aroma notes was not particularly high in the base tea, as it served as a neutral matrix. When a chemical was added to the base tea, the objective was to achieve a high typicality score at an intensity similar to the target aroma note for which the reference tea was selected.

The mean intensity and typicality scores for the rooibos and honeybush attributes are presented in Tables 3 and 4**,** respectively. The majority of the typicality scores for the perceived aroma of the chemicals differed significantly ($P < 0.05$) from that of the target aroma in the respective reference teas. One could argue that the aroma of one chemical compound does not necessarily elicit a similar aroma perception to that of the reference tea. In contrast, the typicality scores of aroma chemicals evaluated for several rooibos lexicon attributes, i.e. isophorone ('rooibos-woody'), maltyl isobutyrate ('caramel'), (*Z*)-3-hexenol ('green grass'), 4-oxoisophorone ('seaweed') and 2,4,6-trichloroanisole ('musty/mouldy'), and the honeybush lexicon attributes, i.e. 2-acetyl-5-methylfuran ('woody'), levulinic acid ('fynbos-sweet'), maltyl isobutyrate ('caramel'), and 2-acetylpyrrole ('nutty') did not differ significantly ($P \geq 0.05$) from that of the respective reference teas. Subsequently, these aroma chemicals could therefore be regarded as a better match and thus suitable reference standards for the respective lexicons. Many chemicals, e.g. (*E*,*E*)-2,4-heptadienal for the 'sweet spice' of the rooibos lexicon (Table 3), and β-cyclocitral for 'raisin' of the honeybush lexicon (Table 4), had high typicality scores, yet were significantly different from the reference tea ($P < 0.05$). These chemicals merit further investigation in terms of the effect of dosage on typicality.

Furthermore, the most suitable chemical-based reference standard (s) per attribute can also be derived from the results. For instance, for rooibos, (*E*)-2-hexenal had a significantly ($P < 0.05$) higher typicality score than (*E*)-2-heptenal and the former compound could be regarded as a more suitable reference standard for 'apple'. Both nonanal and *o*-cresol may be used for 'hay/dried grass' as their typicality scores did not differ significantly ($P \geq 0.05$).

Table 5
Updated 'fermented' rooibos aroma lexicon with chemical-based reference standards.

General attributes	Specific attributes	Description of aroma attributes	Reference standards[a]	Information sources[b]
Floral aroma	Fynbos-floral	The unique, somewhat sweet aromatics associated with fynbos[c] vegetation.	1) (*R/S*)-linalool (1% in propylene glycol); 120 μL/L tea	A; B; D; E
			2) geranyl acetone (1% in propylene glycol); 240 μL/L tea	B; C; D; E
Woody aroma	Rooibos-woody	Aromatics associated with dry bushes, stems and twigs of rooibos vegetation.	1) rooibos steam condensate (undiluted); 200 μL/L tea	F
			2) isophorone (0.1% in propylene glycol); 600 μL/L tea	B; C; E
Fruity aromas	Apricot	Aromatics associated with apricot jam or dried apricot.	1) β-cyclocitral (0.1% in propylene glycol); 480 μL/L tea	C; D; E
			2) 2-methylbutanal (0.1% in triacetin); 240 μL/L tea	B; D; E
	Apple	Sweet aromatics associated with cooked apples or apple pie.	1) (*E*)-2-heptenal (1% in propylene glycol); 280 μL/L tea	B; C; D
			2) (*E*)-2-hexen-1-al (1% in propylene glycol); 200 μL/L tea	A; B; C; D; E
	Citrus	Sweet aromatics associated with ripe oranges.	1) (*E*)-2-undecenal (1% in propylene glycol); 40 μL/L tea	B; D; E
			2) 6-methyl-5-hepten-2-one (1% in propylene glycol); 320 μL/L tea	C; E
Sweet-associated aromas	Fruity-sweet	Aromatics associated with the sweet/sour smell of non-specific fruit.	1) geranyl -isovalerate (1% in propylene glycol); 240 μL/L tea	A; B; D; E
			2) propyl propionate (1% in propylene glycol); 320 μL/L tea	A; B; C; D; E
	Honey	Aromatics associated with the sweet fragrance of fynbos[c] honey or *Alyssum* flowers.	1) methyl phenylacetate (1% in propylene glycol); 200 μL/L tea	B; D; E
	Caramel	Sweet aromatics characteristic of caramelized sugar.	1) ethyl maltol (1% in propylene glycol); 240 μL/L tea	D; E
			2) maltyl isobutyrate (1% in propylene glycol); 500 μL/L tea	D; E
Spicy aroma	Sweet spice	Aromatics associated with sweet spice, mainly cinnamon.	1) (*E*)-cinnamaldehyde (1% in propylene glycol); 120 μL/L tea	A; B; D; E
			2) (*E,E*)-2,4-heptadienal (0.1% in triacetin); 400 μL/L tea	C; E
Vegetative taints	Hay/dried grass	Slightly sweet aromatics associated with dried grass or hay.	1) *o*-cresol (0.1% in propylene glycol); 640 μL/L tea	A; C; E
			2) nonanal ('hay' capsule); 1 capsule/L tea	G; H
	Green grass	Aromatics associated with freshly cut grass/stale cut grass.	1) (*Z*)-3-hexen-1-ol (1% in propylene glycol); 300 μL/L tea	A; B; D; E
	Rotting plant water	Aromatics associated with the old and rotting vase water of cut flowers.	1) methionol (0.1% in propylene glycol); 960 μL/L tea	B; D; E
			2) ethanethiol ('mercaptan' capsule); 1 capsule/2 L tea	E; H
	Seaweed	Aromatics associated with seaweed.	1) 4-oxoisophorone (0.1% in propylene glycol); 1.6 mL/L tea	C; E
General taints	Burnt caramel	Aromatics associated with burnt sugar, burnt caramel or burnt caramelized vegetables.	1) 4-hydroxy-2,5-dimethyl-3(2*H*)-furanone (10% in triacetin); 20 μL/L tea	A; E
			2) 3-ethyl-2,5-dimethylpyrazine ('burnt caramel' capsule); 1 capsule/L tea	A; E
	Medicinal/rubber	Aromatics associated with Band-Aid®.	1) *p*-ethylphenol (1% in propylene glycol); 160 μL/L tea	A; B; D; E
			2) *p*-cresol (1% in propylene glycol); 600 μL/L tea	A; B; C; D; E
	Dusty	Earthy aromatics associated with dust from a gravel road or soil.	1) L-borneol (1% in propylene glycol); 10 μL/L tea	B; E
			2) (*R/S*)-geosmin ('dry earthy' capsule); 1 capsule/L tea	E; G; H
	Musty/mouldy	Mouldy aromatics associated with mildew or damp cellars.	1) 2,4,6-trichloroanisole (1 g/mL ethanol); 10 μL/L tea	G; H
			2) 2-ethylfuran (1% in propylene glycol); 480 μL/L tea	C; E

[a] Chemical, and its dilution in brackets; volume of diluted chemical added to base tea.

[b] Information sources: A, Acree and Arn (2004); B, Arctander (1969); C, Kerry EMEA (Durban, South Africa; www.kerry.com); D, Sigma-Aldrich (St Louis, MO, USA; www.sigmaaldrich.com); E, The Good Scents Company (Oak Creek, WI, USA; www.thegoodscentscompany.com); F, Rooibos Ltd (Clanwilliam, South Africa); G, AROXA™ (Cara Technology, Leatherhead, UK; www.aroxa.com); H, FlavorActiV™ (Aston Rowant, UK; www.flavoractiv.com).

[c] Fynbos is natural shrubland vegetation occurring in the Western Cape, South Africa.

Table 6
Updated 'fermented' honeybush aroma lexicon with chemical-based reference standards.

General attributes	Specific attributes	Description of aroma attributes	Reference standards [a]	Information sources [b]
Floral aroma	Fynbos-floral	Sweet, floral aroma note associated with the flowers of fynbos[c] vegetation	1) (*R/S*)-linalool (1% in propylene glycol); 400 μL/L tea	A; B; C; D; E
			2) geranyl acetone (1% in propylene glycol); 240 μL/L tea	B; C; D; E
	Rose geranium	Floral aroma note associated with the rose geranium plant	1) nerol (1% in propylene glycol); 160 μL/L tea	A; B; D; E
			2) geraniol (1% in propylene glycol); 240 μL/L tea	A; B; D; E
	Rose perfume	Floral aroma note associated with rose petals or rosewater (Turkish delight)	1) phenethyl acetate (1% in propylene glycol); 200 μL/L tea	A; B; D; E
			2) phenylacetaldehyde (1% in propylene glycol); 120 μL/L tea	A; B; D; E
Plant-like aroma	Woody	Aromatics associated with dry bushes, stems and twigs of the fynbos[c] vegetation	1) guaiacol (1% in propylene glycol); 100 μL/L tea	A; B; D; E
			2) 2-acetyl-5-methylfuran (1% in propylene glycol); 200 μL/L tea	B; D; E
	Pine	Aroma reminiscent of pine needles	1) bornyl acetate (1% in propylene glycol); 200 μL/L tea	B; D; E
			2) camphene (1% in propylene glycol); 800 μL/L tea	A; B; C; D; E
Fruity aromas	Apricot	Sweet-sour aroma reminiscent of apricot jam or dried apricot	1) maltyl isobutyrate (1% in propylene glycol); 920 μL/L tea	D; E
			2) 2-methylbutanal (0.1% in triacetin); 740 μL/L tea	B; C; D; E
	Apple	The sweet, slightly sour aroma of cooked apples	1) (*E*)-2-hexen-1-al (1% in propylene glycol); 140 μL/L tea	B; C; D; E
			2) ethyl isobutyrate (1% in propylene glycol); 160 μL/L tea	B; D; E
	Lemon/ lemongrass	Aromatics associated with general impression of fresh lemons or lemongrass	1) 6-methyl-5-hepten-2-one (1% in propylene glycol); 500 μL/L tea	B; C; D; E
			2) (*R/S*)-ocimene (1% in propylene glycol); 640 μL/L tea	B; C; D; E
	Raisin	Sweet aroma note reminiscent of "hanepoot" raisin	1) β-cyclocitral (0.1% in propylene glycol); 200 μL/L tea	B; C; D; E
			2) β-damascenone (1% in propylene glycol); 80 μL/L tea	B; C; D; E
Sweet-associated aromas	Fruity-sweet	Sweet-sour aromatic reminiscent of non-specific fruit	1) geranyl isovalerate (1% in propylene glycol); 320 μL/L tea	B; D; E
			2) propyl propionate (1% in propylene glycol); 320 μL/L tea	B; C: D; E
	Honey	Aromatics associated with the sweet fragrance of fynbos[c] honey	1) methyl phenylacetate (1% in propylene glycol); 120 μL/L tea	B; D; E
	Caramel	Sweet aromatics characteristic of molten sugar or caramel pudding	1) ethyl maltol (1% in propylene glycol); 240 μL/L tea	D; E
			2) maltyl isobutyrate (1% in propylene glycol); 600 μL/L tea	D; E
	Fynbos-sweet	The sweet aroma note reminiscent of the fynbos[c] plant	1) phenethyl alcohol (1% in propylene glycol); 400 μL/L tea	D; E
			2) levulinic acid (1% in propylene glycol); 1.36 mL/L tea	D; E
Spicy aroma	Sweet spice	Sweet, woody and spice aroma, including ground cinnamon/cassia bark	1) dihydrocoumarin (1% in propylene glycol); 200 μL/L tea	D; E
			2) (*E,E*)-2,4-heptadienal (0.1% in triacetin); 500 μL/L tea	C; E
Nutty aroma	Nutty	Aromatics associated with fresh walnuts or chopped almonds	1) benzaldehyde (1% in triacetin); 150 μL/L tea	C; E
			2) 2-acetylpyrrole (1% in propylene glycol); 1 mL/L tea	D; E
Vegetative taints	Hay/dried grass	Slightly sweet aroma associated with dried grass or hay	1) *o*-cresol (0.1% in propylene glycol); 640 μL/L tea	A; C; E
			2) nonanal ('dry hay' capsule); 1 capsule/L tea	F; G
	Green grass	Aroma associated with cut green grass or decomposing cut grass	1) (*Z*)-3-hexenol (1% in propylene glycol); 800 μL/L tea	A; B; D; E
	Rotting plant water	Aromatics associated with the old and rotting vase water of cut flowers.	1) dimethyl trisulfide (0.1% in propylene glycol); 10 μL/L tea	D; E
			2) ethanethiol ('mercaptan' capsule); 1 capsule/1 L, then 800 mL base tea added to 200 mL of spiked solution	E; G
	Cooked vegetables	An overall aroma note associated with canned/cooked vegetables	1) methional (0.1% in propylene glycol); 160 μL/L tea	D; E
			2) methionol (0.1% in propylene glycol); 940 μL/L tea	D; E
General taints	Burnt caramel	Aroma associated with burnt carbohydrates, especially burnt sugar	1) 4-hydroxy-2,5-dimethyl-3(2*H*)-furanone (10% in triacetin); 40 μL/L tea	A; E
			2) 3-ethyl-2,5-dimethylpyrazine ('burnt caramel' capsule); 1 capsule/1 L, then 750 mL base tea added to 250 mL of spiked solution	A; E; G
	Medicinal/ rubber	Aromatic characteristic of Band-aid® and antiseptic (TCP)	1) *p*-ethylphenol (1% in propylene glycol); 200 μL/L tea	A; B; D; E
			2) *p*-cresol (1% in propylene glycol); 600 μL/L tea	A; B; D; E
	Dusty	Earthy aroma associated with dry dirt road	1) L-borneol (1% propylene glycol); 5 μL/L tea	B; E
			2) (*R/S*)-geosmin ('dry earth' capsule); 1 capsule/L tea	E: F; G
	Smoky	Smoky aroma note associated with burning hay/grass or tobacco	1) guaiacol (1% in propylene glycol); 350 μL/L tea	A; B; D; E
			2) 3-ethylpyridine (0.1% in propylene glycol); 40 μL/L tea	D; E

[a] Chemical, and its dilution in brackets; volume of diluted chemical added to base tea.
[b] Information sources: A, Acree and Arn (2004); B, Arctander (1969); C, Kerry EMEA (Durban, South Africa; www.kerry.com); D, Sigma-Aldrich (St Louis, MO, USA; www.sigmaaldrich.com); E, The Good Scents Company (Oak Creek, WI, USA; www.thegoodscentscompany.com); F, AROXA™ (Cara Technology, Leatherhead, UK; www.aroxa.com); G, FlavorActiV™ (Aston Rowant, UK; www.flavoractiv.com).
[c] Fynbos is natural shrubland vegetation occurring in the Western Cape, South Africa.

For eight rooibos and five honeybush attributes the mean intensity values of one/both aroma chemical(s) in the base tea and the respective reference teas did not differ significantly ($P \geq 0.05$). It is important to note that reference samples that exhibited the highest perceived intensity of the target aromas were selected from our in-house sample collection.

The updated rooibos and honeybush lexicons are provided in Tables 5 and 6, illustrating general and specific sensory attributes, attribute descriptions, reference standard information (compound and concentration) and sources of information.

4. Conclusions

Chemical reference standards have been tested and validated for the rooibos and honeybush sensory lexicons, using the typicality score as parameter for inclusion. For several target aroma notes high typicality scores were obtained, yet single aroma chemical compounds did not fully represent complex target aromas such as 'fynbos-floral' or 'apricot'. An important outcome was the high typicality scores for aroma notes that are generally unknown, but typical of rooibos and honeybush: levulinic acid for 'fynbos-sweet' (honeybush), and isophorone for 'rooibos-woody' (rooibos). The use of a large sample set of honeybush also enabled updating of the generic honeybush sensory wheel. Future research could evaluate different concentrations of selected chemicals to fine-tune the reference standards.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

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