Arabian Journal of Chemistry (2022) 15, 104147

King Saud University

Arabian Journal of Chemistry

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ORIGINAL ARTICLE

Improving the quality of instant tea with low-grade tea aroma

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Received 21 January 2022; accepted 20 July 2022
Available online 25 July 2022

KEYWORDS

Broken mixed fannings;
Instant tea;
Refused tea;
Tea aroma;
Volatile compounds

Abstract Refuse tea is industrially identified as broken mixed fannings (BMF). It is a primary raw material for instant tea production. The aroma escape during the process affects the quality of instant tea used for application in the food and beverage industries. Capturing and adding back aroma to the instant tea is commercially important to overcome this drawback. Hence, the study was focused on profiling the aroma of BMF and respective instant tea based on low country (0–300 m MASL) and upcountry (>600 m MASL) elevation categories of Sri Lanka to identify the impact of growing elevation on aroma profiles to explore blending proportions for better aroma profiles. Furthermore, volatiles that escaped with steam during the instant tea production process were condensed, and this aroma extract was added back to the tea concentrate prior to spray drying to produce the aroma blended instant tea sample (ABIT). Solid-phase microextraction followed by gas chromatography–mass spectrometry was used to identify the volatile compounds in the head-space of instant tea, BMF and ABIT samples. BMF samples significantly contained linalool (9.94 ± 2.94 %), *cis*-linalool oxide (Furanoid) (4.69 ± 1.80 %), geraniol (2.47 ± 1.44 %), and 3-hexen-1-ol (3.46 ± 2.06 %); however, the volatile profile of instant tea samples had high amounts of hydrocarbons (51.60 ± 9.34 %) that did not contribute to creating a pleasant aroma for the product. Hexanal, geraniol, citral, *cis*-jasmon, α-ionone, nerol and E-2-Z-4-hexadienal are characteristic in upcountry BMF and were absent in the low country tea, indicating a difference in the aroma profiles with elevation. ABIT sample showed that there is a clear improvement in the aroma profile by retaining compounds such as linalool (5.48 %), geraniol (0.95 %), hotrienol (0.92 %), and

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Peer review under responsibility of King Saud University.

Production and hosting by Elsevier

https://doi.org/10.1016/j.arabjc.2022.104147

γ-muurolene (0.22 %), which give a pleasant flowery aroma to the product. This study concluded the possibility to improve the aroma profile of instant tea to give the unique aroma of Ceylon tea as per customer requirements in an economically viable manner and this technology can be applied globally.

1. Introduction

"Ceylon tea" produced in Sri Lanka is acclaimed as the best tea globally and has a high demand in international trade. Sri Lanka is the third-largest exporter of tea, with an export market value of 1.2 billion USD (Export Development Board - Sri Lanka, 2021). The aroma of tea plays an important role in determining the quality of tea; therefore, the popularity of Ceylon tea is associated with its superior aroma (Kasuga et al., 2010). Volatile compounds in tea have been researched since the 1930s, and approximately 600 constituents have been characterized in tea leaves or in beverages (Schuh and Schieberle, 2006). The organoleptic appeal in tea aroma correlates to the presence of volatile flavor components in the product (Zheng et al., 2016).

Instant tea is generally made from fermented black tea (Kraujalyte et al., 2016), green (Zhang et al., 2020) and Pu-erh (Du et al., 2019) tea; however, Broken Mixed Fannings (BMF), which consists of sweepings, red leaves, fluff, mature stalks, and fiber particles that accumulate at different stages of the black tea manufacturing process, is commonly used because of its lower price, as it is considered a product with no or low value in the industry. The identification of aroma compounds in instant tea and the quantification of their relative concentrations have been performed by many researchers (Ye et al., 2014; Du et al., 2019; Zhang et al., 2020), and the composition and relative concentrations of aroma compounds in spray-dried instant tea are different from those in black tea (Kraujalyte et al., 2016).

The aroma profile of instant tea can vary with the raw material obtained from different elevational categories and with the processing technology. During the manufacturing of cold soluble instant tea, volatile chemicals with desirable aroma notes can be escaped, thus affecting the aroma profile of instant tea and instant tea-based products.

Approximately 4–6 % of tea production in Sri Lanka is discarded as refuse tea (BMF). Studies have shown that BMF is a potential raw material for aroma extraction and instant tea production (Perera et al., 2015); however, information is scarce on assessing the aroma profile of BMF to enhance the aroma of instant tea or black tea.

Therefore, this study was initiated to determine the quality variations of aroma compounds obtained from BMF of two different elevation categories of Sri Lanka and the instant tea produced from the respective BMF as well as the effectiveness of adding aroma compounds on the quality changes of instant tea.

2. Materials and methodology

2.1. Plant material

Broken mixed fannings which is a collected as sweepings, red leaves, fluff, mature stalks, and fiber particles that accumulate at different stages of the black tea manufacturing process, produced from tea leaves (two leaves and bud) plucked from three different tea estates, Pedro and Nuwara Eliya from the elevation category upcountry (UC) (> 600 m MASL) and Halgolla estate from the low country (LC) (< 300 m MASL), were used for the study. All states grow Cultivar TRI 2025 and follow similar agricultural and management practices, "two leaves and bud" plucking standard, as well as tea manufacturing practices of the Orthodox (Rotorvane) type under the same company brand.

Approximately 2 kg of BMF processed by the orthodox manufacturing method which includes withering, rolling and drying, was collected during August 2020. The collected samples were packed in low-density polyethylene bags of 1 kg each and sealed in aluminum foil-lined Kraft paper bags before being transported to Hayleys Global Beverages (Pvt) ltd., Dickoya, Hatton, where tea liquor extraction was performed.

2.2. Preparation of instant tea

2.2.1. Extraction and concentration of the tea liquor

Tea liquor extractions from BMF obtained from three different states were carried out separately. Approximately 500 g of BMF were brewed with 4.5 L of boiled distilled water (tea leaf to water ratio of 1:9 w/w) for 45 min while maintaining the temperature at approximately 90 °C. The extract obtained was filtered through a nylon mesh of aperture size of 100 μm to remove fine insoluble solids in the extract. The extract was then concentrated to a total soluble solid content of approximately 25 g/100 mL using a heating magnetic stirrer (Witeg SMHS-6, Witeg Labortechnik GmbH, Germany) while maintaining the temperature of the tea liquor at approximately 75 °C. The concentrated tea was kept under refrigerated conditions at 4 °C to prevent spoilage.

2.2.2. Spray drying of the tea concentrate

Drying of the concentrates was carried out at the Palmyrah Research Institute, Kaithady, Jaffna. The concentrated samples were transported in a Styrofoam box filled with ice to avoid spoilage. The concentrates were spray dried to obtain instant black tea powder using a spray dryer (SP-1500, Shanghai Sunyitech Co., ltd, Pudong Shanghai, China) while maintaining the inlet temperature at 175–185 °C and outlet temperature at 70–80 °C.

2.2.3. Aroma recovery

A mix of BMF samples obtained from each estate was used for aroma recovery studies, which is similar to commercial practice. A composite sample of BMF, 35 % from Pedro estate, 35 % from Nuwara Eliya estate, and 30 % from Halgolla estate, was prepared and brewed as described earlier (100 g of BMF brewed with 900 mL of hot water at 90 °C). The brew was condensed in a rotary evaporator (Witegvapour, Witeg Labortechnik GmbH, Germany) at 75 °C until a total soluble solid content of approximately 25 g/100 mL was achieved and the steam was passed through the condenser of the rotary evaporator. At the end of the process approximately 15 mL of aroma condensate was collected and this process was repeated until approximately 300 mL of condensate was collected.

2.2.4. Aroma blended sample preparation

To determine the optimum volume of aroma extract to be added back to the tea concentrate, a sensory evaluation was conducted. Since the instant tea industry practices a variety of extraction and concentration techniques resulting in different final dry solid and Brix levels, the amount of aroma condensate added was taken as a percentage of the dry solid content in the tea extract. To that end, the dry solid concentration of the composite sample's tea concentrate was calculated, and aroma condensate equal to 0 % (control), 15 %, 30 %, 45 %, 60 %, and 75 % (w/w) of the dry solid content was added back to 100 mL of the tea concentrate obtained from the composite sample. The diluted tea concentrates thus obtained were rated for the quality of the perceived aroma by 15 trained panelists using a 9-point hedonic scale. The scores obtained by the diluted samples were analyzed against the scores obtained by the control using a Friedman test and a Wilcoxon signed-rank test to determine a statistically significant difference.

The minimum aroma added-back percentage was thus determined and was added to the tea concentrate made from the composite BMF sample and was also spray-dried to produce an aroma blended instant tea (ABIT) sample.

2.3. Aroma profile analysis

2.3.1. Sample preparation: Headspace solid-phase microextraction (HS-SPME)

To analyze the aroma profile of BMF and instant tea, a slightly adjusted procedure outlined by Yang et al. (2018) for headspace solid-phase microextraction with gas chromatography–mass spectrometry (HS-SPME/GC–MS) was used. A 50/30 µm DVB/CAR/PDMS Stableflex (2 cm) 24 Ga (Supelco, Bellefonte, PA, USA) SPME fiber fixed to a Manual SPME holder (Supelco, USA) was used for this analysis. Before each run, the SPME fiber was conditioned for 20 min by inserting them into the GC injector port while maintaining the injection port temperature at 250 °C and oven temperature at 150 °C.

BMF samples were milled using a "Panasonic MX-AC400" grinding mill (Panasonic Corporation, India) and were sieved with a stainless steel sieve of mesh size 40 (420 µm aperture) to ensure homogeneity. Accurately 0.50 g of BMF sample was added to a 12 mL headspace vial and closed by a PTFE/silicone septum (Agilent Technologies, USA). The vial was equilibrated in a hot water bath maintained at 90 °C for 20 min. Then, the needle of the SPME fiber was inserted into the vials by manually penetrating the septum, and the fibers were exposed to the headspace above the samples for another 20 min while maintaining the equilibrium temperature at 90 °C. Afterward, the fiber was removed and manually inserted directly into the injection port of the GC–MS, and the analytes were thermally desorbed in spitless mode at 280 °C for 4 min. The same procedure was repeated for an accurately measured quantity of 0.5 g of instant tea as well.

2.3.2. GC–MS operating conditions

GC–MS was performed with an Agilent Technologies 7890A GC system coupled to an Agilent Technologies 5975C inert XL EI/CI MSD with a triple-axis detector (Agilent Technologies, USA). Samples were analysed using an HP-5MS UI (30 m × I.D. 0.25 mm × film thickness 0.25 µm) GC column (Agilent Technologies, USA). The analytical conditions used were as follows: The oven temperature was held at 40 °C for 0 min, raised to 190 °C at a rate of 3 °C°C/min, held for 2 min, increased to 280 °C at 10 °C°C/min, and held for 15 min. Spitless mode was used; helium (99.9995 % purity) was used as the carrier gas at a rate of 1.0 mL/min. The mass spectrometer was operated in electron-impact (EI) ionization mode. The scan range was 50–550 m/z. The temperatures at the ionization source and interface were 230 and 150 °C, respectively.

In the qualitative analysis, compound similarity was identified by comparing the obtained spectra with those of reference compounds from the National Institute of Standards and Technology (NIST 08) database and by Van Den Dool & Kratz Retention indices which were calculated using C9 to C16 n-alkane compounds which were detected with an accuracy above 90 % The relative peak area % of each volatile was obtained over the total peak area. Total percentages of different chemical classes were determined by the sum of the relative peak area percentages of the compounds belonging to each chemical class. The initial report generated by GC–MS contained approximately 150–200 detected compounds in the headspace of each sample. The detected compounds were screened for any known artificial peaks, and the final volatile profile thus obtained was used for analysis. The compounds that were identified with an accuracy percentage of < 80 % were excluded as unknown compounds. The relative area percentage of each component was obtained by peak area normalization.

3. Results and discussion

3.1. Aroma profiles: instant tea and BMF

The volatile profiles of BMF with qualitative differences were assessed between estates belonging to different growing elevations of Sri Lanka: high (>600 MASL) and low (<300 MASL) and found that linalool, cis - linalool oxide (Furanoid), geraniol, 3-hexen-1-ol, phenylacetaldehyde, phenylethyl alcohol, eugenol, (E,E)-2,4-heptadienal and methyl salicylate were present in high intensity in Broken Mixed Fannings (BMF) (Table 1 and Fig. 1). However, these compounds were not detected or were detected in low relative percentages in the instant tea samples made according to the typical instant tea manufacturing process. Fig. 1a & b show that the abundance values (ion count) in the chromatograms of instant tea samples were lower than those of BMF. The observations indicate that most of the volatile compounds that were present in BMF had evaporated at the concentration (heat evaporation) stage of instant tea processing, resulting in a product with low concentrations of volatile aroma compounds. The use of high temperatures for extraction also causes a substantial loss in the aroma and flavour of the product, as reported by Mason and Zhao (1994). The production of aroma blended instant tea (ABIT) has shown that some of the lost aroma can be retained in the final product, as shown in Fig. 1d.

3.2. Aroma profiles of instant tea and its raw material (BMF)

It was determined that there is a clear difference between instant tea and BMF leaf tea, where the compounds detected

Table 1 Relative peak area percentages of the 10 most prominent peaks in GC–MS chromatograms of Aroma blended instant tea (ABIT), Nuwara Eliya BMF (NEBMF), Nuwara Eliya instant tea (NEIT), Pedro BMF (PBMF), Pedro instant tea (PIT), Halgolla BMF (HBMF) and Halgolla instant tea (HIT).

Compound	RI	Identification	Aroma Blended Instant Tea (ABIT) RTA%	Nuwara Eliya BMF (NEBMF)	Nuwara Eliya Instant Tea (NEIT)	Pedro BMF (PBMF)	Pedro Instant Tea (PIT)	Halgolla BMF (HBMF)	Halgolla Instant Tea (HIT)	Aroma Note	Reference for the aroma note
3-Hexen-1-ol	864	MS/RI	6.67	2.32	–	5.83	–	2.22	–	Green	(Nie et al., 2020)
Unknown*	874	–	2.48	–	–	–	–	–	–	N/A	_
Nonane	900	MS/RI	–	–	–	–	–	–	4.85	N/A	_
Butyrolactone	911	MS/RI	–	–	3.04	–	–	–	3.6	Creamy	(Guo et al., 2019)
Benzaldehyde	958	MS/RI	3.3	–	–	–	–	–	–	Sweet[a,b], bitter [b], oily [b], almond[a,b]	(Lee et al., 2013) [a], (Vararu et al., 2016) [b]
4-Ethyltoluene	959	MS/RI	–	–	3.88	–	–	–		N/A	–
2-Ethyltoluene	959	MS/RI	–	–	–	–	–	–	10.04	N/A	–
Heptane, 2,2,4,6,6-pentamethyl-	989	MS	2.45	13.68	–	11.09	–	10.82	–	N/A	–
Benzene, 1,2,3-trimethyl-	992	MS	–	–	–	–	–	–	3.09	N/A	
Decane	1000	MS/RI	–	–	3.57	–	–	–	11.76	Alkane odour	(Mahmud et al., 2020)
Unknown*	1003	–	–	–	4.58	–	3.69	–	–	N/A	–
Unknown*	1022	–	–	–	–	–	–	–	2.32	N/A	–
Unknown*	1024	–	–	2.76	–	–	–	–	–	N/A	–
2,2,4,4-Tetramethyloctane	1024	MS	–	–	–	2.17	–	–	–	N/A	–
D-Limonene	1026	MS	–	–	–	–	–	4.4	–	Lemon, orange	(Vararu et al., 2016)
Benzyl Alcohol	1033	MS/RI	–	–	2.48	1.17	–	–	–	Fruity[a], Roasted[b]	(Ito et al., 2002) [a], (Zhang et al., 2019)[b]
Benzene, 1-methyl-3- propyl-	1049	MS/RI	–	–	–	–	–	–	2.19	N/A	–
cis-Linaloloxide	1071	MS/RI	2.83	1.43	–	1.7	–	3.29	–	Leafy, citrus	(Ito et al., 2002)
cis-Linalool Oxide (Furanoid)	1087	MS/RI	5.58	3.12	–	4.3	–	6.66	–	Leafy, citrus	(Ito et al., 2002)
Undecane	1100	MS/RI	–	–	5.44	–	–	–	5.94	Alkane odour	(Mahmud et al., 2020)
Linalool	1101	MS/RI	5.48	8.96	–	13.25	–	7.61	–	Floral	(Ni et al., 2020)
Phenylethyl Alcohol	1112	MS/RI	–	–	–	–	–	1.8	–	Floral	(Zhang et al., 2019)
N-Ethylsuccinimide	1135	MS/RI	–	1.45	3.12	–	–	–	2.88	Burnt	(Dai et al., 2020)
2H-Pyran-3-ol, 6-ethenyltetrahydro-2,2,6-trimethyl-(Linalool Oxide Pyranoid / Epoxylinaol)	1174	MS/RI	–	–	–	–	–	3.13	–	Sweet, woody	(Gong et al., 2017)
Methyl salicylate	1193	MS/RI	–	–	–	3.64	–	–	–	Floral[a], green[b]	(Deng et al., 2017) [a], (Ito et al., 2002) [b]

Table 1 (*continued*)

Compound	RI	Identification	Aroma Blended Instant Tea (ABIT) RTA%	Nuwara Eliya BMF (NEBMF)	Nuwara Eliya Instant Tea (NEIT)	Pedro BMF (PBMF)	Pedro Instant Tea (PIT)	Halgolla BMF (HBMF)	Halgolla Instant Tea (HIT)	Aroma Note	Reference for the aroma note
Dodecane	1200	MS	2.42	1.89	3.74	1.75	–	–	2.99	Alkane odour	(Mahmud et al., 2020)
Geraniol	1256	MS/RI	–	1.45	–	3.49	–	–	–	Sweet, floral	(Ni et al., 2020)
Unknown*	1280	–	–	–	–	–	2.1	–	–	N/A	–
Tridecane	1300	MS/RI	–	–	1.84	–	–	–	–	Alkane odour	(Mahmud et al., 2020)
Dodecane, 2,6,11-trimethyl-	1327	MS/RI	–	–	–	–	2.6	–	–	N/A	–
Copaene	1375	MS/RI	–	–	–	–	–	1.62	–	Woody, spicy	(Iwasa et al., 2015)
Caryophyllene	1418	MS/RI	–	–	–	–	–	6.2	–	Mint, spice, wood	(Gao et al., 2017)
Unknown*	1496	–	–	–	–	–	3.6	–	–	N/A	–
2,4-Di-*tert*-butylphenol	1513	MS/RI	10.1	–	–	–	4.24	–	–	Green[a], Phenolic[b]	(Zhang et al., 2019) [a], (Vararu et al., 2016) [b]
Unknown*	1524	–	–	–	–	–	3.27	–	–	N/A	–
Unknown*	1541	MS	–	–	1.99	–	4.18	–	–	N/A	–
Unknown*	1552	MS	–	–	–	–	2.31	–	–	N/A	–
Phenol, 2,4,6-tris(1,1-dimethylethyl)-	1608	MS	–	1.63	–	–	–	–	–	N/A	–
Bicyclo[4.4.0]dec-1-ene, 2-isopropyl-5-methyl-9-methylene-	1641	MS/RI	2.9	–	–	–	–	–	–	N/A	–
2-Bromo dodecane	1704	MS/RI	–	–	–	–	2.03	–	–	N/A	–
Unknown*	1743	–	–	–	–	–	1.86	–	–	N/A	–

RI: Van Den Dool and Kratz Retention Index on a HP5-MS Column; RTA%: Relative Peak Area Percentage; N/A: Information regarding the aroma note of the compound is not available in the literature.

* Compounds that were not accurately identified by MS and RI.

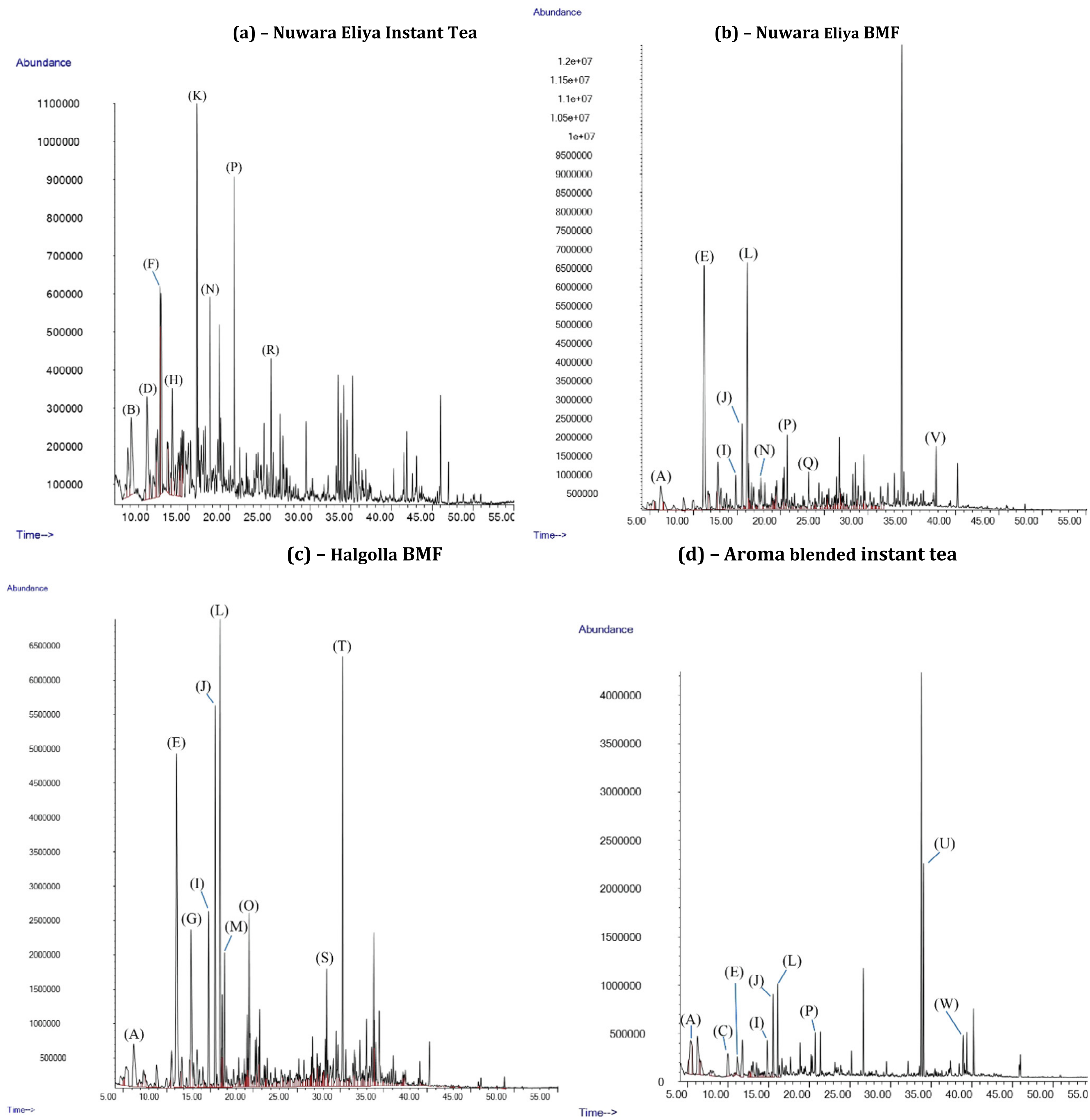

Fig. 1 (a) Total ion chromatogram of Nuwara Eliya Instant Tea sample (b) Total ion chromatogram of Nuwara Eliya BMF sample (b) Total ion chromatogram of Halgolla BMF sample (d) Total ion chromatogram of Aroma Blended Instant Tea sample. Indicated are the peaks with the highest relative peak area percentages (excluding known artificial peaks); (A) 3-Hexen-1-ol, (B) Butyrolactone, (C) Benzaldehyde, (D) 4-Ethyltoluene (E) Heptane, 2,2,4,6,6-pentamethyl-, (F) Decane, (G) D-Limonene, (H) Benzyl Alcohol, (I) *cis*-Linaloloxide, (J) *cis*-Linalool Oxide (Furanoid), (K) Undecane, (L) Linalool, (M) Phenylethyl Alcohol, (N) *N*-Ethylsuccinimide, (O) 2H-Pyran-3-ol, 6-ethenyltetrahydro-2,2,6-trimethyl- [Epoxylinalol], (P) Dodecane, (Q) Geraniol, (R) Tridecane, (S) Copaene, (T) Caryophyllene, (U) 2,4-Di-*tert*-butylphenol, (V) Phenol, 2,4,6-tris(1,1-dimethylethyl)-, (W) Bicyclo[4.4.0]dec-1-ene, 2-isopropyl-5-methyl-9-methylene-.

in the highest relative peak areas for instant tea have not previously been identified to have a particular aroma note. In some cases, the most prominent compounds in instant tea gave an alkane or a burnt aroma note. Table 1 shows the 10 most prominent peaks present in the tested samples.

3.2.1. Nuwara Eliya instant tea (NEIT) and Nuwara Eliya BMF (NEBMF)

Nuwara Eliya Tea Estate produces a tea, which has been described as light and bright with a delicate character which gives off fruity & floral notes. The tea is grown at an elevation of 1889 MASL within the *Mahagastotte* Valley. As mentioned in Table 2, the percentages of heterocyclic compounds, hydrocarbons, esters, and aromatic alcohols are higher in NEIT than in NEBMF. Apart from the aroma compounds mentioned in Table 1, which were detected with high relative peak area percentages, several pyrroles, pyrrolidines, pyrans and tetrahydrofurans were also detected in NEIT. These heterocyclic compounds included pyrrolidine-2,4-dione (ammoniacal, animal-like odour), 2H-Pyran-3-ol, 6-ethenyltetrahydro-2,2,6-trimethyl- (Epoxylinalol) (floral aroma) and 2-acetylpyrrole (bready, walnut, licorice-like aroma), whose aroma notes have been previously identified by Gong et al. (2017) and Zhang et al. (2019).

Among other aromatic compounds, terpenes such as cubene (sharp), geranyl acetone (floral, fruity), and o-cymene (grass/flower, orange) were detected in the NEIT sample, and these aroma notes have been previously reported by Dai et al. (2020) as a part of the aroma profile of green tea. Results confirm that the Instant tea from Nuwara Eliya estate still secures its natural tea aroma notes similar to green tea. In the NEBMF sample, the number of detected aroma compounds was much larger, with many of them being terpenes or terpenoids, having a floral or fruity note, which could be the reason why Nuwara Eliya tea is highly regarded for its aroma.

In addition, NEBMF contained furfural, which Chen et al. (2013) has been reported to have an almond/burnt sugar aroma and aldehydes such as hexanal, heptanal, and (E,E)-2,4-hexadienanal, which gives a green aroma note, as per the findings of Panten and Surburg (2015).

3.2.2. Pedro instant tea (PIT) and Pedro BMF (PBMF)

Pedro estate is the tea estate located at the highest elevation (1910 MASL) in Sri Lanka (Nianthi, 2019) and is known to produce high-quality black tea with a light, bright & fruity character. The PBMF sample contained higher total peak area percentages for terpenes, heterocyclic compounds, hydrocarbons, aldehydes and ketones, esters, and alcohols compared to PIT (Table 2).

Unlike NEIT, the PIT sample detected a higher number of terpenes and a lower number of heterocyclic compounds (Table 2). Of the detected volatiles in the headspace of PIT in smaller relative percentages, compounds that give off a floral/fruity aroma, such as α-terpineol, nerolidol, geranyl acetone, benzyl alcohol, and benzeneacetaldehyde, were detected, suggesting a more pleasant aroma in instant tea made with BMF from Pedro estate. Apart from that, butyrolactone, which imparts a sweet/cream aroma (Chen et al., 2013; Guo et al., 2019), and caprolactone, which gives a cereal-like aroma, have also been detected (Wei et al., 2013). 1H-Pyrrole-2-carboxaldehyde, 1-ethyl- and *N*-ethylsuccinimide, which have been reported by Dai et al. (2020) and Guo et al. (2021) to have a burnt, roasted or smoky aroma, could play a key role in the strong aroma that is characteristic of spray-dried instant tea. However, *N*-ethylsuccinimide is detected in low relative percentages in PIT, although it has been detected in high percentages in NEIT and HIT samples. In BMF from Pedro estate, similar to Nuwara Eliya BMF, several aldehydes and terpenes have been detected in low relative percentages. Previous studies have identified these aldehydes (hexanal, heptanal, 2,4-hexadienanal, (E,E)-, 2,4-heptadienal, (E,E)-) as having green, fatty and grassy aromas (Guo et al., 2021).

Methyl salicylate has been noted as a compound present in tea from Uva province (Kasuga et al., 2010). This could probably be due to the harsh growing conditions in that region and this compound has been detected in PBMF. These similar climatic conditions may have influenced the production of aroma precursors in the tea leaves. Among esters, methyl jasmonate, which is an important fatty acid derivative that gives oolong tea a floral aroma, has also been detected in BMF from Pedro estate, along with 3-hexen-1-ol, acetate, (Z)- which gives a fruity, banana-like aroma (Feng et al., 2021; Guo et al., 2021). Furthermore, indole, which is an important constituent in jasmine aroma, has also been detected in the headspace of PBMF.

Table 2 Distribution of chemical classes in the tested samples in terms of relative peak area.

Compound (%)	ABIT	NEIT	NEBMF	PIT	PBMF	HIT	HBMF
Total Unknown*	20.75	18.71	16.41	44.62	6.97	20.77	8.51
Non aromatic alcohols	6.83	ND	2.70	ND	6.14	ND	2.22
Aromatic alcohols	3.02	3.90	1.73	1.38	2.09	1.41	2.64
Ethers	2.28	ND	ND	ND	ND	ND	ND
Esters	5.05	3.50	2.55	4.62	6.87	1.43	1.99
Aldehydes	4.42	0.87	2.55	0.40	2.56	0.99	1.87
Ketones	1.09	0.92	1.55	1.33	0.97	ND	0.83
Acids	ND	ND	ND	0.34	ND	ND	ND
Hydrocarbons	23.60	50.86	39.08	35.50	38.42	61.97	24.39
Lactones	1.54	3.04	1.04	1.18	1.07	3.60	ND
Nitrogenous compounds	0.95	ND	ND	1.23	0.75	0.57	0.13
Uncategorized (Miscellaneous)	0.54	2.63	1.55	3.56	1.77	1.01	1.14
Heterocyclic compounds	14.15	8.69	7.95	1.22	7.93	4.73	16.01
Terpenes	15.76	2.31	22.89	4.28	24.42	3.24	40.37

(ABIT – Aroma Blended Instant Tea, NEIT- Nuwara Eliya Instant Tea, NEBMF- Nuwara Eliya BMF, PIT- Pedro Instant Tea, PBMF- Pedro BMF, HIT- Halgolla Instant Tea, HBMF- Halgolla BMF). ND- Not detected.

* Unknown compounds refer to the peaks that were not accurately identified by comparing the obtained spectra with those of reference compounds from NIST 08.

3.2.3. Halgolla instant tea (HIT) and Halgolla BMF (HBMF)

Halgolla estate is situated in a low country (207 MASL) in the Kegalle district of Sabaragamuwa region. The high rainfall experienced in the Kelani vally has resulted in lush tea bushes that produces a tea which is said to have a thick colour and a robust flavour. BMF sourced from Halgolla had the highest number of different terpene compounds as well as the highest total peak area percentages compared to the other samples. Compared to HIT, the HBMF sample contained higher percentages of terpenes, heterocyclic compounds, aldehydes, esters, and alcohols.

When compared with other instant tea samples, the number of detected compounds was lowest in the HIT sample. Among those that were detected in low relative percentages, 1H-pyrrole-2-carboxaldehyde, which gives a roasted nutty aroma, benzyl alcohol and benzeneacetaldehyde, which give a floral and flowery aroma, and *N*-ethylsuccinimide, which gives a burnt aroma, were prominent.

However, in BMF from the Halgolla estate, fewer aldehydes and the highest number of terpene and terpene derivatives were detected. Although Halgolla instant tea does not seem to have a pleasant aroma profile, Halgolla BMF seems to be a good source to collect aroma extracts with higher terpene/terpenoid contents.

3.3. Different classes of organic compounds present in the volatile profile of BMF and instant tea

To better understand the composition of the volatile profile of each sample, the detected compounds were categorized into groups, as shown in Table 2.

3.3.1. Terpenes and terpenoids

The major volatiles in tea leaves are mostly derived from terpenoid pathways (Shi et al., 2015). Terpenes generally impart a desirable typical aroma note and contribute floral, fruity, rosy, and floral odors to various tea (Kraujalyte et al., 2016). In the analyzed samples, monoterpenes, diterpenes, sesquiterpenes, and carotenoids were detected. Of those, monoterpenes accounted for a higher percentage in each sample, followed by sesquiterpenes. The breakdown of the distribution of terpenes in the samples is given in Table 3. The highest terpene content was recorded for the Halgolla BMF (HBMF) sample, which had 40.37 % terpenes and terpenoids. In all cases, the terpene content, which is important for the aroma of black tea, was reduced by approximately 10-fold, but the aroma blended instant tea (ABIT) sample managed to retain most of it. These observations indicate that terpenes and terpenoids that escape during the extraction and concentration stages of instant tea manufacturing can be collected and added back to obtain a product with a superior aroma. The most abundant terpenes found in BMF were linalool and linalool oxides. Linalool oxides were detected in instant tea samples as well but in much lower relative percentages. Both of these compounds have been reported to give off a sweet aroma. Limonene, linalool, linalool oxides, and 1H-pyrrole-2-carboxaldehyde have been previously reported in instant black tea by Kraujalyte et al. (2016). Limonene present in HBMF is not present in Halgolla Instant Tea, but it has been carried over to ABIT during the aroma addback process. Another key observation is that geraniol, which has a floral aroma, is present only in the high-grown BMF and is absent in low-grown BMF samples.

3.3.2. Aromatic and nonaromatic alcohols

From the BMF samples, the highest relative peak area percentage for nonaromatic alcohols was detected in the PBMF sample (6.14 %), while that of the ABIT sample was 6.83 %, which is significant since none of the other instant tea samples retained any of the nonaromatic alcohols. However, even the non-aroma blended instant tea samples showed a high relative percentage of aromatic alcohols, with NEIT having the highest percentage (3.90 %). 3-Hexen-1-ol, E-2-hexen-1-ol, 1-octen-3-ol, benzyl alcohol, and phenylethyl alcohol were among the detected alcohols, and benzyl alcohol was detected across all seven samples. Benzyl alcohol was detected in rather high relative percentages in the instant tea samples, and according to Zhang et al. (2019), it imparts a roasted aroma. Schuh and Schieberle (2006) reported that Z-3-hexen-1-ol imparts a green aroma, and it was detected in high relative percentages in all three tested BMF samples. Z-3-hexen-1-ol and E-2-hexen-1-ol have been previously reported in instant black tea by Kraujalyte et al. (2016). However, among the analyzed instant tea samples, it was detected only in the ABIT sample. E-2-hexen-1-ol has been reported to impart a green odor as reported by Nie et al. (2020) and was detected only in the NEBMF sample. Kraujalyte et al., 2016 reported that 1-octen-3-ol exerts a mushroom odor, and due to its very low odor threshold, it is thought to have a significant impact on the overall aroma and was detected in ABIT, NEBMF, and PBMF samples.

3.3.3. Esters

In the tested samples, a large number of complex esters were identified, and all seven samples showed high percentages of esters, with the PBMF sample recording the highest (6.87 %) (Table 2). Butanoic acid and 3-hexenyl ester were detected in both high-grown BMF samples, and butanoic acid and butyl ester were detected in ABIT samples. Wang et al. (2008)

Table 3 Distribution of the terpene/terpenoid content among subclasses in terms of relative peak area percentages.

Compounds (%)	ABIT	NEIT	NEBMF	PIT	PBMF	HIT	HBMF
Terpenes (Total)	**15.76**	**2.31**	**22.89**	**4.28**	**24.42**	**3.24**	**40.37**
Monoterpene	9.34	2.31	15.72	2.01	20.72	3.24	19.56
Diterpene	ND	ND	0.47	ND	ND	ND	0.44
Sesquiterpene	6.22	ND	5.20	2.27	2.21	ND	18.84
Carotenoids	0.20	ND	1.50	ND	1.49	ND	1.53

ABIT – Aroma Blended Instant Tea, NEIT- Nuwara Eliya Instant Tea, NEBMF- Nuwara Eliya BMF, PIT- Pedro Instant Tea, PBMF- Pedro BMF, HIT- Halgolla Instant Tea, HBMF- Halgolla BMF.

reported methyl salicylate as a compound that is important for overall tea aroma formation. It was detected in high relative percentages in all three BMF samples and was detected in ABIT, although none of the other instant tea samples contained methyl salicylate.

3.3.4. Lactones and heterocyclic compounds

The percentage of lactones in the instant tea samples was higher than that in the BMF samples (Table 2). Four lactones that were previously reported were detected in the tested samples. They were dihydroactinidiolide, furfural, γ-caprolactone, and γ-butyrolactone. Butyrolactone was detected in high relative percentages in all four instant tea samples, including ABIT. γ-Caprolactone, which was detected in PIT, was also detected in ABIT. Both of these compounds are furanone compounds previously reported in instant black tea, which are products of the Maillard reaction and Strecker degradation, which could be due to the elevated temperatures involved in instant tea production (Kraujalyte et al., 2016). The NEBMF sample contained dihydroactinidiolide and furfural, which were reported to emit peach-like and caramel-nutty aromas, respectively.

When other heterocyclic compounds were considered, the HBMF sample contained the highest total peak area percentage, although instant tea samples had a much wider variety of heterocyclic compounds. *N*-Ethylsuccinimide is found in black tea as a derivative of the Strecker degradation product of theanine during fermentation, as stated by Kuo et al. (2011). *N*-Ethylsuccinimide and 2-acetylpyrrole have been previously reported by Chung et al. (2015) as components of oolong tea that are produced during the baking stage. Hence, it can be presumed that the presence of these two compounds in the tested instant tea samples is a result of the high temperatures involved in the production of instant tea. These compounds, which give a roasted aroma, could have a strong influence on the overall aroma of instant tea after the evaporation of other pleasant aroma compounds that were initially present in black tea BMF.

3.3.5. Aldehydes and ketones

Out of the seven instant tea and BMF samples analyzed, the highest percentage of aldehydes was found to be in the aroma blended instant tea sample (4.42 %). In all cases, NEIT, PIT, and HIT had lower percentages of aldehydes than the BMF from which they were produced (Table 2). In the high-grown BMF samples, hexanal was present but was absent in the low-grown sample. Heptanal, which Vararu et al. (2016) reported giving a fresh, fatty, green, herbal note, was detected in all three BMF samples. E-2-Z-4-hexadienal has been reported to impart a green and fruity aroma, which was also characteristic of the two high-grown BMF samples (Takeo and Mahanta, 1983; Schuh and Schieberle, 2006). Apart from the above, the cyclic aldehyde benzaldehyde, which imparts a fruity aroma as stated by Takeo and Mahanta (1983), was detected in all three BMF samples and the ABIT sample, while benzene acetaldehyde, which gives off a floral aroma, was detected in all seven samples. Hexanal, benzaldehyde, and benzene acetaldehyde were detected in instant black tea in previous studies, and aldehydes have been reported to have a lower odor threshold and thus contribute significantly to the overall aroma of tea (Kraujalyte et al., 2016). When ketones were considered, the NEBMF sample had the highest percentage of ketones in the volatile profile.

3.4. Enrichment of the aroma profile of instant tea using aroma condensate extracted from BMF

When the non-aroma blended instant tea samples (NEIT, PIT, and HIT) were considered, it was clear that most of the pleasant aroma compounds escaped during the production process. However, as shown in Table 1, the most prominent compounds in the aroma profile of the ABIT sample were similar to those of the BMF samples, indicating the retention of pleasant aroma compounds.

The present study further assessed the influence of aroma condensate addition on the overall acceptability of instant tea powder by using the BMF obtained from Nuwara Eliya, Pedro, and Halgolla, in which BMF is commonly sourced by commercial instant tea manufacturing processes.

The Friedman test conducted for the sensory evaluation study showed that the significantly ($P < 0.05$) highest total acceptance was shown by the tea concentrate samples with an addition of aroma condensate equal to 60 % (score 8.1) and 75 % (score 8.3) of the dry solid content of the tea concentrate. Fig. 2 shows the average scores obtained by the different aroma blended tea concentrates. The Wilcoxon sign rank test indicated that 60 % and 75 % of the samples showed a significant improvement in the perceived aroma over the control (tea concentrate without any aroma condensate addition) ($Z = 2.821$, $P < 0.05$, $r = 0.51$). Considering its economic value in commercial production, 60 % aroma addition was selected compared to using 75 % aroma. Hence, aroma condensate equal to 60 % of the dry solid content of the tea concentrate was added to the tea concentrate from the blended BMF sample to produce the aroma blended instant tea sample, which was used for further analysis.

The ABIT sample, which is the focal point of this study, has retained terpenes, terpenoids and aldehydes, which are important aroma compounds that are present in BMF but are present in much lower relative percentages in the other tested instant tea samples.

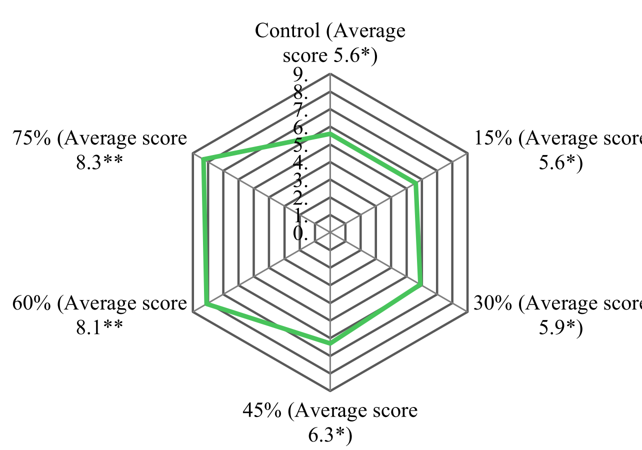

Fig. 2 Radar graph for the average scores of the sensory evaluation study for each aroma blended tea concentrate sample. * and ** indicate means that are significantly different ($p < 0.05$).

Table 4 Aroma compounds detected in the headspace of ABIT in low relative percentages.

Compound category	Compound	Aroma note	Reference
Lactone	Butyrolactone	Creamy	(Guo et al., 2019)
Alcohol	1-Octen-3-ol	Mushroom, forest-earthy odor	(Panten and Surburg, 2015)
Monoterpene hydrocarbon	D-Limonene	Fruity, lemon-like	(Guo et al., 2021)
Alcohol	Benzyl Alcohol	Fruity, Roasted,	(Ito et al., 2002; Zhang et al., 2019)
Aldehyde	Benzeneacetaldehyde	Floral, rose, cherry-like	(Guo et al., 2021)
Pyrrole	1H-Pyrrole-2-carboxaldehyde, 1-ethyl-	Burnt, roasted, smoky	(Guo et al., 2021)
Lactone	γ-Caprolactone	Cereal-like	(Wei et al., 2013)
Pyrrole	2-Acetylpyrrole	Sweet, caramel-like, honeylike, nutty	(Misnawi and Ariza, 2011)
Monoterpene Alcohol	Hotrienol	Fresh, floral, fruity	(Guo et al., 2021)
Alcohol	Phenylethyl Alcohol	Pleasant rose-like smell	(Tsukatani et al., 2003)
Pyrrolidine	*N*-Ethylsuccinimide	Burnt, roasted, smoky	(Dai et al., 2020)
Pyran	2H-Pyran-3-ol, 6-ethenyltetrahydro-2, 2,6-trimethyl-(Epoxylinalol)	Sweet, woody	(Gong et al., 2017)
Monoterpene Alcohol	α-Terpineol	Sweet, floral, lilac	(Khaleel et al., 2018; Zhu et al., 2021)
Ester	Methyl salicylate	Peppermint	(Dai et al., 2020)
Monoterpene Alcohol	Geraniol	Sweet, floral	(Ni et al., 2020)
Phenolic compound	m-Eugenol/Chavibetol	Spicy	(Guo et al., 2021)
Sesquiterpene	γ-Cadinene	Herbal	(Luo et al., 2017)
Carotenoids	β-Ionone	Cedar wood-like	(Plummer et al., 1995)
Lactone	Dihydroactinidiolide	stale odour, sweet	(Dai et al., 2020)
Sesquiterpene	γ-Muurolene	Flower	(Dai et al., 2020)
Sesquiterpene	Copaene	Woody, nuts	(Dai et al., 2020)

Table 4 outlines the other aroma compounds (not reported in Table 1) across different compound categories that were detected in the headspace in lesser percentages. When the aroma note of each compound is considered, there is a wide variety of compounds with pleasant aroma notes as opposed to the few that were present in non-aroma blended instant tea samples (NEIT, PIT, and HIT). The ABIT sample was also found to contain aroma compounds such as *N*-ethylsuccinimide, 2-acetylpyrrole, and benzyl alcohol, which are possibly responsible for the roasted aroma in other instant tea samples, but the presence of compounds such as linalool, geraniol, and hotrienol, which give a flowery aroma to tea, could give ABIT a better aroma in the final product, confirming the improvement in the overall aroma profile that was found during the initial sensory evaluation of tea concentrates with added aroma condensates.

4. Conclusion

Certain aroma compounds, such as hexanal, geraniol, citral, *cis*-jasmon, α-ionone, Nerol, and E-2-Z-4-hexadienal, are characteristic of high-grown BMF, indicating a variation in the aroma profile according to the growing elevation.

The addition of aroma condensate to the concentrated tea extract just before spray drying improved the aroma profile of instant tea, especially when considering the high percentage of terpenes and terpenoids retainsaed in ABIT after the instant tea manufacturing process.

Hence this study confirmed that, aroma blended instant tea can be produced to fulfill the customer preferences of different markets by incorporating condensed aroma extracted from BMF of different elevations. These findings can be applied to produce premium instant tea manufactured from BMF by commercially interested parties in an economically viable manner, since this method helps to add unique aroma characteristics of tea sourced from different origins.

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.

Acknowledgements

The authors wish to extend their sincere gratitude to Hayley's Global Beverages (Pvt) Ltd. financial support for chemical analysis was provided by AHEAD/RA3/ICE/WUSL/FST, Wayamba University of Sri Lanka.

Appendix A. Supplementary material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.arabjc.2022.104147.

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