Characterization of key aroma compounds in two types of Keemun tea

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Abstract

Keemun tea is one of the most popular Chinese black teas, and it is highly appreciated by consumers because of its sweet, floral, and slightly smoky odor. In this study, two types of Keemun tea that differ in terms of raw material and manufacturing process, namely "*gōngfū*" and "*míngyōu*" type Keemun tea, respectively, were investigated by aroma extract dilution analysis (AEDA).

From the AEDA results, 34 odorants with flavor dilution (FD) factors ranging from 64 to 1024 were detected from the volatiles obtained from the isolated fractions of Keemun tea infusions. In particular, geraniol, 4-hydroxy-2,5-dimethyl-3(2H)-furanone, and coumarin exhibited the highest FD factor, which was followed by methional, 2-phenylethanol, phenylacetic acid, and 3-methyl-2,4-nonanedione. These odorants were detected in both Keemun teas. Stable isotope dilution assays (SIDA) were performed, and odor activity values (OAVs) were calculated for the quantitative evaluation of 38 odorants: 27 key odorants with an OAV ≥ 1 were identified. The obtained quantitative data permitted the preparation of aroma recombinates from both types of Keemun tea. Comparative aroma profile analyses between the recombinates and their respective Keemun tea indicated excellent similarity in terms of the overall aroma, thus validating these volatiles as the key components that contribute to the unique odor profile of Keemun tea.

Introduction

Tea (*Camellia sinensis*), which is one of the most popularly consumed beverages in the world, is mainly cultivated in tropical, subtropical, and temperate climates. India, Sri Lanka, and China are the main tea-producing countries. The following two principal varieties are grown in the tea-producing areas: small-leaved Chinese plant (*Camellia sinensis V. sinensis*) and a large-leaved Assamese plant (*Camellia sinensis V. assamica*). Darjeeling and Keemun tea are classified into the former group, while Assamese and Ceylon tea are classified into the latter group. Among Chinese plants, numerous studies have reported on the Darjeeling tea aroma [1]. Although Keemun tea exhibits unique flowery, sweet, and slightly smoky notes [2,3], few studies have reported on the Keemun tea aroma.

Black tea production generally comprises the following four steps: withering, rolling, fermentation, and firing. In particular, several biochemical reactions occur in tea leaves during fermentation. Keemun tea is categorized into two types mainly based on the manufacturing method. One is "*gōngfū*"-type Keemun tea (GK) that is used for exports, while the other is "*míngyōu*"-type Keemun tea (MK) that is used for domestic

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consumption. The rolling process (i.e., rubbing and twisting processes) is different for GK and MK. GK is tightly rolled using a machine, while MK is softly rolled by hand.

This study aimed to clarify the key aroma compounds in Keemun black tea by aroma extract dilution analysis (AEDA) and stable isotope dilution assays (SIDA) as well as to determine whether the difference in the rolling process considerably affects the volatile profiles of tea infusions.

Experimental

First, tea leaves (6 g) were soaked in hot water (95°C, 300 mL). After 5 min, tea leaves were separated by filtration, and the infusion was cooled to 15°C using an ice bath. Second, the beverage (50 mL) was repeatedly extracted with dichloromethane (2 × 50 mL), and the volatile compounds were isolated by solvent-assisted flavor evaporation [1].

GC-O was employed to analyze the aroma extract, and the most important aroma-active compounds were determined by AEDA. After identification (RI on two capillary columns, odor quality, and mass spectra), the aroma compounds with the highest FD factors were quantified by SIDA. Finally, odor activity values (OAVs) of the key odorants were calculated from the concentrations of the aroma compounds and their odor thresholds.

Aroma reconstitution models were prepared by utilizing natural concentrations of the key odorants with an OAV greater than or equal to one dissolved in water. Sensory analysis was performed in a sensory room with single booths. The sensory panel comprised 15–21 trained assessors.

Results and discussion

Screening of aroma-active compounds by AEDA

The elucidation of the aroma-active compounds by AEDA revealed 34 odorants in the two types of Keemun tea with FD factors ranging from 64 to 1024. The highest FD factors in both Keemun tea were observed in case of odorants such as flowery-smelling geraniol and sweet-smelling 4-hydroxy-2,5-dimethyl-3(*2H*)-furanone (FD 1024). However, in GK, oat-like smelling (*E*,*E*,*Z*)-nonatrienal (FD 1024) was followed by the cooked potato-like smelling methional, flowery-smelling 2-phenylethanol, and sweet-smelling coumarin (FD 512). Meanwhile, in MK, coumarin (FD 1024) was followed by hay-like-smelling 3-methyl-2,4-nonanedione (FD 512).

Quantitation of the key odorants by SIDAs

Aroma-active compounds that exhibited high FD factors from AEDA in addition to four compounds (i.e., α-ionone, (*Z*)-4-heptenal, (*E*,*E*)-2,4-decadienal, and (*E*,*Z*)-2,6-nonatrienal, respectively) were quantified. All compounds concentrations were determined by SIDA, and OAVs were calculated on the basis of these concentration and odor thresholds [4] in water revealed 27 key odorants in each Keemun tea (Table 1). Geraniol, 2-phenylethanol, and linalool were determined as the key floral odorants, and 4-hydroxy-2,5-dimethyl-3(*2H*)-furanone, coumarin, and (*E*)-β-damascenone were determined as the key sweet odorants; finally, 4-vinylguaiacol, 4-vinylphenol, and guaiacol were determined as the key smoky odorants in the Keemun tea.

Sensory profile analysis

To validate the results obtained from these investigations, 27 key odorants were recombined in their natural concentrations, and each aroma model was compared to the original Keemun tea infusions by aroma profiling (Figure 1). Both mixtures considerably matched the original Keemun tea infusions in terms of all attributes; these investigations demonstrated that the aroma of the two types of Keemun tea can be simulated by 27 compounds.

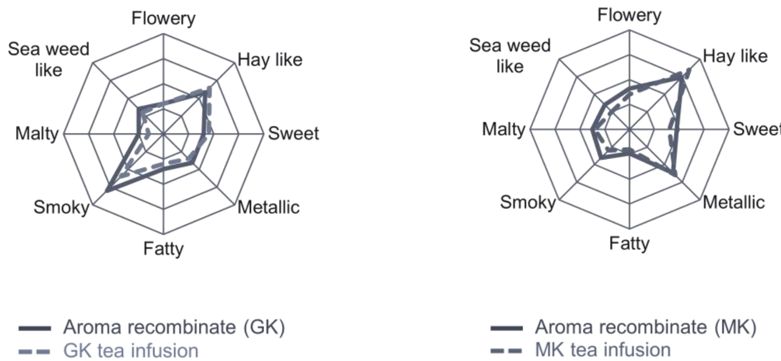

Figure 1: Comparative aroma profiles of aroma recombinate and original Keemun tea infusions

The comparison of the aroma profiles of the original Keemun tea revealed clear differences among metallic, malty, and smoky attributes. Odorants responsible for these attributes were *trans*-4,5-epoxy-(*E*)-2-decenal (metallic), 4-vinylguaiacol (smoky), 2-methylbutanal, and 3-methylbutanal (malty), and their OAVs were clearly different in the two types of Keemun tea (Table 1).

Table 1: FD factors and OAVs of aroma-active compounds (OAV ≥ 1) in Keemun tea (GK and MK)

Odorant	*Odor quality*	*FD factor*		*OAV*	
		GK	*MK*	*GK*	*MK*
geraniol	flowery, fruity	1024	1024	290	530
4-hydroxy-2,5-dimethyl-3(2*H*)-furanone	caramel, sweet	1024	1024	3	5
(*E*,*E*,*Z*)-2,4,6-nonatrienal	oat	1024	64	36	19
methional	cooked potato	512	32	7	7
2-phenylethanol	flowery, honey	512	256	3	4
coumarin	woodruff, sweet	512	1024	1	2
linalool	flowery	256	32	530	610
(*E*)-*β*-damascenone	cooked apple, sweet	256	256	23	25
trans-4,5-epoxy-(*E*)-2-decenal	metallic	256	256	8	15
eugenol	clove	256	32	1	1
3-ethylphenol	phenol	256	256	2	3
4-vinylguaiacol	clove, smoky	256	256	2	<1
3-hydroxy-4,5-dimethyl-2(5*H*)-furanone	celery, seasoning	256	256	2	2
4-vinylphenol	phenol, smoky	256	16	3	2
phenylacetaldehyde	honey, bees wax	128	64	76	54
guaiacol	smoky, sweet	128	256	9	9

Table 1: continued

Odorant	*Odor quality*	*FD factor*		*OAV*	
		GK	*MK*	*GK*	*MK*
3-methyl-2,4-nonanedione	hay, fishy	64	512	63	67
2,3-butanedione	buttery	64	32	11	15
3-methylbutanal	malty	32	64	421	618
2-methylbutanal	malty	32	64	155	220
hexanal	green, grassy	32	64	24	43
2-acetyl-1-pyrroline	roasted, popcorn	16	64	1	2
3-methylindole (skatole)	fecal, mothball	16	64	2	3
1-octen-3-one	mushroom	4	64	5	8
(*Z*)-4-heptenal	fishy, fish oil	4	8	15	10
(*E*,*E*)-2,4-decadienal	fatty, fried	-	-	12	11
(*E*,*Z*)-2,6-nonadienal	cucumber	8	8	8	8

In conclusion, a majority of the key odorants in Table 1 had been previously identified as the major contributors to the aroma of Darjeeling tea [1]; meanwhile, some smoky-smelling compounds such as 4-vinylphenol and guaiacol and sweet-smelling compounds such as coumarin have been reported to be crucial contributors to the aroma of Keemun tea. Our study has revealed the key aroma compounds that can characterize the overall aroma of Keemun tea and the potent aroma compounds that differentiate between the two types of Keemun tea; however, further investigation is necessary to clarify the presence of a high number of odorants that contribute to smoky attributes in the Keemun tea rolled by a machine and a high number of odorants contributing to the metallic and malty attributes in the Keemun tea rolled softly by hand.

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