



Characterization and modelling of odor-active compounds release behavior from Fu-brick tea during boiling-water extraction by molecular sensory science approach

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ABSTRACT

The odor-active compounds in Fu-brick tea and their release behaviors during boiling-water were studied. Fifty-one odor-active compounds were identified, and their release behaviors were identified by continuously recovering 16 sections of condensed water combined with sensory, instrumental, and nonlinear curve fitting analysis. The odor intensities of condensed water and the concentrations of odor-active compounds could be significantly fitted ($p < 0.01$) to power-function type curves. Hydrocarbons showed the fastest release rate, while organic acids showed the slowest. The release rates had very little correlation with their concentrations, molecular weights, and boiling points. Most odor-active compounds ($\geq 70\%$) released need to evaporate more than 24 % of the added water during boiling-water extraction. Meanwhile, on the basis of odor activity value (OAV) calculation, the aroma recombination experiments were performed to explore the odor-active compounds that made major contributions to the formation of the aroma profile of each condensed water.

1. Introduction

Aroma profile of tea is the comprehensive presentation of the combined action of a large number of volatile odor-active compounds (mg/kg level or lower) released into the perceptual space, which is one of the most important factors to measure the flavor quality of tea as well as its deep-processing products (Ma et al., 2022). Flavor quality of tea was notably identified as one of the main factors that directly determined the market value and significantly influenced consumers' perception and acceptance (Deng et al., 2021). The individual volatile odor-active compounds differ according to their molecular structure, which leads to differences in the solubility, boiling point, and the volatility between volatile compounds (Herrera-Jiménez, Escalona-Buendía, Ponce-Alquicira, Verde-Calvo, & Guerrero-Legarreta, 2007). These differences may directly affect the release behavior of volatile odor-active compounds and aroma profile of tea. In depth study of the release

behavior of tea volatile odor-active compounds will not only help to clarify the formation mechanism of the aroma profile of tea, but also help to recover the volatile odor-active compounds that released during the processing of instant tea and improve the flavor quality of instant tea according to their release behavior.

Fu-brick tea, a unique microbial post-fermented tea, is mainly produced in Hunan, Shanxi, and Sichuan provinces of China (Li et al., 2020; Nie et al., 2019). The steaming, piling, pressing, fungal fermentation, and drying are the key processes in the production of Fu-brick tea (Ma et al., 2021). Among them, fungal fermentation process was considered to be the key process to form the quality of Fu-brick tea (Xu, Mo, Yan, & Zhu, 2007; Zhang, Zhang, Chambers IV, & Dai, 2020). This process produces large amounts of probiotics called *Eurotium cristatum*, commonly known as 'golden flower', which give Fu-brick tea a strong taste with unique 'fungal/flower' floral and health benefits according to previous studies (anti-obesity, anti-hyperlipidemia, antioxidant, anti-

Abbreviations: GC–MS, Gas chromatography–mass spectrometry; GC–O, Gas chromatography–olfactometry; OAV, Odor activity value; DSA, Descriptive sensory analysis; CW, Condensed water; EC, Exponential coefficients; CC, Correlation coefficient.

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tumor, anti-microbial, and others) (Cao et al., 2018; Fu et al., 2011; Li, Luo, Wang, Fu, & Zeng, 2019; Mo, Zhang, Li, & Zhu 2008; Zhang, Zhang, Zhou, Liang, & Wan, 2013). The odor-active compounds are the material basis for the formation of the unique fragrance of Fu-brick tea. In our previous study, we found that decanal, nonanal, heptanal, (*E,E*)-2,4-nonadienal, benzeneacetaldehyde, benzaldehyde, naphthalene, 1-methylnaphthalene, 2-methylnaphthalene, (*E,E*)-3,5-octadien-2-one, 2-pentylfuran, 4-methylanisole, and dihydroactinolide were related to the formation of 'green' attribute; dehydro- β -ionone, dihydro- β -ionone, (+)-carotol, and linalool oxide II were related to the formation of 'floral and fruity' attribute; α -terpineol was related to the formation of 'woody' and 'stale' attributes (Zheng et al., 2022). However, a considerable amount of them were released and lost during the production of instant Fu-brick tea by extraction and concentration. How to effectively recover odor-active compounds to improve the flavor quality of instant Fu-brick tea, the first task is to clarify the release behaviors of odor-active compounds in the processing, which has a negative impact to the flavor quality of instant Fu-brick tea.

To investigate the release behavior of volatile odor-active compounds in the process of the extraction or concentration of foods, the food industries often used condensation procedures to recover volatile odor-active compounds evaporated with water (Dawiec-Liśniewska, Szumny, Podstawczyk, & Witek-Krowiak, 2018). And then, the release behaviors of odor-active compounds and the sensory quality of the released volatile odor-active compounds were determined by using quantitative and descriptive sensory analysis (DSA), so as to guide the recovery of condensed water (rice-in odor-active compounds). Finally, the condensed water was concentrated and added back to sample to restore the natural flavor of the deep processed products (Sampaio, Biasoto, Marques, Batista, & Silva, 2013). At present, the study on evaluating the release behavior of odor-active compounds by recovering condensed water and then improving the flavor quality of final products was mainly concentrated in the fruit juices processing (Sahin, 2013). There are few studies related to the release behavior of volatile odor-active compounds in the deep processing of tea, which is worth of in-depth study.

The objectives of this study are the following: (1) to identify the odor-active compounds in Fu-brick tea; (2) to evaluate the sensory quality of condensed water recovered in different stages; (3) to model the release behavior of odor-active compounds of Fu-brick tea during boiling-water extraction; (4) to determine the composition of the key odor-active compounds in each section of condensed water.

2. Materials and methods

2.1. Materials

Six representative Fu-brick tea samples (processed in 2015–2020) were selected according to the "Methodology for Sensory Evaluation of Tea (Chinese Standards, GB/T 23776–2018)". The selected samples were ground and passed through 30 mesh before experiments. The experiment sample was prepared by mixing the tea power of the six selected samples with equal mass.

2.2. Reagents and chemicals

Dichloromethane (PubChem CID: 6344; $\geq 99.5\%$), *n*-butanol (PubChem CID: 263; $\geq 99.9\%$), anhydrous sodium sulfate (PubChem CID: 24436; $\geq 99.0\%$), (*E,E*)-2,4-nonadienal (PubChem CID: 5283339; $> 90\%$), geraniol (PubChem CID: 637566; $\geq 99\%$), 2-heptanone (PubChem CID: 8051; 98%), hexanoic acid (PubChem CID: 8892; $\geq 99.5\%$), *D*-limonene (PubChem CID: 440917; $\geq 99\%$), benzyl alcohol (PubChem CID: 244; $\geq 99.5\%$), acetophenone (PubChem CID: 7410; $\geq 99.5\%$), 6-methyl-3,5-heptadiene-2-one (PubChem CID: 5370101; 98%), phenylethyl alcohol (PubChem CID: 6054; $\geq 99.5\%$), (*E*)-2-nonenal (PubChem CID: 5283335; 95%), 4-*tert*-butylphenol (PubChem CID: 7393; $\geq 99.5\%$),

terpinen-4-ol (PubChem CID: 11230; 98%), octanoic acid (PubChem CID: 379; $\geq 99.5\%$), nonanoic acid (PubChem CID: 8158; $> 99\%$), geranial (PubChem CID: 638011; 98%), γ -nonalactone (PubChem CID: 7710; 98%), 2,4-di-*tert*-butylphenol (PubChem CID: 7311; 97%), nerolidol (PubChem CID: 5284507; 97%), cedrol (PubChem CID: 65575; 98%), and *n*-hexadecanoic acid (PubChem CID: 985; $\geq 99\%$) were purchased from Macklin (Shanghai, China). β -Linalool (PubChem CID: 6549; 98%), α -terpineol (PubChem CID: 17100; $> 95\%$), linalool oxide III (PubChem CID: 26396; $\geq 99\%$), β -ionone (PubChem CID: 638014; 97%), hexanal (PubChem CID: 6184; $\geq 99\%$), heptanal (PubChem CID: 8130; $\geq 98\%$), benzaldehyde (PubChem CID: 240; $\geq 98\%$), 6-methyl-5-hepten-2-one (PubChem CID: 9862; 98%), (*E,E*)-2,4-heptadienal (PubChem CID: 5283321; 90%), octanal (PubChem CID: 454; 99%), isophorone (PubChem CID: 6544; $> 97\%$), and methyl salicylate (PubChem CID: 4133; $\geq 99.5\%$) were purchased from Aladdin (Shanghai, China). Dihydro- β -ionone (PubChem CID: 519382; $\geq 90\%$) and safranal (PubChem CID: 61041; 90%) were purchased from Bidepharm (Shanghai, China). 2-Ethyl-1-hexanol (PubChem CID: 7720; $\geq 99\%$), linalool oxide I (PubChem CID: 22310; 99%), linalool oxide II (PubChem CID: 6432254; 96%), citronellal (PubChem CID: 7794; 97%), β -cyclocitral (PubChem CID: 9895; 95%), 1,2,3-trimethoxybenzene (PubChem CID: 12462; 98%), α -ionone (PubChem CID: 638014; $\geq 90\%$), dihydroactinidiolide (PubChem CID: 27209; 98%), 3-octen-2-one (PubChem CID: 5363229; 97%), (*Z*)-geranyl acetone (PubChem CID: 1713001; 99%), and *n*-alkanes solution (C₈–C₃₂) were purchased from J&K Chemical (Beijing, China). Benzeneacetaldehyde (PubChem CID: 998; $\geq 98\%$) and 2,5-octanedione (PubChem CID: 6420399; $\geq 95\%$) were purchased from Acmechem (Shanghai, China). (*E*)-2-Octenal (PubChem CID: 5283324; $\geq 96\%$) was purchased from TCI (Shanghai, China). (*E,E*)-3,5-Octadien-2-one (PubChem CID: 5352876; 98%) and 1,2-dimethoxybenzene (PubChem CID: 7043; $> 99\%$) were purchased from Sigma-Aldrich.

2.3. Volatile compounds of Fu-brick tea extracted by HS-SPME

The volatile compounds of Fu-brick tea were extracted according to our previous study with slight modification (Li et al., 2020). The detailed process are as follows: 1.00 g tea, 0.5 g sodium chloride, and 5.0 mL boiling ultra-pure water were introduced into a 20 mL vial, and then the vial was tightly sealed with silicone septa (Sigma Chemical Co., Ltd). The vial was placed into a water bath with a constant temperature ($80\text{ }^{\circ}\text{C}$) and kept for 15 min (sample equilibrium before extraction). A $65\text{ }\mu\text{m}$ polydimethylsiloxane/divinylbenzene (PDMS/DVB; Catalogue Number: 57310-U) fiber was fixed on a manual holder for HS-SPME analysis. After sample equilibrium, the fiber was inserted into the vial and exposed to the headspace of sample vial for 45 min, and then the fiber was immediately inserted into the gas chromatography (GC) injector and maintained it at $250\text{ }^{\circ}\text{C}$ for 5 min.

2.4. Evaluation of the release behavior of odor-active compounds

The release behaviors of odor-active compounds of Fu-brick tea were evaluated according to our previous study (Wang, He, Zhang, Du, Xiao, & Xu, 2020). Five kilograms of Fu-brick tea and 25 L of purified water (about $80\text{ }^{\circ}\text{C}$) were added into a 50 L multi-functional extractor equipped with a condensed water recovery vessel (DC-NSG-50, Shanghai Dacheng Laboratory Equipment Co., Ltd, Shanghai, China). In the process of boiling extraction of tea, the water vapor (rich-in volatile odor-active compounds) released from extractor was condensed by a stainless steel cooler, and the condensed water was recycled in sections (500 mL for each section) until the entire odor intensity was very weak. All the condensed water samples recovered in sections were stored in the refrigerator ($-18\text{ }^{\circ}\text{C}$) for standby. The release behaviors of total odor-active compounds and single odor-active compound based on their concentrations in recovered condensed water.

2.5. Descriptive sensory analysis of condensed water

To evaluate the flavor quality of each condensed water, six assessors (four females and two males) with more than five years experiences in the flavor evaluation of tea were selected for descriptive sensory analysis. Before experiments, all assessors selected and approved the 'woody', 'stale', 'green', 'herbal', 'floral/fruity', and 'off-flavor' attributes through detailed discussion and our previous sensory analysis (the relationship between aroma-active compounds and sensory attributes in Fu-brick tea) on Fu-brick tea (Zheng et al., 2022). The evaluation experiment referred to the method of our previous study (Wang, He, Zhang, Du, Xiao, & Xu, 2020). The condensed water (50 mL) was added into a tasting glass and coded with a three-digit number. And then, the tasting glasses were randomly assigned to each assessor in the sensory evaluation room. The intensities ('1' indicated 'weak', '5' indicated 'moderate', and '9' indicated 'strong') of entire aroma and odor attributes ('woody', 'stale', 'green', 'herbal', 'floral/fruity', and 'off-flavor') were evaluated and recorded, and the final presentation of intensity was the average value recorded by six assessors. In order to explain the relationship between the number of recoveries (x) and the odor intensity of the corresponding condensed water (y), the sensory data were subjected to nonlinear curve fitting analysis according to the previous researches (Sampaio, Biasoto, Marques, Batista, & Silva, 2013; Wang, He, Zhang, Du, Xiao, & Xu, 2020).

2.6. GC-O analysis

A Thermo Trace 1300 GC equipped with an ODP-3 sniffing port (Gerstel, Inc., Germany) was used to perform GC-O analysis. RTX-5MS column (30 m \times 0.25 mm internal diameter, 0.25 μ m film thickness; Restek, Bellefonte, PA) and flame ionization detector (FID, 280 $^{\circ}$ C) were used for the separation and detection of effluents, respectively. A 'Y' shaped splitter was installed at the end of the GC column and used to divide the effluents equally between FID detector and olfactometry. High purity nitrogen (greater than 99.99 %) was used as carrier gas with a flow rate of 1 mL/min. The injector temperature was set to 250 $^{\circ}$ C. The temperature program of GC was as follows: the initial temperature was set to 50 $^{\circ}$ C and maintained for 2 min, increased to 160 $^{\circ}$ C at 2 $^{\circ}$ C/min, and then increased to 250 $^{\circ}$ C at 10 $^{\circ}$ C/min, and maintained for 2 min. The Humidified air flowing and transfer line temperature of olfactometry were set to 60 mL/min and 250 $^{\circ}$ C, respectively.

Taking 'GB/T 16291.1-2012 (Sensory analysis-General guidance for the selection, training, and monitoring of assessors)' as the criterion, six assessors (four females and two males) with more than five years experiences in the flavor evaluation of tea were selected and trained for GC-O analysis. Ten commercial standards (hexanal, *D*-limonene, linalool oxide II, β -linalool, methyl salicylate, 1,2,3-trimethoxybenzene, (*Z*)-geranylacetone, β -ionone, dihydroactinidiolide, and dihydro- β -ionone) were selected to train six assessors for more than 25 h, until all assessors were very familiar with the odor description and odor intensity ('1', '5', and '9' represented 'weak', 'moderate', and 'strong' odor intensities, respectively) of each selected compound at different concentrations. During GC-O analysis, all assessors were required to record each captured odor and its intensity, and the final presentation of intensity of each odor-active compound was the average value recorded by six assessors.

2.7. GC-MS analysis

Identification of odor-active compounds was performed by using Thermo Trace 1300 GC equipped with ISQ MS under the same parameters (injector temperature, capillary column, oven temperature program, and flow rate of carrier gas [helium, >99.999 %]) as GC-O analysis. Other parameters of GC-MS, including solvent delay time, ion source temperature, interface temperature, mass scan range, and EI energy were set to 3.0 min, 150 $^{\circ}$ C, 230 $^{\circ}$ C, 280 $^{\circ}$ C, 35-450 aum, and 70

eV, respectively. After GC-MS analysis, the collected mass spectra were compared with the commercial database (NIST17), and then the retention indices, commercial standards, and odor characteristics were also used to verify the qualitative results.

2.8. Quantitation of odor-active compounds in each condensed water

The odor-active compounds in the condensed water that collected in sections were extracted and quantified according to our previous study with slight modification (Wang, He, Zhang, Du, Xiao, & Xu, 2020). The detailed extraction process: 5 mL condensed water and 100 μ L internal standard (*n*-decanol, 66.5 μ g/mL) were added into a 10 mL separatory funnel and mixed evenly, and then 2 mL dichloromethane (CH_2Cl_2) was added to extract the odor-active compounds. After static stratification, the CH_2Cl_2 was taken out, and then anhydrous sodium sulfate was added and maintained at 4 $^{\circ}$ C for 24 h to remove the water. Subsequently, the extraction samples were analyzed by GC-MS. The quantification of odor-active compounds by using commercial standards with five different concentrations. In preparation, the commercial standards were dissolved in methanol, and diluted to 5 mL with ultra-pure water. And then, the extraction samples were prepared by using the extraction method of odor-active compounds in condensed water. After GC-MS analysis, the standard curves were established by using the known concentrations of commercial standards and their corresponding detection peak areas ([peak area of odor-active compound]/[peak area of internal standard] = slope[concentration of odor-active compound]/[concentration of internal standard] + intercept). Due to the lack of commercial standards for 3,5-octadien-2-one and grandlure II, these two odor-active compounds were quantified by using the calibration curves of (*E, E*)-3,5-octadien-2-one and α -terpineol, respectively.

2.9. Key odor-active compounds in each condensed water

Odor activity value (OAV, concentration of odor-active compound/odor threshold) was used to evaluate the contribution of each odor-active compound. The OAV of each odor-active compound was calculated by using its concentration in condensed water and its odor threshold. The odor-active compounds with OAVs ≥ 1 were considered to be the main contributors to the aroma profile of the analyzed samples. The odor thresholds of these odor-active compounds were obtained from reported studies, books, and "<https://www.vcf-online.nl/VcfHome.cfm>", and the detailed source information was provided in the "supplementary material".

To investigate the qualitative and quantitative accuracy of key odor-active compounds, the aroma recombination test was performed for further verification. The odor-active compounds with OAVs ≥ 1 were added into ultra-pure water according to their concentrations detected in condensed water and mixed evenly. And then, the recombination samples were sealed and kept for 24 h at 4 $^{\circ}$ C before sensory analysis. The higher the sensory results similarity between the recombination sample and the corresponding condensed water, the higher the qualitative and quantitative accuracy of key odor-active compounds.

2.10. Statistical analysis

The detection data was pre-treatment by using MS Excel 2010 (Microsoft Corp., Redmond, WA, USA). The nonlinear curve fitting analysis was performed by using OriginPro software (version 9.5.1, OriginLab Inc., USA).

3. Results and discussion

3.1. Odor-active compounds in Fu-brick tea

Fifty-one odor-active compounds were captured and identified in Fu-brick tea by GC-O and GC-MS with odor intensities greater than 1,

including 13 alcohols, 13 ketones, 13 aldehydes, 4 organic acids, 2 methoxy-phenolic compounds, 1 ester, 2 lactones, 2 phenols, and 1 hydrocarbon (Table S1). Among them, the concentration of (*Z*)-geranyl acetone was the highest, at 37.30 mg/kg, followed by β -ionone (35.62 mg/kg), methyl salicylate (26.79 mg/kg), α -ionone (19.39 mg/kg), linalool oxide II (11.05 mg/kg), terpinen-4-ol (9.81 mg/kg), *n*-hexadecanoic acid (9.74 mg/kg), 2,5-octanedione (9.14 mg/kg), nerolidol (8.45 mg/kg), 3-octen-2-one (8.11 mg/kg), β -cyclocitral (7.53 mg/kg), linalool oxide I (7.44 mg/kg), 2,4-di-*tert*-butylphenol (7.44 mg/kg), 1,2,3-trimethoxybenzene (7.29 mg/kg), *D*-limonene (6.70 mg/kg), cedrol (6.37 mg/kg), grandlure II (6.23 mg/kg), dihydroactinidiolide (6.04 mg/kg), β -linalool (5.48 mg/kg), geraniol (5.29 mg/kg), phenylethyl alcohol (5.11 mg/kg), and 6-methyl-5-hepten-2-one (5.10 mg/kg). The concentrations of these odor-active compounds are basically consistent with previous reports (Lv, Zhang, Shi, & Lin, 2017; Nie et al., 2019; Zheng et al., 2022). Among them, (*Z*)-geranyl acetone was described as smelling like ‘rose’ and ‘green’ odors in Fu-brick tea, which was also detected in black and ripened Pu-erh teas and was considered to play an important role in the formation of their characteristics flavor (Liu et al., 2022; Xu et al., 2016). α -Ionone and β -ionone were derived from α -carotene and β -carotene, respectively, which were identified to play a vital role in the formation of the ‘woody’ attribute of many teas due to their extremely low odor threshold (0.6 μ g/kg, 0.007 μ g/kg) (Wang et al., 2022a). Methyl salicylate was detected to present ‘wintergreen’ and ‘mint’ odors, which was identified to be not only the key odor-active compound of Fu-brick tea, but also the key odor-active compound of Pu-erh tea, Liupao tea, and Qing-brick tea (Ma et al., 2021). Linalool oxide I and linalool oxide II were described as smelling like ‘floral’ odor, which were identified as the important contributors to the ‘fungal flower’ aroma formation of Fu-brick tea (Zheng et al., 2022). Terpinen-4-ol was identified to provide ‘pepper’, ‘woody’, and ‘earthy’ odors, which was also detected as one of the odor-active compounds in Pu-erh tea (Wang et al., 2022a). *n*-Hexadecanoic acid was identified to provide ‘rancid’ and ‘pungent’ odors, which was the most frequently detected acid compound in Fu-brick tea (Xu, Mo, Yan, & Zhu, 2007). 2,5-Octanedione was identified to provide ‘green’, ‘sweet’, and ‘buttery’ odors, which was also detected as one of the odor-active compounds in Pu-erh tea (Wang et al., 2022a). Nerolidol was described as smelling like ‘rose’ odor, which was identified as the main contributors to the ‘floral’ attribute of Fu-brick tea (Xu, Mo, Yan, & Zhu, 2007). 3-Octen-2-one was detected to present ‘earthy-like’ odor, which was identified as an odor-active compounds with relatively low OAV in Fu-brick teas (Li et al., 2020). The reported results were inconsistent with our research, which might be due to the difference of Fu-brick tea samples (Zheng et al., 2022). β -Cyclocitral, a key odor-active compound for the formation of ‘fungal flower’ of Fu-brick tea, which could produce by the degradation of β -carotene (Li et al., 2020). 2,4-Di-*tert*-butylphenol was described as ‘phenolic’ and ‘herbal’ odors in Fu-brick tea, which was also detected as an odor-active compound in other Dark teas (Li et al., 2020; Ma et al., 2021). 1,2,3-Trimethoxybenzene was detected to present ‘stale’ odor, which was regarded as the most important contributor to the ‘stale’ attribute of Fu-brick tea and Pu-erh tea (Li et al., 2020; Wang et al., 2022b). It was considered to be formed by the microbial degradation and methylation of tea catechins as well as thermal degradation during the post-fermentation process (Pang et al., 2019). *D*-Limonene was described as smelling like ‘lemon’ odor, which was identified as the main contributor to the ‘fruity’ attribute of Fu-brick tea (Li et al., 2020). Cedrol was identified to provide ‘woody’ odor, which was considered as one of the key aroma compounds in the formation of the unique flavor of Fu-brick tea (Zheng et al., 2022). Grandlure II was identified to provide ‘fruity’ odor, which was first detected in Fu-brick tea. Dihydroactinidiolide was described as smelling ‘woody’, which was identified to be derived from the degradation of β -carotene based on microbial activity or thermal degradation during fermentation processing of Fu-brick tea (Xu, Mo, Yan, & Zhu, 2007). β -Linalool was identified to provide pleasant ‘floral’ and ‘citrus-like’ scents, which underwent

significant oxygenation during the fermentation of Fu-brick tea (Zheng et al., 2022). Geraniol was described as smelling like ‘rose’ odor, which was identified as the key contributor to the floral attribute of Fu-brick tea due to its relatively low odor threshold (10 μ g/kg) (Du, Plotto, Baldwin, & Rouseff, 2011). Phenylethyl alcohol was described as smelling like ‘rose’, ‘floral’, and ‘honey’ odors, which was identified to be close to green floral in Sichuan Dark brick tea (Nie et al., 2019). 6-Methyl-5-hepten-2-one was identified to provide ‘pepper’, ‘mushroom’, and ‘rubber’ odors, which was detected to have a higher odor activity value (OAV) in Sichuan Fuzhuan brick tea than Sichuan Dark brick tea (Nie et al., 2019).

3.2. Sensory assisted dynamics recovery of odor-active compounds during extraction

The odor-active compounds were dynamically recovered during boiling-water extraction of Fu-brick tea. Sixteen sections condensed water were recovered, and all the condensed water samples were evaluated by assessors according to the sensory evaluation procedure. The relationship between the odor intensity of each condensed water recovery times is shown in Fig. 1. The odor intensities of condensed water that recovered in the first and second times showed ‘strong’ on the sensory scales, and decreased with the increasing of recovery times. The odor intensity of condensed water reached to ‘moderate’ in the sensory scale at the sixth times. Since the entire odor of condensed water came from the odor-active compounds released from Fu-brick tea, the sensory results suggest that most odor-active compounds that contribute to the unique aroma of Fu-brick tea could be released in the early stage of extraction. Similar results appeared in the study of the release behavior of odor-active compounds in Pu-erh tea and fruit juices (Sampaio, Biaisoto, Marques, Batista, & Silva, 2013; Wang, He, Zhang, Du, Xiao, & Xu, 2020). When the data in Fig. 1 were subjected to nonlinear curve fitting analysis, it was found that these data could be significantly fitted ($p < 0.01$) to a power function type curve. Which is, in fact, the model that best explains the relationship between chemical stimuli and sensory perception.

The intensities of aroma attributes of the collected condensed water were perceived by assessors, and the results were shown in Fig. 2. Among them, the ‘woody’, ‘green’, and ‘herbal’ scents showed a trend of increasing first and then decreasing. This phenomenon of the first

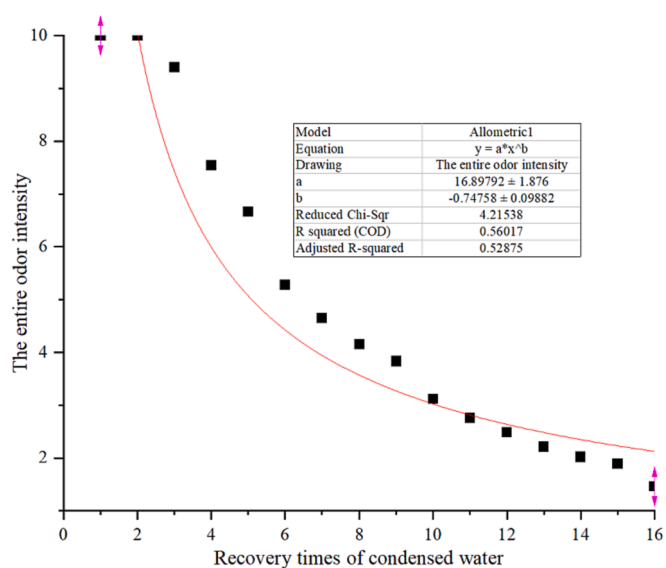


Fig. 1. The water piecewise recovered in the condensates during the extraction process of Fu-brick tea versus the intensity of the characteristic Fu-brick tea aroma presents in each condensed water. (The intensity scale: 1 = weak; 5 = moderate; 9 = strong. y = aroma intensity, x = recovery times).

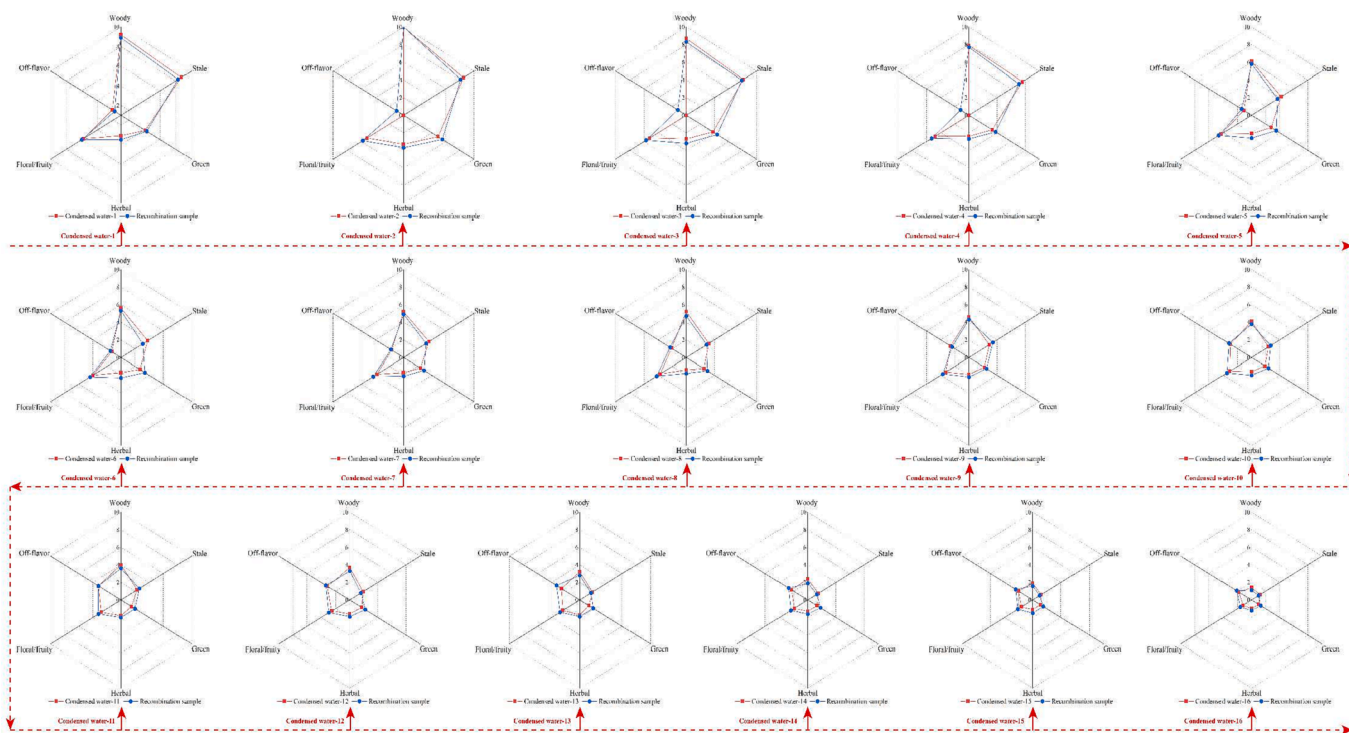


Fig. 2. The change trend of odor attributes in each condensed water.

increase of odor intensities might be due to the fact that the tea was not fully soaked at the beginning of extraction, resulting in a slight lower release rate of odor-active compounds. With the tea fully soaked, the release rate of odor-active compounds increased, their concentrations recovered for the second time were relatively high, and the intensities of aroma attributes were also increased. The 'stale' and 'floral/fruity' scents showed a decreasing trend, the intensity of 'stale' scent began to decrease slowly with the increase of condensed water recovery times, while the odor intensity of the fifth recovered condensed water decreased sharply. This phenomenon might be due to the fact the intensity of 'stale' was not proportional to the concentrations of corresponding odor-active compounds when these compounds exceed certain concentrations. However, when these compounds were lower than a certain concentration, the intensity of 'stale' scent changed greatly with the decrease of their concentrations. Similar results have been reported on the odor-active compounds recovery of Pu-erh tea during the boiling-water extraction (Wang, He, Zhang, Du, Xiao, & Xu, 2020). The 'off-flavor' scent decreased first and then increased with the increase of condensed water recovery times. The 'off-flavor' scent of the condensed water recovered for the first time came the off-flavor components carried by Fu-brick tea. With the increase of recovery times, the 'off-flavor' scent was mostly caused by the breaking of the interaction between odor-active compounds. Similar phenomenon was also found in the recovery of odor-active compounds Pu-erh tea during the boiling-water extraction (Wang, He, Zhang, Du, Xiao, & Xu, 2020).

3.3. Odor-active compounds in the recovered condensed water

Liquid-liquid extraction combined with GC-MS was used to identify and conduct quantify the odor-active compounds in the recovered condensed water. A total of 51, 49, 47, 47, 42, 38, 38, 37, 36, 35, 31, 30, 29, 23, 20, and 14 odor-active compounds were identified in condensed water1 (CW1), condensed water2 (CW2),, and condensed water16 (CW16), respectively (Table 1). Terpinen-4-ol (81.06 mg/L), 3-octen-2-one (65.25 mg/L), *D*-limonene (47.27 mg/L), 6-methyl-5-hepten-2-one (29.75 mg/L), 2-ethyl-1-hexanol (29.26 mg/L), methyl salicylate (27.85 mg/L), 1,2-dimethoxybenzene (26.02 mg/L), (*E,E*-

2,4-nonadienal (25.54 mg/L), (*Z*)-geranyl acetone (24.95 mg/L), dihydro- β -ionone (20.48 mg/L), and linalool oxide II (20.38 mg/L) have relatively high concentrations in CW1. While methyl salicylate (80.72 mg/L), (*Z*)-geranyl acetone (69.29 mg/L), β -ionone (59.46 mg/L), α -ionone (42.78 mg/L), β -cyclocitral (24.46 mg/L), grandlure II (23.84 mg/L), 2,5-octanedione (23.18 mg/L), linalool oxide II (23.09 mg/L), 4-*tert*-butylphenol (22.44 mg/L), and 1,2,3-trimethoxybenzene (18.44 mg/L) have relatively high concentrations in CW2. Compared with CW1, the concentrations of most odor-active compounds increased significantly in CW2. With the increase of recovery times, the concentrations of most odor-active compounds decreased in varying degrees, and the number of odor-active compounds also showed a decreasing trend in condensed water. The change trends of these odor-active compounds were consistent with the results of sensory evaluation.

To evaluate the release behavior of those odor-active compounds from Fu-brick tea, the nonlinear curve fitting analysis was performed by using the recovery times of condensed water (x) and the corresponding concentrations of odor-active compounds (y) in each condensed water. The results showed that the experimental data of alcohols, ketones, hydrocarbons, methoxy-phenolic compounds, esters, organic acids, lactones, phenols, and aldehydes could be significantly fitted power-function kinetic curves ($p < 0.05$) (Fig. 3). Which is, in fact, the models that best explain the relationship between recovery times and the concentrations of odor-active compounds. Considering the exponential coefficients (EC) of the fitted models, the release rate of hydrocarbons (EC = -3.405) was the fastest, followed by methoxy-phenolic compounds (EC = -1.110), aldehydes (EC = -0.992), alcohols (EC = -0.955), esters (EC = -0.728), phenols (EC = -0.705), and ketones (EC = -0.700). The release rates of lactones (EC = -0.367) and organic acids (EC = -0.329) were the slowest. Similar results were found in the study on the release behavior of odor-active compounds of Pu-erh tea that the release rates of alcohols and aldehydes were faster than esters and ketones (Wang, He, Zhang, Du, Xiao, & Xu, 2020). The release rate of methoxy-phenolic compounds in Fu-brick tea was relatively fast, but it had been proved that the release rate in Pu-erh tea was the slowest. This phenomenon might be caused by the different matrix of Fu-brick tea and Pu-erh tea.

Table 1
Odor-active compounds detected in each condensed water.

Time (min)	RI	Odor-active compounds	Concentrations of odor-active compounds in each condensed water (mg/L)															
			CD-1	CD-2	CD-3	CD-4	CD-5	CD-6	CD-7	CD-8	CD-9	CD-10	CD-11	CD-12	CD-13	CD-14	CD-15	CD-16
4.12	800	Hexanal	12.19	9.50	3.27	2.45	1.73	1.57	1.22	1.07	0.86	0.81	0.74	0.69	0.54	0.48	0.33	
4.85	887	2-Heptanone	0.53	1.46	0.16	0.16	0.10											
5.10	905	Heptanal	0.34	5.80	1.75	1.32	0.57	0.53	0.43	0.36	0.35	0.28	0.19	0.15	0.15	0.11	0.09	
6.78	961	Benzaldehyde	1.40	0.76	0.62	0.25	0.24	0.23	0.21	0.19	0.17	0.16	0.13	0.09	0.09	0.13	0.10	
7.22	975	Hexanoic acid	0.10	0.35	1.02	1.26	1.42	1.50	1.77	2.43	2.66	2.89	3.07	1.88	1.82	1.77	0.83	0.26
7.66	990	6-Methyl-5-hepten-2-one	29.75	5.96	3.11	0.57	0.55	0.44	0.29	0.24	0.19	0.15						
8.01	1001	(<i>E,E</i>)-2,4-Heptadienal	5.44	8.29	8.22	1.13	1.13	1.03	0.92	0.70	0.69	0.52						
8.25	1006	Octanal	5.34	8.36	2.54	1.42	0.17											
8.82	1019	<i>D</i> -Limonene	47.27	4.41	1.27	0.51												
9.26	1029	2-Ethyl-1-hexanol	29.26	3.74	0.77	0.23												
9.45	1033	Benzyl alcohol	0.82	0.67	0.24	0.26	0.23	0.22	0.20	0.18	0.17	0.14						
9.72	1039	3-Octen-2-one	65.25	3.49	1.95	1.28												
9.91	1043	Benzeneacetaldehyde	0.98	1.88	0.94	0.10	0.63	0.25	0.23	0.13								
10.55	1057	(<i>E</i>)-2-Octenal	11.11	8.60	2.96	2.10												
10.92	1065	Acetophenone	0.79	1.08	0.89	0.76	0.39	0.38	0.28	0.23	0.25	0.18	0.10	0.11	0.07			
11.15	1070	3,5-Octadien-2-one	11.20	11.21	4.11	2.67	0.49											
11.25	1072	Linalool oxide I	14.10	15.45	8.68	6.77	3.09	2.72	2.58	2.23	2.15	1.93	2.01	1.69	1.05	0.89	0.46	0.09
12.01	1089	Linalool oxide II	20.38	23.09	13.89	11.28	4.31	3.92	3.66	3.44	2.85	2.82	2.60	2.17	1.30	0.97	0.54	
12.24	1095	(<i>E,E</i>)-3,5-Octadien-2-one	8.30	10.50	5.71	4.28	0.89	0.80	0.73	0.70	0.65	0.54	0.43	0.32	0.14	0.05		
12.59	1102	β -Linalool	9.50	10.26	4.64	4.17	3.20	3.01	2.26	2.10	1.71	1.51	1.37	0.82	0.92	0.96	0.68	0.29
12.83	1106	6-Methyl-3,5-heptadiene-2-one	7.39	9.14	5.84	4.69	2.25	2.04	1.95	1.79	1.60	1.29	1.12	1.08	0.81			
13.19	1112	Phenylethyl alcohol	3.03	5.17	4.65	4.50	4.25	4.10	3.46	2.93	2.38	2.33	2.26	2.13	1.21	1.08	0.58	
13.57	1118	Isophorone	0.54	1.27	0.91	0.81	0.64	0.61	0.34	0.40	0.19	0.14	0.09					
14.67	1137	1,2-Dimethoxybenzene	26.02	8.33	3.51	1.83												
15.75	1155	(<i>E</i>)-2-Nonenal	4.49	4.16	2.55	1.49	0.42											
16.15	1162	Citronellal	2.55	4.36	2.95	2.62	2.52	2.27	1.59	1.31	0.70	0.71	0.56	0.51	0.12			
16.25	1164	linalool oxide III	1.65	2.14	1.65	1.52	1.58	1.46	1.11	0.94	0.83	0.75	0.70	0.64	0.42	0.35	0.20	0.12
16.54	1169	4-tert-Butylphenol	18.78	22.44														
16.68	1171	Terpinen-4-ol	81.06															
16.75	1172	Octanoic acid	1.30	2.39	2.47	2.33	2.23	1.63	1.41	0.92	1.06	1.04	0.81	0.75	0.53	0.41	0.16	0.13
17.46	1185	α -Terpineol	1.12	10.26	6.63	5.12	3.20	2.52	2.31	2.18	1.93	1.44	1.11	1.06	0.88	0.87	0.58	0.25
17.64	1188	Methyl salicylate	27.85	80.72	46.29	25.10	8.32	7.81	7.22	5.43	4.57	4.42	3.89	3.57	0.49			
17.96	1193	Safranal	0.70	0.95	0.51	0.34	0.17	0.14	0.12	0.10	0.10							
18.83	1209	(<i>E,E</i>)-2,4-Nonadienal	25.54	2.13														
19.18	1216	β -Cyclocitral	1.39	24.46	12.55	9.01	3.26	3.14	2.32	2.59	2.03	1.53	1.30	1.06	0.93	0.74	0.46	
20.65	1246	2,5-Octanedione	9.66	23.18	16.22	11.13	5.43	5.42	3.37	2.40	2.35	1.38	0.34	0.29				
21.32	1259	Geraniol	0.84	9.59	7.22	5.02	4.77	3.54	3.03	2.84	1.83	1.69	1.95	1.40	0.77	0.45		
22.58	1285	Nonanoic acid	0.39	2.06	2.18	2.18	2.03	1.39	1.20	0.99	0.80	0.72	0.62	0.29	0.23			
22.69	1287	Grandlure II	12.32	23.84	4.27	4.00	2.12	1.85	1.53	1.44	1.33	0.59						
24.92	1325	1,2,3-Trimethoxybenzene	9.37	18.44	9.95	6.92	3.46	3.58	2.43	2.25	2.21	1.39	1.26	1.09	0.75	0.73	0.52	0.12
25.58	1335	Geranial	12.35	9.10	4.99	4.32	2.42	1.26	0.29									
27.69	1368	γ -Nonalactone	5.12	4.66	3.50	3.40	3.24	2.81	2.26	1.66	1.18	1.20	1.09	0.99	0.37	0.11		
31.63	1429	α -Ionone	0.11	42.78	25.67	14.54	11.16	10.38	9.56	9.46	7.89	6.77	5.45	5.22	4.19	3.75	2.07	1.03
33.27	1455	(<i>Z</i>)-Geranyl acetone	24.95	69.29	51.61	40.79	17.55	18.14	17.99	16.33	13.50	11.75	9.85	8.84	7.46	6.52	3.92	1.42
35.08	1483	β -Ionone	5.03	59.46	45.21	36.26	26.28	25.32	17.99	16.94	15.66	12.70	11.29	10.36	9.58	9.54	3.64	1.74
36.81	1514	2,4-Di-tert-butylphenol	0.27	1.98	3.98	7.36	7.65	7.77	6.55	6.15	5.53	5.17	4.94	3.40	2.63	1.01	0.71	0.49
37.27	1524	Dihydroactinidiolide	0.11	7.42	6.06	5.92	5.46	4.98	4.25	3.63	2.93	2.91	2.78	2.79	1.59	1.43	0.87	0.43
39.71	1576	Nerolidol	7.75	10.15	13.91	15.61	7.30	5.07	4.36	3.66	1.33	1.06	1.00	0.73	0.21			
41.38	1610	Cedrol	1.79	8.90	7.26	5.84	5.21	4.61	3.55	3.51	3.14	2.74	2.28	2.33	1.70	1.69	1.26	
43.53	1648	Dihydro- β -ionone	20.48															
60.55	1965	n-Hexadecanoic acid	0.41	16.67	12.17	10.94	9.01	6.00	6.17	5.88	6.09	3.84	3.33	3.06	2.49	0.13	0.11	0.09

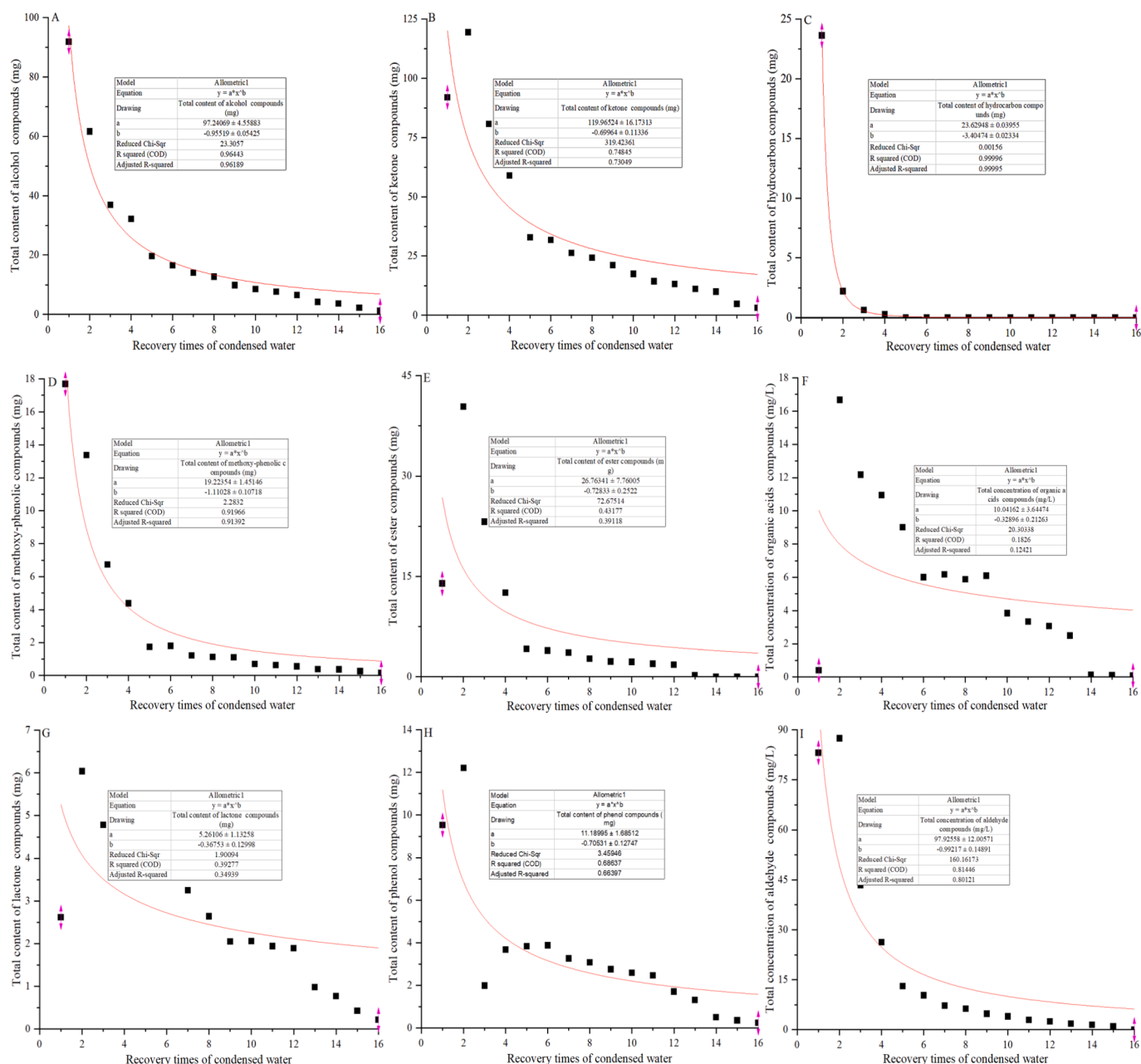


Fig. 3. The water piecewise recovered in the condensates during the extraction process of Fu-brick tea versus the contents of odor-active alcohols (A), ketones (B), hydrocarbons (C), methoxy-phenolic compounds (D), esters (E), organic acids (F), lactones (G), phenols (H), and aldehydes (I) in each condensed water. (y = content of each chemical class, x = recovery times).

Among those odor-active compounds, terpinen-4-ol (EC = -518.78), dihydro- β -ionone (EC = -516.50), 3-octen-2-one (EC = -4.04), (*E,E*)-2,4-nonadienal (EC = -3.71), *D*-limonene (EC = -3.41), 2-ethyl-1-hexanol (EC = -3.05), 6-methyl-5-hepten-2-one (EC = -2.30), 1,2-dimethoxybenzene (EC = -1.89), 4-*tert*-butylphenol (EC = -1.33), (*E*)-2-octenal (EC = -1.31), 3,5-octadien-2-one (EC = -1.18), geraniol (EC = -1.12), (*E*)-2-nonenal (EC = -1.10), hexanal (EC = -1.09), and octanal (EC = -1.06) had relatively high release rates. While α -terpineol (EC = -0.43), geraniol (EC = -0.39), α -ionone (EC = -0.38), β -ionone (EC = -0.34), phenylethyl alcohol (EC = -0.34), *n*-hexadecanoic acid (EC = -0.33), cedrol (EC = -0.32), dihydroactinidiolide (EC = -0.21), and 2,4-di-*tert*-butylphenol (EC = -0.02) were relatively slow (Table S2). The correlations between exponential coefficients (EC) and concentrations, molecular weights, and boiling points of odor-active compound were analyzed, respectively. The results showed that the release rates of odor-active

compounds had very little correlation with their concentrations (correlation coefficient [CC] = 0.03), molecular weights (CC = -0.15), and boiling points (CC = -0.11) (Table S3). The release rates of odor-active compounds may be related to their structure or the interaction between the odor-active compounds and the matrix, which needs further study.

According to the release amount of the odor-active compounds, only 6 compounds released more than 70% in CW1, accounting for 11.76% of the total odor-active compounds. For the second recovery, there were only 10 odor-active compounds with cumulative release of more than 70%, accounting for 19.61% of the total odor-active compounds. Until the eighth recovery, 37 odor-active compounds released more than 70%, accounting for 72.55% of the total odor-active compounds. When the cumulative release amount of all odor-active compounds exceeds 70%, it needs to be recovered 12 times, that is, 24% of the added water was

evaporated and recovered. The results showed that the complete release of most odor-active compounds was a slow process. In order to fully recover these odor-active compounds during boiling-water extraction of Fu-brick tea, it is necessary to recover more than 24 % of the added water. Similar results were found in the recovery of volatile/odor-active compounds in fruit juice and Pu-erh tea. Meanwhile, the researchers also found that in order to recover most of key odor-active compounds in fruit juice and Pu-erh tea, 10 %-35 % of the water evaporated off from the fruit juice and Pu-erh tea must be recovered during processing (Sampaio, Biasoto, Marques, Batista, & Silva, 2013; Wang, He, Zhang, Du, Xiao, & Xu, 2020).

3.4. Key odor-active compounds in each condensed water

The contributions of odor-active compounds to the entire aroma of CW depends not only on their concentrations, but also on their thresholds. At present, it was considered that odor-active compounds with OAVs ≥ 1 usually played a vital role in the formation of the aroma characteristics of analytical samples (Zhu, Niu, & Xiao, 2021). To deeper elucidate the contributions of odor-active compounds in each condensed water, their OAVs were calculated by using the quantitative results and reported thresholds, and the calculation results were shown in Table S4. By calculating the OAVs, 40, 40, 37, 37, 31, 28, 28, 27, 26, 26, 24, 23, 23, 17, 16, and 6 odor-active compounds with OAVs ≥ 1 were found in CW1, CW2, ..., CW16, respectively. Among them, dihydro- β -ionone showed the highest OAV (20480000), followed by β -ionone (248571–8494285), phenylethyl alcohol (38666–344666), (*E,E*)-2,4-heptadienal (16250–259062), (*E,E*)-2,4-nonadienal (13312–159625), α -ionone (183–71300), (*E*)-2-nonenal (4200–44900), β -linalool (725–25650), 1,2,3-trimethoxybenzene (160–24587), octanal (425–20900), cedrol (2520–17800), hexanal (300–11082), 3-octen-2-one (191–9739), geraniol (409–8718), 1,2-dimethoxybenzene (577–8208), β -cyclocitral (153–8153), heptanal (100–6444), *D*-limonene (51–4727), (*E*)-2-octenal (525–2777), methyl salicylate (12–2018), nerolidol (21–1561), and (*Z*)-geranylacetone (24–1155).

To evaluate the accuracy of screening results of key odor-active compounds and their quantitative results, the odor-active compounds with OAVs ≥ 1 were added into ultra-pure water according to their concentrations detected in condensed water for the preparation of recombination samples. And then, the recombination samples were sealed and kept for 24 h at 4 °C before sensory analysis. The sensory results of recombination samples and their corresponding condensed water were shown in Fig. 2. The aroma profiles of condensed water samples could be successfully imitated to a certain extent by combining odor-active compounds. The main attributes (woody, stale, green, and floral/fruity) of recombination samples were consistent with the corresponding condensed water samples. The 'herbal' and 'off-flavor' attributes of most recombination samples were higher than that of the corresponding condensed water samples, which might be caused by the purity of commercial standards <100 % or the interaction between odor-active compounds. The exact reasons need to be further studied. Overall, odor recombination further verified the accuracy of screening key odor-active compounds in condensed water.

4. Conclusion

Most of odor-active compounds were released during the deep-processing of Fu-brick tea. But their release behaviors are not clear, which affects the effective recovery of odor-active compounds and the improvement of the flavor quality of deep-processing products of Fu-brick tea. In this study, the odor-active compounds in Fu-brick tea and their release behaviors during boiling-water were identified and evaluated. Fifty-one odor-active compounds were identified in Fu-brick tea, and their release behaviors were identified by continuously recovering 16 sections of condensed water combined with sensory, instrumental, and nonlinear curve fitting analysis. The odor intensities of condensed

water and the concentrations of odor-active compounds could be significantly fitted ($p < 0.01$) to power-function type curves. Hydrocarbons showed the fastest release rate, while organic acids showed the slowest. The release rates of odor-active compounds had very little correlation with their concentrations, molecular weights, and boiling points. Most odor-active compounds (≥ 70 %) released need to evaporate more than 24 % of the added water during boiling-water extraction. Meanwhile, the aroma recombination experiments were also performed to explore the odor-active compounds that made major contributions to the formation of the aroma profile of each condensed water on the basis of OAVs calculation. This research results can provide theoretical support for recovering these compounds and improving the flavor quality of deep-processing products of Fu-brick tea.

CRedit authorship contribution statement

Chao Wang: Conceptualization, Methodology, Software, Data curation, Funding acquisition, Formal analysis, Writing – original draft, Writing – review & editing. **Wazhen Xu:** Data curation, Formal analysis, Methodology, Resources, Software, Validation, Writing – review & editing. **Yuqi Yuan:** Data curation, Formal analysis, Methodology, Resources, Supervision, Writing – review & editing. **Yuke Zhai:** Validation, Formal analysis, Investigation, Resources, Data curation. **Tengfei Hu:** Validation, Formal analysis, Investigation, Resources, Data curation. **Jianan Huang:** Supervision, Project administration, Funding acquisition. **Zhonghua Liu:** Supervision, Project administration, Funding acquisition. **Qin Li:** Supervision, Project administration, Funding acquisition.

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.

Data availability

The data that has been used is confidential.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.fochx.2022.100551>.

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