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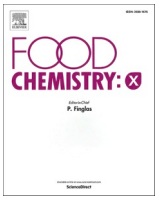

Effect of osmanthus hydrolat on the aroma quality and volatile components of osmanthus black tea

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ABSTRACT

Osmanthus fragrans is an evergreen shrub with a pleasant fragrance and a wide range of applications in many fields. The condensed hydrolat obtained during the drying process of its fresh flowers was collected in a low-temperature vacuum environment and its sensory evaluation and volatile components were studied. The main aroma compounds in *Osmanthus fragrans* were dihydro-*β*-ionone, nonanal, *β*-cyclocitral, *β*-ionone, benzaldehyde, *α*-ionone, and 6-methyl-5-hepten-2-one, whose contents were used as the main evaluation criteria, and the hydrolats obtained under different scenting and drying times were compared. This process can effectively collect the aroma components in *Osmanthus fragrans* and the optimal drying conditions were 50 °C for 5 h. The hydrolat was used to provide the scent of osmanthus black tea, which had a fresher and mellower taste, while the fragrance of osmanthus was abundant. These results show that osmanthus hydrolat can be used to provide the scent of floral black tea.

Chemical compounds studied in this article: (−)-Catechin (PubChem CID: 1203); (−)-epigallocatechin gallate (PubChem CID: 65064); (−)-epicatechin gallate (PubChem CID: 367141); (−)-epigallocatechin (PubChem CID: 72277); (−)-epicatechin (PubChem CID: 72276); (−)-gallocatechin gallate (PubChem CID: 199472); (−)-catechin gallate (PubChem CID: 6419835); (−)-gallocatechin (PubChem CID: 9882981).

1. Introduction

Osmanthus fragrans (*O. fragrans*) is one of the ten famous flowers found in China, and includes four main varieties: *O. fragrans* var. latifolius (Silver O.fragrans), *O. fragrans* var. thunbergii (Gold O.fragrans), *O. fragrans* var. aurantiacus (Dan O.fragrans), and *O. fragrans* var. semperflorens (Four Seasons O.fragrans). *Osmanthus fragrans* has anti-oxidation (Ouyang et al., 2015), anti-inflammatory (Tang et al., 2021), anti-aging (Wang, Luan, et al., 2022) and other properties. The floral fragrance of *O. fragrans* is one of its most important qualities, which also has anti-anxiety and anti-depression effects (Yang et al., 2023). Recently, *Osmanthus fragrans* has been widely introduced and planted as an important garden flower and tree, and it is used for the production of flavors, fragrances, and aromatic oils (Wu, 2022). The extracts of *Osmanthus fragrans* include *O. fragrans* essential oil, *O. fragrans* hydrosol, *O. fragrans* hydrolat, etc.

Essential oils are oily liquid substances with directional odor, which are usually prepared via steam distillation (Zhou et al., 2023). The main volatile components of osmanthus essential oil are 1,2-epoxylinalool, *β*-linalool, 5-ethenyltetrahydro-*α*,*α*,5-trimethyl-2-furanmethanol, and *β*-ionol (Hu et al., 2010). Hydrosol is the by-product obtained during the extraction of flower essential oil via steam distillation (Xu et al., 2020). There are few studies on pure dew, such as spearmint pure dew (Han et al., 2023) and rose pure dew (Ling et al., 2021). Guo et al. (2023) studied the volatile components in *O. fragrans* extract and absolute, and found that the relative content of alcohols was highest, which were the main contributors to its characteristics. At the same time, several studies have shown that dihydro-*β*-ionol, geranylgeraniol, nonanal, *β*-ionone, and dihydro-*β*-ionone significantly contribute to the aroma of osmanthus extract and absolute. Flower hydrolat is the original liquid from

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flower cells collected via a low temperature dehumidification process (Zhang et al., 2010). To our knowledge, little studies have been reported on the hydrolat of *O. fragrans*.

The scent of traditional scented tea is mainly provided using an exogenous scenting method utilizing fresh flowers. Floral essential oil-casein nanoparticles are also used for tea aroma (Xu et al., 2021). There is no related research on scenting osmanthus black tea with hydrolat. The complex scenting process using fresh flowers requires multiple scenting steps, which is also limited by the flowering period of fresh flowers. However, the yield of flower essential oil is often low, which requires a large number of fresh flowers and high cost.

In this study, the hydrolat of *O. fragrans* was collected via condensation under a low-temperature vacuum environment. It was found that the collected hydrolat contained most of the volatile components in *O. fragrans*, which provide the aroma characteristics of *O. fragrans*. The preparation process does not use chemical reagents and the collected hydrolat has a natural floral fragrance. Solid-phase microextraction-chromatography-mass spectrometry (SPME-GC–MS) is a method that can be used to analyze the differences in the aroma components (Tang et al., 2023). Consequently, the aroma components of *O. fragrans*, *O. fragrans* hydrolat, and *O. fragrans* residue were studied based on this technique and the characteristic aroma components of *O. fragrans* hydrolat were determined. The optimal preparation process parameters were determined using the sensory evaluation of *O. fragrans* hydrolat combined with the results of the volatile component analysis, which provides a reference for the preparation of floral hydrolat. Subsequently, the *O. fragrans* hydrolat was used for the fragrance of the tea dhool, reducing the scenting steps of the scented tea, so as to enhance the added value of the osmanthus extract.

2. Materials and methods

2.1. Materials and chemicals

Fresh leaves with one bud and two or three leaves were picked in Pan'an, China in October 2023. Fresh *O. fragrans* var. *thunbergii* was collected in Jinhua, Zhejiang Province, China in October 2023.

(−)-Epigallocatechin gallate (EGCG), (−)-gallocatechin gallate (GCG), (−)-epicatechin (EC), (−)-catechin (C), (−)-epigallocatechin (EGC), (−)-epicatechin gallate (ECG), and gallic acid (GA) [≥99% pure by high-performance liquid chromatography (HPLC)] were purchased from Sigma-Aldrich (Shanghai, China). Acetonitrile (ACN, HPLC grade) and glacial acetic acid (≥99.9%) were obtained from Shanghai Aladdin Biochemical Technology Co. Ltd. (Shanghai, China). Ethyl caprate (decanoate; 99%) was purchased from Beijing Zhongsheng Ruitai Technology Co., Ltd. (Beijing, China), and purified water from Hangzhou Wahaha Group Co., Ltd. (Hangzhou, China).

2.2. Preparation of Osmanthus fragrans hydrolat

Picked fresh *O. fragrans* was selected, the stems and other impurities removed, and the flowers screened. 80.0 g of *O. fragrans* were placed in a rotary evaporation bottle and the rotary evaporation conditions were as follows: rotation speed 45 rpm; vacuum degree 20 mbar; temperature 40, 50, and 60 °C; and time 4, 5, and 6 h. The condensed *O. fragrans* hydrolat (OH) (Table 1 A) and remaining *O. fragrans* residue (OR) (Table 1 A) were collected.

2.3. Calculation of the hydrolat collection rate

The following formula was used for calculations:

$$C = \frac{\Delta M}{M} \times 100\%$$

where ΔM is the mass of the collected hydrolat (g) and M is the mass of fresh *O. fragrans* (g).

Table 1
Sample coding table. (A) Sample number of *O. fragrans* hydrolat and *O. fragrans* residue. (B) Sample number of *O. fragrans* black tea.

(A) Sample number of *O. fragrans* hydrolat and *O. fragrans* residue.

Time /h Temperature /°C	4	5	6
40	OH-1/OR-1	OH-4/OR-4	OH-7/OR-7
50	OH-2/OR-2	OH-5/OR-5	OH-8/OR-8
60	OH-3/OR-3	OH-6/OR-6	OH-9/OR-9

(B) Sample number of *O. fragrans* black tea.

Time /h Temperature /°C	4	8	12
65	OT-1	OT-4	OT-7
70	OT-2	OT-5	OT-8
75	OT-3	OT-6	OT-9

2.4. Sensory evaluation

Referring to the method provided in GB/T 14454.2–2008 (Administration of Quality Supervision, I. A. Q., and China, S. A. o, 2008), 3 mL of each group of *O. fragrans* hydrolat was added to a glass tube and perfume paper dipped into the *O. fragrans* hydrolat to a depth of ~1 cm. The sensory quality was evaluated by a sensory group composed of ten experienced team members (five males and five females). The floral, sweet, and freshness of the *O. fragrans* hydrolat aroma were scored. The descriptors were evaluated on a nine-point Hedonic Scale: 0 for unscented, 3 for weak aroma, 6 for moderate aroma, and 9 for strong aroma; the overall acceptability was scored using a similar process.

2.5. Analysis of the volatile compounds

2.5.1. O. fragrans and O. fragrans residue aroma extraction using SPME

One gram of fresh *O. fragrans or O. fragrans* residue was weighed in a 20-mL headspace bottle, and 10 μL of ethyl decanoate (internal standard, 50 μg/mL) was added to quickly cover the sample and equilibrated for 20 min at 60 °C in a constant temperature water bath. The headspace volatiles were absorbed for 60 min using a divinylbenzene/carboxen/polydimethylsiloxane [50/30 μm, stable flex (2 cm)] coating fiber (Supelco, Inc., Bellefonte, PA, USA). Subsequently, the volatiles were desorbed at 250 °C for 5 min in the GC–MS injector.

2.5.2. O. fragrans hydrolat aroma extraction using SPME

Next, 5.0 mL of *O. fragrans* hydrolat was transferred to a 20-mL headspace vial. NaCl was added at a –mass concentration of 0.35 g/mL and 10 μL of ethyl decanoate (internal standard, 50 μg/mL) was added for rapid capping. Equilibrium was maintained at 60 °C for 20 min. The head-space volatiles were then absorbed for 60 min using a divinylbenzene/carboxen/polydimethylsiloxane [50/30 μm, stable flex (2 cm)] coating fiber (Supelco, Inc., Bellefonte, PA, USA). Subsequently, the volatiles were desorbed at 250 °C for 5 min in the GC–MS injector.

2.5.3. GC–MS analysis of the volatile compounds

An Agilent 6890 GC interfaced with an Agilent HP 5975 MSD ion trap MS (Wilmington, DE) fitted with a DB-5MS capillary column (30 m × 250 μm × 0.25 μm) was used for volatile compound analysis. The GC conditions were as follows: Inlet temperature, 250 °C; carrier gas, high purity helium (99.999%); flow rate, 1.0 mL/min. The temperature program was as follows: 40 °C for 2 min; increase to 60 °C at 2 °C/min; increase to 160 °C at 5 °C/min; increase to 250 °C at 10 °C/min and held for 2 min. The MS analysis was performed at 70 eV in EI mode over a mass range of 40–400 *m/z* using an ion source temperature of 230 °C.

2.5.4. Identification

The volatile compounds were identified using the National Institute of Standards and Technology (NIST) library 98 L mass spectral search program and an internally created GC–MS data analysis program (Wang, Fu, et al., 2022). The compounds were identified using the MS library and retention index. In this study, an internal standard method was used to calculate the relative content of the aroma components with reference to the internal standard concentration and peak area. The aroma contributions of each volatile component were evaluated by calculating their relative odor activity value (rOAV) (Gao et al., 2023). The rOAV is defined as the ratio of the relative concentration of an aroma component to its odor threshold (i.e., the minimum concentration of a substance that can cause human sensory stimulation).

2.6. Scenting of osmanthus black tea using the hydrolat

Preparation of black tea dhool (BT): The production process of black tea was as follows: Withering to 60% of the water content of fresh leaves at room temperature, followed by rolling for 50 min. The leaves were fermented at 35–45 °C and 90–94% humidity for 4 h, and finally dried to obtain the black tea sample. The initial drying conditions were 110 °C for 30 min and the redrying conditions were 80 °C for 35 min. Subsequently, the black tea was divided into 60.0 g each for scenting.

Preparation of *O. fragrans* hydrolat: 80.0 g of *O. fragrans* were prepared after screening. The *O. fragrans* hydrolat was collected at 50 °C over 5 h (OH-5), and the other conditions were the same as those described in Section 2.2.

Preparation of *O. fragrans* black tea: The osmanthus hydrolat was sprayed on the black tea sample, which was divided into three groups. After scenting for 4, 8, and 12 h, one part of the scented black tea was taken from each group and baked at 65, 70, and 75 °C for 35 min to obtain the osmanthus black tea sample (OT) (Table 1B).

2.7. Sensory evaluation of the tea infusions

Nine tea samples were brewed using boiling water at a tea/water ratio of 1:50 (*w*/w) for 4 min. A sensory panel of ten experienced team members (five men and five women) assessed the sensory quality. The tea samples were scored using different aroma attributes: Floral, fruity, sweet, green, and roasted, as described in GB/T 23776–2018 (Administration of Quality Supervision, I. A. Q., and China, S. A. O, 2018). The descriptor was evaluated using 9 points: 0 points indicated no smell, 3 points indicated weak aroma, 6 points indicated obvious aroma, and 9 points indicated strong aroma.

2.8. Determination of main quality components

Ten kinds of tea samples were brewed using boiling water at a tea-water ratio of 1:50 (*w*/w) for 4 min to obtain the tea infusion samples.

The total concentration of tea polyphenols (TP) in the tea infusions was measured using the Folin–Ciocalteu reagent (Cao, Wang, et al., 2021). To a 10-mL volumetric flask containing 1 mL of sample or GA standard (0–100 mg/L), 5 mL of Folin–Ciocalteu reagent (10%, *v*/v) was added, and 4 mL of Na_2CO_3 (7.5% v/v) then mixed with the resulting solution after 5 min. The reaction was kept at room temperature for 60 min and the absorbance was measured at 765 nm using a UV–Vis spectrophotometer (UV3600, Shimadzu, Tokyo, Japan).

The total concentration of free amino acids (AA) in the soaking solution was determined using ninhydrin reagent: 0.5 mL of phosphate buffer (pH 8.0) and 0.5 mL of 2% ninhydrin solution were added to a 25-mL volumetric flask containing 1 mL of sample or glutamic acid standard (0–0.9 mg/mL) (Cao, Wang, et al., 2021). The mixture was heated in water bath at 100 °C for 15 min and then cooled to a constant volume. The absorbance was measured at 570 nm after 10 min.

The catechins, caffeine (Caf), and gallic acid (GA) components in the tea infusions were determined using HPLC (Agilent Technologies, Santa Clara, CA, USA). The samples were filtered through a 0.45-μm filter. A Diamonsil C18 column (4.6 mm × 250 mm, 5 μm; Dikma Technologies, Lake Forest, CA, USA) maintained at 40 °C was used. The mobile phases were aqueous 2% acetic acid (A) and ACN (B). The mobile phase composition was 6.5% B at 0 min, which was then linearly ramped to 15% B at 16 min and 6.5% B at 25 min, and then held until 30 min. The flow rate was 1 mL/min and the detection wavelength was 280 nm (Cao, Fu, et al., 2021; Xu et al., 2017).

2.9. Analysis of the aroma components of osmanthus black tea

2.9.1. Tea aroma extraction using SPME

Each tea sample (0.5 g) was sealed into a 20-mL glass vial and ethyl caprate (internal standard, 10 μL, 10 mg/L) and boiling deionized water (5 mL) added. After equilibrating the mixture for 5 min, the vial was transferred to a water bath heated at 60 °C, and the head-space volatiles were absorbed for 60 min using a divinylbenzene/carboxen/polydimethylsiloxane [50/30 μm, stable flex (2 cm)] coating fiber (Supelco, Inc., Bellefonte, PA, USA). Subsequently, the volatile compounds were desorbed at 250 °C for 5 min in the GC–MS injector.

2.9.2. GC–MS analysis of the volatile compounds

The temperature program was as follows: 40 °C for 2 min, increase to 85 °C at 2 °C/min, and hold for 2 min; increase to 180 °C at 2.5 °C/min, hold for 2 min; increase to 230 °C at 10 °C/min and hold for 2 min. The remaining conditions were the same as those described in Section 2.5.

2.10. Statistical analysis

All experiments were conducted in triplicates. Multivariate analysis techniques were performed using SIMCA 14.1 (Umetrics AB, Umea, Sweden). IBM SPSS Statistics (version 23.0, SPSS Inc., Chicago, IL) was employed for data analysis, using one-way analysis of variance ($p < 0.05$) and significance testing. GraphPad Prism 8 (GraphPad Software Inc., San Diego, CA, USA) and Origin 2021 (OriginLab, Northampton, MA, USA) was used for plotting graphs.

3. Results and discussion

3.1. Analysis of the extraction rate of O. fragrans hydrolat

The yield of *Osmanthus fragrans* cell fluid was the highest under the conditions of 50 °C, 5 h and 60 °C, 6 h, reaching 79.00%. Under the conditions of 40 °C, 4 h, the yield of *Osmanthus fragrans* cell fluid was lowest, reaching 47.25% (Fig. S1A).

3.2. Sensory evaluation of O. fragrans hydrolat

The floral, sweet, and freshness of osmanthus hydrolat were reduced at the same temperature with an increase in time (Fig. S1B). At the same time, the floral score of *O. fragrans* hydrolat decreased with an increase in temperature. The higher the temperature, the more significant this decrease, and the scores for sweet and freshness also decrease to varying degrees. Among them, the floral score of CS-5 was the most obvious, but the freshness was slightly reduced; CS-1 exhibits the highest freshness because under these conditions, the temperature was the lowest, the time was the shortest, and the state of *O. fragrans* was maintained. The freshness of the *O. fragrans* hydrolat decreases with an increase in temperature and time.

3.3. Volatile components analysis

3.3.1. Analysis of the volatile components in O. fragrans

According to GC–MS analysis, 125 volatile components (Table S1) were detected in the fresh flowers of *O. fragrans* (CK), including 33 esters, 25 aldehydes, 20 ketones, 16 alcohols, 10 hydrocarbons, 10 olefins,

3 ethers, 3 oxygen-containing heterocyclic compounds, 1 acid, and 4 others. The content of total volatile substances in the fresh flowers of *Osmanthus fragrans* was 5.50 mg/L (Fig. 1A), among which the content of ketones was highest (3.52 mg/L), and the content of acids was the lowest (1.95 μg/L). According to the proportion of the different aroma compounds, the aroma components in *O. fragrans* were mainly ketones (64.11%), esters (13.87%), and aldehydes (12.93%) (Fig. 1B).

Ketones are the main aroma components in *Osmanthus fragrans* (Fu et al., 2019). A large number of ketones were detected in the flowers of *O. fragrans*, including dihydro-*β*-ionone (woody, violet, fruity), *α*-ionone (woody, violet, fruity), *β*-ionone (woody, violet, fruity), and 6-methyl-5-hepten-2-one (fatty, green, citrus-like), the contents of each reach 2.20, 0.81, 0.25, and 0.10 mg/L, respectively. The aroma quality of *Osmanthus fragrans* is significantly affected by many factors, such as the variety of *Osmanthus fragrans*, flowering period, growth environment etc. (Guo et al., 2022). Meanwhile, the aroma components in the different varieties of *Osmanthus fragrans* are quite different (Cai et al., 2014; Fu et al., 2017). This is mainly manifested in the composition and ratio of the aroma components. The content of ionones in the *O. fragrans* var. *thunbergii* cultivar group was high, mainly exhibiting a woody aroma

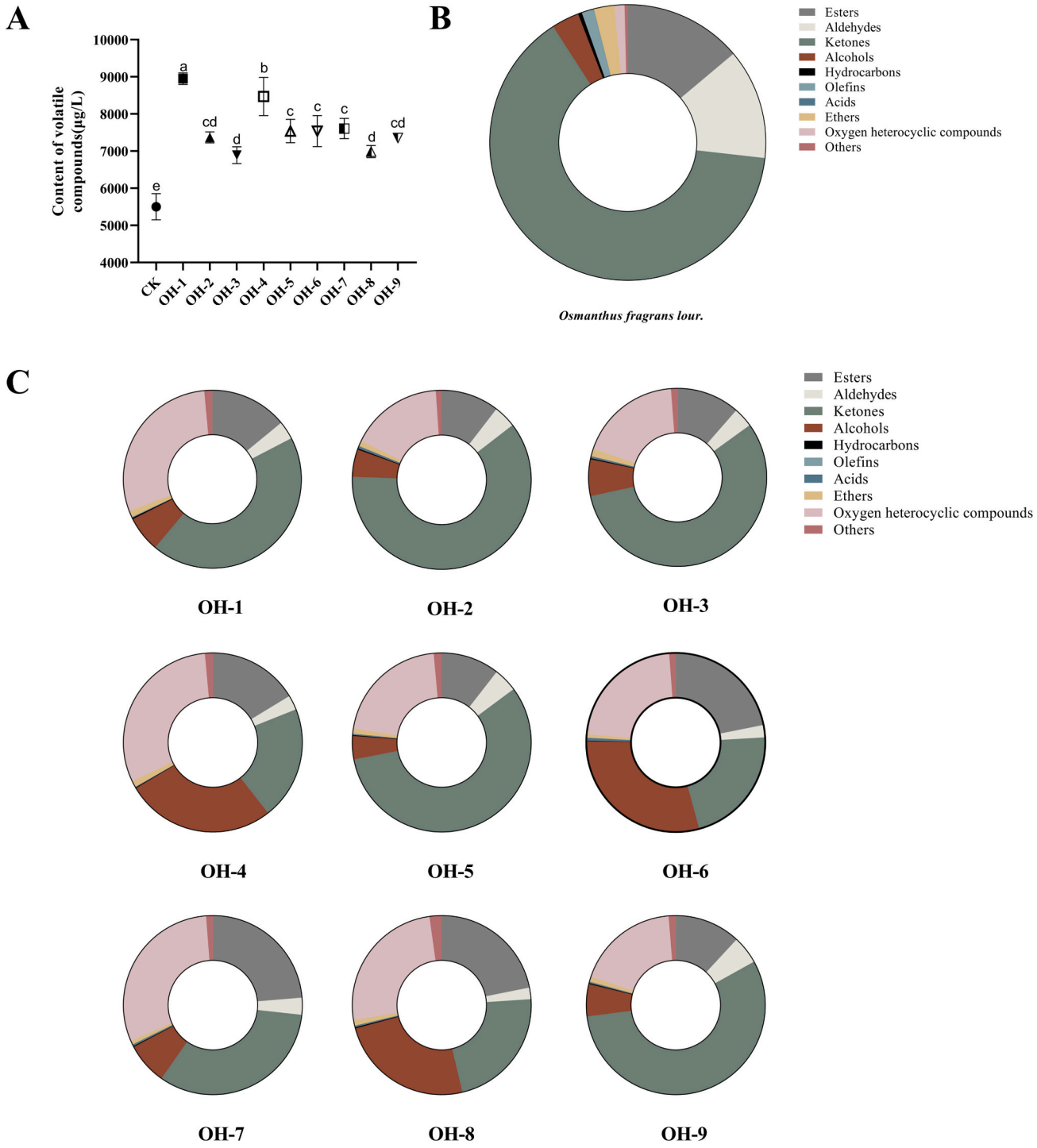

Fig. 1. Analysis of aroma components of *O. fragrans* and. Hydrolat. (A) Total content of volatile components. (B) The proportion of aroma components of CK (*O. fragrans*). (C) The proportion of aroma components in hydrolat.

type (Zhu et al., 2022). In this study, the volatile composition of *O. fragrans* var. *thunbergii* was found to contain more than half of the ionones (Table S1). Violet ketone and its derivatives are mainly produced by carotenoid cleavage (Sun et al., 2024), exhibiting woody, violet, and fruity aroma characteristics. Violet ketone compounds provide the typical aroma characteristics of *O. fragrans* (Zhou et al., 2023). The aroma types of the ester compounds are mainly fruity (Niu & Zhao, 2023), such as γ-decalactone, nonanoic acid ethyl ester, and (*Z*)-3-hexenyl isobutyrate. Among them, the content of γ-decalactone was highest, reaching 0.43 mg/L, which is the characteristic aroma substance in *O. fragrans* (Yang et al., 2023). The main aldehydes were β-cyclocitral, nonanal and 2-hexenal, with the contents of 0.16, 0.15 and 0.09 mg/L, respectively. β-Ionone, cis-linalool oxide furanoid, trans-linalool oxide furanoid, and linalool are the common components found in most varieties of *O. fragrans* (Xin et al., 2013). In this study, β-ionone and trans-linalool oxide furanoid were detected. The results of study show that the relative contents of dihydro-β-ionone, α-ionone, γ-decalactone, β-ionone, β-cyclocitral, and nonanal were the highest in regard the volatile components in *O. fragrans*.

3.3.2. Analysis of the volatile components in O. fragrans hydrolat

O. fragrans hydrolat collected under different conditions had significant differences in terms of the aroma components. A total of 161 volatile components were detected in the nine *O. fragrans* hydrolat samples (Table S1), including 35 esters, 30 aldehydes, 29 ketones, 28 alcohols, 13 hydrocarbons, 5 olefins, 5 acids, 4 ethers, 4 oxygen-containing heterocyclic compounds, and 9 others. From the perspective of the total amount of volatile compounds, the highest was observed in OH-1 (up to 8.87 mg/L) and the lowest was observed in OH-8 (6.99 mg/L) (Fig. 1A). Obviously, the total content of volatile substances in the hydrolat of *O. fragrans* was significantly higher than that in fresh *O. fragrans*.

The effect of temperature on the aroma content of the *O. fragrans* hydrolat was obvious. At the same time, the total content of volatile compounds shows a downward trend with an increase in temperature, and the decrease was the most obvious at 4 h. Under the conditions of constant temperature, 40 and 50 °C showed a downward trend with an increase in time, while 60 °C increased slightly with an increase in time. The results of sensory evaluations show that the *O. fragrans* hydrolat obtained at 40 °C had a higher flower aroma and freshness. Zhou et al. (2023) have studied the effects of different heat treatments (80–120 °C) on the volatile components in *O. fragrans*, and indicates that the content of volatile components in *O. fragrans* decreased significantly with the increase of temperature. This was consistent with the results of the present study. Furthermore, this research has attempted to use the same vacuum environment under the conditions of <40 °C, however the *O. fragrans* hydrolat could not be obtained efficiently.

Fig. 1C shows that the proportion of aroma components in different hydrolat were different. The volatile components in OH-1, OH-2, OH-3, OH-5, OH-7, and OH-9 were mainly ketones, oxygen-containing heterocyclic compounds, and esters. The relative content of ketones in OH-2, OH-3, OH-5, and OH-9 can reach more than half of the total amount, and the highest proportion of ketones in OH-2 was 61.00%. Samples of OH-4, OH-6, and OH-8 were mainly composed of oxygen-containing heterocyclic compounds, alcohols, ketones, and esters, and the relative contents of these four types of compounds in each sample were similar.

Ketones were the most abundant volatile components in the hydrolat of *O. fragrans*, which mainly include dihydro-β- ionone, α-ionone, β-ionone, and 6-pentyl-2H-pyran-2-one. It can be clearly seen that the content of dihydro-β- ionone with woody and violet fragrance characteristics was the highest with a maximum content of 2.97 mg/L. The oxygen-containing heterocyclic compounds were mainly trans-linalool oxide furanoids with floral fragrance, and the highest content was 2.56 mg/L in OH-1. The ester compounds were mainly γ-decalactone with fruity aroma, and the highest content in OH-6 was 1.72 mg/L.

3.3.3. Identification of the main aroma components in the hydrolat of O. fragrans

In order to explore the differences in the aroma compositions of osmanthus hydrolat obtained at different temperatures and times of collection, the characteristic differential aroma compositions of the nine groups of samples were further analyzed using the partial least squares discriminant analysis (PLS-DA) model with fitting indices of $R^2X = 0.993$, $R^2Y = 0.959$, and $Q^2 = 0.844$ (Fig. 2A, B). To further investigate the robustness of the model, the cross-validation model was validated using a permutation test over 200 times (Fig. 2C). The intersection of the Q^2 regression line with the vertical axis was <0 ($R^2 = 0.369$, $Q^2 = -0.826$), and these indicators prove the reliability of the applied PLS-DA model. It is believed that the results can be used to analyze the differences in the aroma components in *O. fragrans* hydrolat collected at different temperatures and time. A total of 33 key differential compounds with VIP >1 were screened using PLS-DA for subsequent analysis (Fig. 2D).

The contribution of aroma compounds to the sensory quality of osmanthus hydrolat can be approximated by OAV. More importantly, compounds with relatively high OAV are considered to contribute more to their aroma. To this effect, the rOAVs of 33 key differential compounds were compared and found in Table 2. A total of 9 aroma components with rOAVs >1 were screened out, which were trans-linalool oxide furanoid, β-ionone, α-ionone, dihydro-β-ionone, phenylacetaldehyde, benzaldehyde, nonanal, methyl benzoate and jasmone. These components are considered to be the main contribution of the aromatic substances in the hydrolat of *O.fragrans* because they have a lower odor threshold and relatively higher rOAVs. The characteristic aroma components. At the same time, it was found that the characteristic aroma components of *O. fragrans* were dihydro-β-ionone, nonanal, β-cyclocitral, β-ionone, benzaldehyde, α-ionone, 6-methyl-5-hepten-2-one, these seven components.

In-depth analysis shows that the main aroma components in the nine samples were significantly different (Fig. 2E). The total content observed in OH-1 was the highest, reaching 6.32 mg/L, followed by OH-5 (5.73 mg/L). The contents in OH-6 and OH-8 were lower than those of the flowers (Fig. 2F).

3.3.4. Analysis of the volatile components in O. fragrans residue

The remaining osmanthus residues obtained under different conditions exhibit significant differences in their aroma components. After GC–MS analysis, 162 aroma components were found (Table S2), which could be divided into 38 esters, 29 aldehydes, 28 ketones, 26 alcohols, 13 alkenes, 13 alkanes, 10 oxygen-containing heterocyclic compounds, and 5 others. The aroma was dominated by ketones, oxygen-containing heterocyclic compounds, and alcohols. The highest total aroma content in the remaining flower residue was observed in OR-1, reaching 5.27 mg/L, and the lowest content was found in OR-6 (only 0.95 mg/L) (Fig. S2).

When compared with fresh *Osmanthus fragrans*, the total volatile compound concentration in the OR group was lower than the total aroma in the fresh flowers and lower than the corresponding OH group. Obviously, with an increase in temperature, the total aroma shows a downward trend. Rotary evaporation is equivalent to a drying process, the higher the temperature, the faster the aroma volatilization rate; the longer the time, the greater the amount of aroma volatilization, which may be one of the reasons for the decrease in the total amount of aroma compounds.

3.3.5. Comparison of the main aroma substances in O. fragrans

The analysis of the main aroma components in *O. fragrans* in the OH group showed that the content of OH-2 was the highest, reaching 4.19 mg/L, and the content of OH-8 was the lowest (only 1.33 mg/L) (Fig. S3A). The total content observed in OH-2 and OH-5 was higher than that of *O. fragrans*, and the content of dihydro-β-ionone was high. The contents observed in OH-4, OH-6, and OH-8 were much lower than

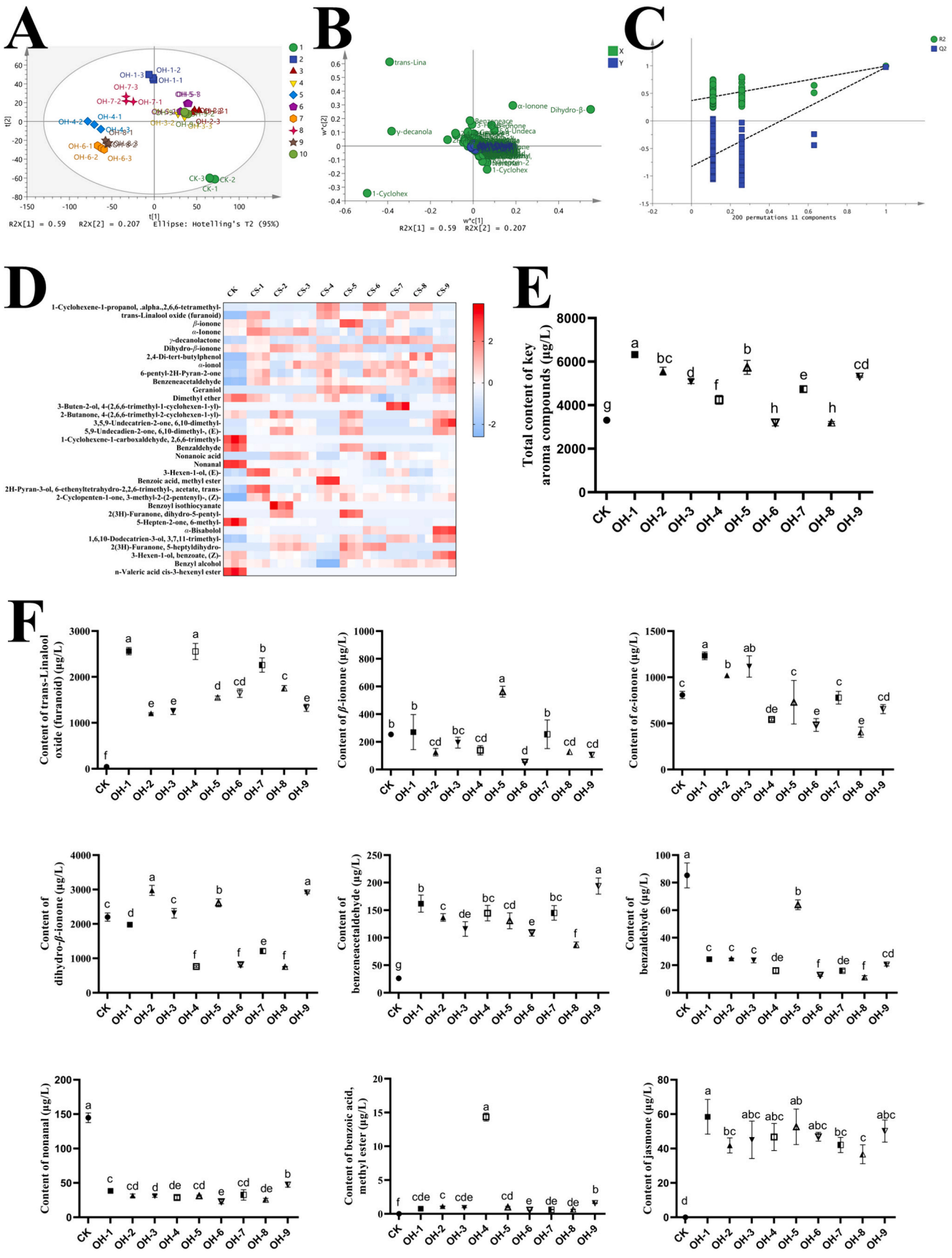

Fig. 2. Analysis of volatile components in *O. fragrans* hydrolats. (A) The score scatter plot of PLS-DA of different osmanthus black tea. (B) Loading scatter plot. (C) Validation of the PLS-DA model. (D) Heat map of main differential compounds in osmanthus black tea. (E) Total amount of main aroma substances (F) The content of 9 main aroma substances in hydrolats.

Table 2
The key compounds responsible for CK (*O. fragrans)* and hydrolats with significant high odor-activity values (VIP > 1).

Volatile compounds	Odor Description[A]	rOAVs[B]										OT[C] (μg/L)
		CK	OH-1	OH-2	OH-3	OH-4	OH-5	OH-6	OH-7	OH-8	OH-9	
Dihydro-*β*-ionol	Woody	-	-	-	-	-	-	-	-	-	-	-
trans-Linalool oxide (furanoid)	Floral	0.73	42.68	20.10	20.80	42.56	25.85	27.41	37.65	29.18	22.12	60a
β-ionone	Woody, berry, floral and fruity	30.18	32.17	14.93	23.15	16.53	66.94	6.41	30.35	15.25	12.54	8.4b
α-Ionone	Sweet-floral odor, violet	10.63	16.21	13.43	14.68	7.12	9.60	6.35	10.25	5.33	8.61	76b
γ-decanolactone	Peach, fat	0.43	1.16	0.66	0.70	1.28	0.67	1.56	1.72	1.46	0.76	1000c
Dihydro-*β*-ionone	Fruity	2198.00	1978.16	2974.59	2311.39	761.93	2612.79	813.98	1211.01	756.54	2908.46	1a
2,4-Di-tert-butylphenol												
α-ionol	-	-	-	-	-	-	-	-	-	-	-	-
6-pentyl-2H-Pyran-2-one	Sweet floral, violet, woody.	-	-	-	-	-	-	-	-	-	-	-
Benzeneacetaldehyde												
Geraniol	Coconut flavor	0.41	1.14	0.55	0.68	1.15	0.61	1.27	1.07	1.13	0.87	150d
Dimethyl ether												
	-	0.87	5.39	4.56	3.86	4.82	4.35	3.63	4.83	2.90	6.46	30d
3-Buten-2-ol, 4-(2,6,6-trimethyl-1-cyclohexen-1-yl)-	Sweet rose odor	0.00	0.00	0.00	0.00	18.82	22.29	14.51	0.00	0.00	18.26	1.1a
		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	50000e
2-Butanone, 4-(2,6,6-trimethyl-2-cyclohexen-1-yl)-	Chloroform-like odor; sweet -	-	-	-	-	-	-	-	-	-	-	-
3,5,9-Undecatrien-2-one, 6,10-dimethyl-(*E*)-		-	-	-	-	-	-	-	-	-	-	-
5,9-Undecadien-2-one, 6,10-dimethyl-	Woody,floral, powdery,fruity											
β-cyclocitral												
Benzaldehyde	Sweet, spicy	0.00	0.01	0.02	0.01	0.00	0.02	0.00	0.00	0.02	0.05	800d
Nonanoic acid												
Nonanal	Fresh green, fruity	0.00	0.67	2.20	0.95	0.00	2.15	0.00	0.00	0.00	2.17	60d
3-Hexen-1-ol, (*E*)-												
Benzoic acid, methyl ester		32.19	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	5d
2H-Pyran-3-ol, 6-ethenyltetrahydro-2,2,6-trimethyl-,acetate,trans-	Herbal, Rose Oxide, Fruity	17.06	4.88	4.99	4.71	3.21	12.79	2.53	3.19	2.25	4.08	2.4a
Jasmone	Almond, burnt	0.00	0.00	0.01	0.01	0.00	0.00	0.01	0.00	0.00	0.00	3000d
Benzoyl isothiocyanate	sugar	55.65	14.78	12.18	11.86	11.03	12.00	8.78	12.52	9.89	18.22	2.6a
2(3H)-Furanone, dihydro-5-pentyl-	Cheese flavor	0.30	1.26	0.66	0.77	0.72	0.56	0.46	0.77	0.33	0.54	110d
5-Hepten-2-one, 6-methyl-	Wax flavored, pea flavored											
α-Bisabolol	Green	0.00	1.45	2.16	1.73	27.57	1.92	1.07	1.21	1.27	3.03	0.52d
1,6,10-Dodecatrien-3-ol, 3,7,11-trimethyl-	Phenolic	-	-	-	-	-	-	-	-	-	-	-
2(3H)-Furanone, 5-heptyldihydro-	-											
3-Hexen-1-ol, benzoate, (*Z*)-		0.00	30.76	22.02	23.71	24.58	27.70	24.68	22.15	19.29	26.39	1.9d
Benzyl alcohol		-	-	-	-	-	-	-	-	-	-	-
n-Valeric acid cis-3-hexenyl ester	Floral											
	-	0.00	0.00	0.87	0.00	0.00	1.23	0.00	0.00	0.00	0.00	9.7d
	-	2.14	0.22	0.29	0.27	0.08	0.25	0.05	0.11	0.05	0.20	50b
	Coconut, peach	-	-	-	-	-	-	-	-	-	-	-
	Fatty, green, citrus-like	0.00	0.01	0.01	0.01	0.01	0.01	0.00	0.01	0.01	0.01	2250d
	Spice, flower	0.00	0.00	2.21	2.26	0.00	2.93	3.28	0.00	0.00	0.00	2.1d
	Floral Green Waxy Citrus	-	-	-	-	-	-	-	-	-	-	-
	Woody	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	2000d
	-	-	-	-	-	-	-	-	-	-	-	-
	Fruity Sweet, flower											

[A] Odor Description were referred from websites(https://pubchem.ncbi.nlm.nih.gov/;http://www.flavornet.org/flavornet.html).'-', no odor description was found in the literature.

[B] rOAVs: odor activity value, the ratio of the concentration of a certain aroma component to its odor threshold.

[C] OT: odor threshold in water were obtained from: a, Sheng et al., 2021; b, Wang et al., 2022; c, Jose A. Moreno, Luis Zea, Lourdes Moyano, Manuel Medina, 2005; d, https://www.vcf- online.nl/VcfHome.cfm; e, https://www.chemicalbook.com.

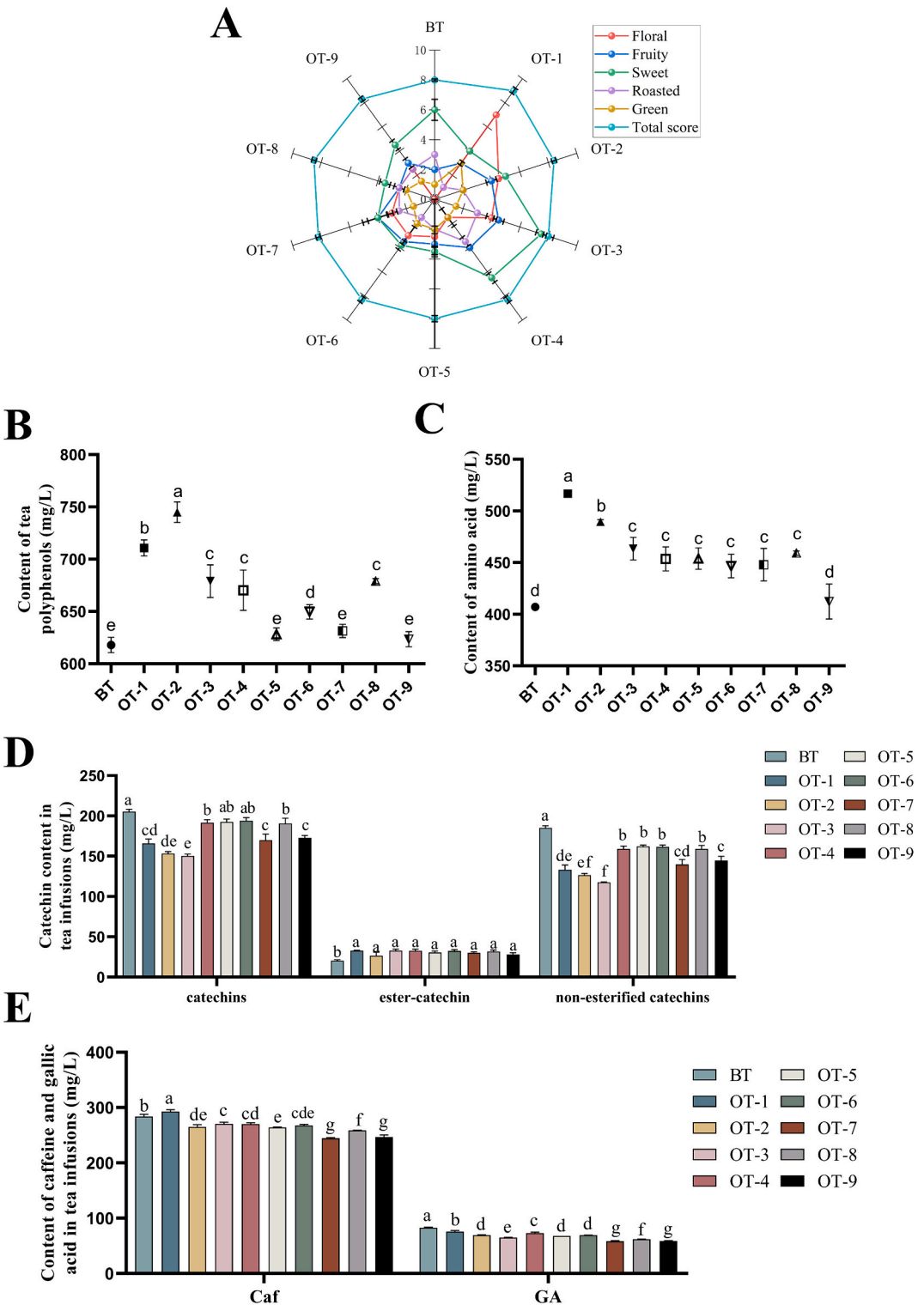

Fig. 3. Analysis of sensory and quality components of osmanthus black tea. (A) Radar Chart of Aroma. (B) Content of TPs. (C) Content of AAs. (D) Content of catechins. (E) Content of Citral. (F) Content of Caffeine and GA.

those in *O. fragrans*. At different temperatures, the collection time of 4 h was better in order to collect the main aroma substances of *O. fragrans*.

Only dihydro-β-ionone, β-cyclocitral, β-ionone, and α-ionone were detected in the OR group. It can be seen that the contents of dihydro-β-ionone and α-ionone were much lower than those in the fresh flowers of *O. fragrans* (Fig. S3B). With the exception of OR-1 and OR-4, the content of β-cyclocitral in the remaining 7 groups of the flower residues was lower than that in the fresh flowers. At the same time, the content of β-ionone in the OR group was higher than that in *O. fragrans* flowers. The content of the main aroma substances in the flower residue decreases with an increase in temperature, and found that the total content was lower than that in the fresh flowers of *O. fragrans* and lower than the concentration in the corresponding *O. fragrans* hydrolat samples.

In summary, it was found that the main aroma components in the hydrolats were higher than their corresponding *O. fragrans* residues, indicating that this method can effectively collect the characteristic aroma components in *O. fragrans*, so that the collected *Osmanthus fragrans* hydrolat has a natural *O. fragrans* fragrance. β-Cyclocitral was detected in the fresh flowers and residue of *O. fragrans*, but not in the hydrolats. It was speculated that β-cyclocitral was an oily substance, which could be dissolved in organic solvents, such as chloroform and dichloromethane. The main matrix of the hydrolats obtained under a low temperature vacuum environment was water (Zhang et al., 2010), so β-cyclocitral was not detected in the hydrolat samples. Because the collection rate of hydrolat was the highest under the conditions of 50 °C and 5 h, and the concentration of main characteristic substances in *O. fragrans* was highest, with obvious *Osmanthus fragrans* aroma, it has potential to provide scent for black tea.

3.4. Sensory evaluation of osmanthus black tea

The aroma of black tea is mainly sweet and roasted, while the aroma of black tea after scenting is mainly floral, fruity, and sweet. After scenting with the hydrolat, the floral aroma was significantly increased, and the fruity and floral characteristics were also enhanced. The overall aroma quality was best in OT-1 (Fig. 3A).

3.5. Analysis of TPs, AAs, catechins, GAs, and Cafs

The content of tea polyphenols in the osmanthus black tea infusions was higher than that of BT (Fig. 3B). The tea polyphenol content observed in OT-2 was the highest, reaching 744.92 mg/L, followed by OT-1 with a content of 710.79 mg/L, and BT was the lowest (only 617.94 mg/L). Among them, the tea polyphenol content of OT-1, OT-2, OT-3, OT-4, OT-6, and OT-9 was significantly higher than that of BT, so the taste of osmanthus black tea was slightly more bitter than that of BT.

With the exception of OT-9, the content of free amino acids in the other tea infusions was significantly higher than that of BT. Among them, the content of free amino acids in OT-1 was the highest, reaching 516.68 mg/L, followed by OT-2 (489.59 mg/L), and the content in BT was the lowest (only 407.00 mg/L) (Fig. 3C). Therefore, the taste of osmanthus black tea was more abundant and the entrance was fresh.

The catechin content in BT and the nine samples was detected by HPLC. Seven catechin components (GC, EGC, C, EGCG, EC, ECG and CG) were detected. The total content of catechins in the tea infusion of black tea was 205.31 mg/L, which was significantly higher than that of osmanthus black tea prepared with osmanthus hydrolat (Fig. 3D). The highest content of catechins in osmanthus black tea was OT-6, the highest content was 193.97 mg/L, and the lowest content of catechins was OT-1, which was 150.29 mg/L. The content of ester catechins (such as EGCG) with obvious bitterness and astringency was the lowest in BT, which was 20.31 mg/L, slightly lower than that in 9 kinds of osmanthus black tea. The highest content of ester catechins in osmanthus black tea was observed in OT-3 (32.73 mg/L) and the lowest content was observed in OT-9, which only contained 28.06 mg/L. Moreover, the content of non-esterified catechins (such as EC) with a sweet aftertaste was the highest in BT, reaching 185.03 mg/L, which was higher than that in 9 kinds of osmanthus black tea. The highest content of non-ester catechins in osmanthus black tea was OT-5, containing 162.14 mg/L, and the lowest content was OT-3, only 117.55 mg/L.

Caffeine, another bitter substance found in tea infusions, can be combined with other flavors to enhance the flavor (Lin et al., 2020). The content of caffeine in the tea infusions of osmanthus black tea was lower than that of BT with the exception of OT-1 (Fig. 3E). Among them, the caffeine content of OT-1 was the highest, reaching 292.71 mg/L, while the content of OT-7 was the lowest (244.42 mg/L). The content of gallic acid in the osmanthus black tea infusions was lower than that of BT. Among them, the highest content of gallic acid was observed in OT-1 with a content of 75.92 mg/L, and the lowest content observed in OT-7 (only 58.45 mg/L).

The osmanthus black tea obtained using the aroma-producing hydrolats had a higher content of tea polyphenols and ester catechins in the tea infusions, and the tea infusions exhibit a slightly bitter and astringent taste, but the content of free amino acids and non-ester catechins was significantly higher than that of black tea. The tea infusions taste fresh and sweet, and the taste was rich. The black tea was dried after spraying with the hydrolat. The drying process is a thermochemical process and some alcohols, aldehydes and esters will change their aroma. At the same time, bitter substances such as volatile ester catechins and caffeine will be cleaved, and the fruity and sweet aroma of black tea enhanced, improving the taste of the tea infusions (Xu et al., 2014).

3.6. Analysis of the aroma components in osmanthus black tea

In BT, a total of 82 volatile components were detected by GC–MS (Table S3), including 20 aldehydes, 18 hydrocarbons, 13 esters, 12 alcohols, 11 ketones, 4 oxygen-containing heterocyclic compounds, and 4 others. The total content of volatile compounds was 724.74 μg/L, and the proportion of aroma components was mainly alcohols (56.34%), followed by hydrocarbons (15.09%) and esters (11.26%) (Fig. 4A).

A total of 118 volatile compounds were detected in osmanthus black tea (Table S3), including 28 aldehydes, 24 alcohols, 22 esters, 22 hydrocarbons, 11 ketones, 6 oxygen-containing heterocyclic compounds and 5 other compounds. From the perspective of the total amount of volatile compounds (Fig. 4B), sample OT-1 has the highest content (1.07 mg/L), followed by OT-3 (1.03 mg/L), and OT-9 was the lowest (0.58 mg/L). It was found that the volatile compounds of osmanthus black tea scented using this method were mainly alcohols, followed by ketones, aldehydes, and oxygen-containing heterocyclic compounds (Fig. 4C).

Ketones are mainly derived from the oxidation of polyunsaturated fatty acids and amino acids (Huang et al., 2019), and generally have floral, fruit and vegetable, and creamy aromas. The ketones in the nine groups of tea samples were mainly β-ionone and dihydro-β-ionone. The highest content of β-ionone was observed in OT-3 (135.59 μg/L) and the lowest was OT-9 (only 66.21 μg/L); the highest content of dihydro-β-ionone was observed in OT-3, reaching 48.90 μg/L, and the lowest content was found in OT-9, which only contained 20.27 μg/L.

Related flavor studies have shown that aldehydes generally have fruity, sweet, and grassy flavors (Sheng, Lin, et al., 2021). The aldehydes in the nine groups of tea samples were mainly phenylacetaldehyde and 2-hexenal. The highest content of phenylacetaldehyde was observed in OT-6, reaching 88.37 μg/L, and the lowest was found in OT-9 (58.46 μg/L); the highest content of 2-hexenal was observed in OT-1, reaching 20.18 μg/L, and the lowest content was found in OT-9, which only contained 16.14 μg/L.

The common aroma characteristics of oxygen-containing heterocyclic compounds are nutty, caramel, and sweet (Scalone et al., 2019). The mainly oxygen-containing heterocyclic compounds in the nine groups of tea samples was trans-linalool oxide furanoid (woody and floral with camphor flavor). The content observed in OT-1 was the highest, reaching 183.92 μg/L, and the lowest was found in OT-9, containing 91.93

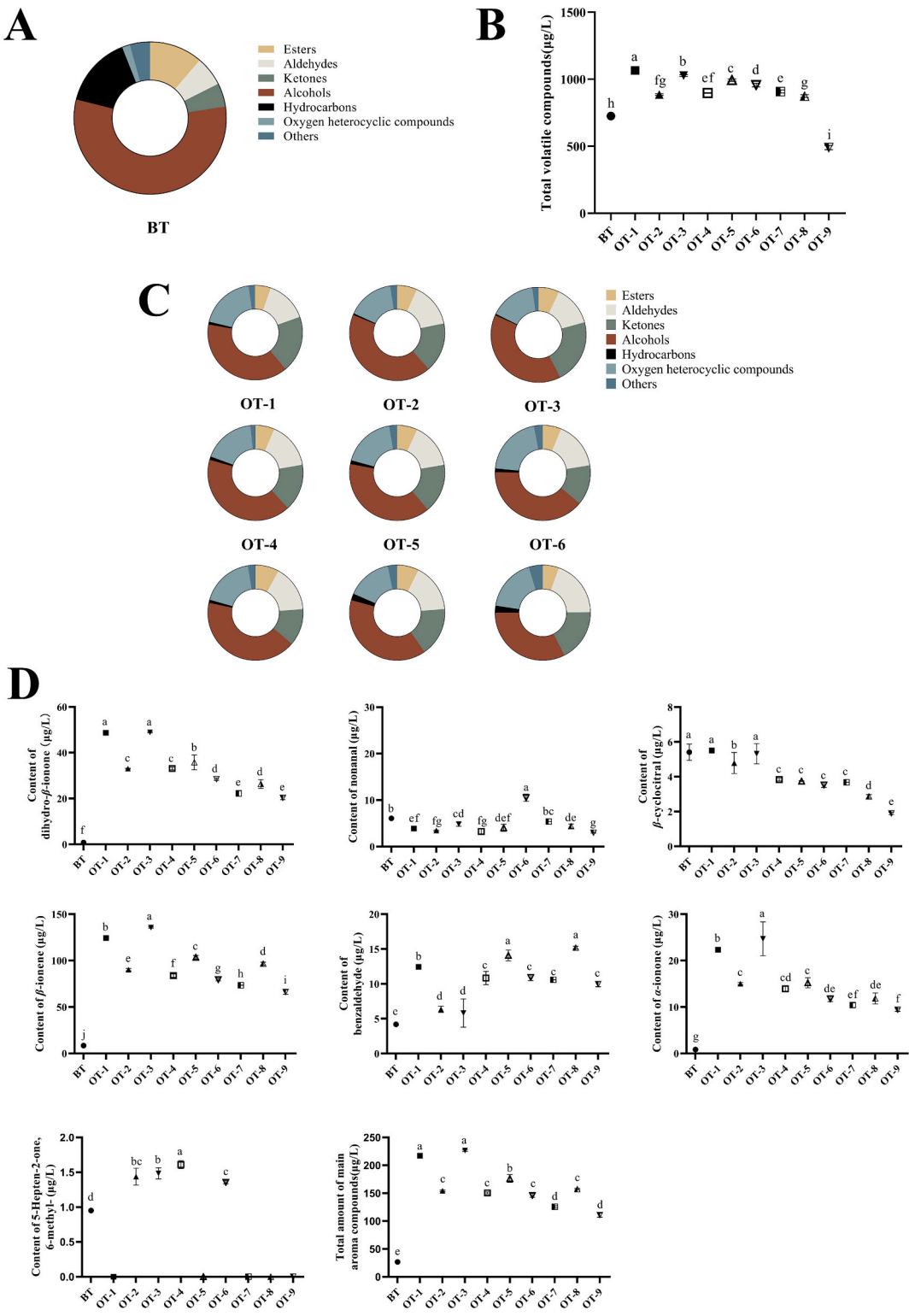

Fig. 4. Analysis of aroma components of osmanthus black tea. (A) The proportion of various volatile components of BT. (B) Total content of volatile components (C) The proportion of various volatile components of osmanthus black tea. (D) The content of 7 kinds of characteristic aroma components of *O. fragrans* in osmanthus black tea.

μg/L.

3.7. Comparison of characteristic aroma of O. fragrans

When combined with Section 3.3.3, it can be seen that the characteristic aroma components of *Osmanthus fragrans* are dihydro-*β*-ionone, nonanal, *β*-cyclocitral, *β*-ionone, benzaldehyde, *α*-ionone, and 6-methyl-5-hepten-2-one, seven volatile compounds. The contents of these seven substances in black tea scented by osmanthus hydrolat were analyzed (Fig. 4D). The contents of dihydro-*β*-ionone, *β*-ionone, benzaldehyde, and *α*-ionone in osmanthus black tea were significantly higher than those in BT, and the contents of *β*-cyclocitral and benzaldehyde

decreased with an increase in the scenting time and drying temperature. The total content of the main aroma components in the nine groups of osmanthus black tea was the highest in sample OT-3 with the highest total content of 226.60 μg/L, followed by OT-1 and OT-5 with a total content of 217.17 and 176.43 μg/L, respectively. The lowest content was observed in OT-9, with only 110.68 μg/L. The total content of the main aroma components in the nine groups of osmanthus black tea was 226.60 μg/L, followed by OT-1 and OT-5, with a total content of 217.17 and 176.43 μg/L, respectively. In general, the osmanthus black tea scented with osmanthus hydrolat exhibits the obvious characteristic aroma of osmanthus. The traditional scenting of scented tea assumes that the concentration difference between flowers and tea leaves will make the aroma and water molecules diffuse to the tea leaves together (Meng et al., 2024). There is a moisture gradient between the tea dhool and the hydrolat, and the aroma in the hydrolat will be transferred to the tea dhool to achieve the scenting effect. In addition to physical adsorption, the mechanism of aroma absorption of flower tea may also be accompanied by chemical adsorption, resulting in a relatively stable complex (Zhang et al., 2016).

Traditional osmanthus black tea is made from tea dhool and fresh flowers. When compared with previous research studies (Meng et al., 2024), under the premise of the same mass ratio of fresh leaves to fresh flowers, the content of the seven characteristic aroma components of osmanthus in traditional osmanthus black tea (scenting after redrying) was 455.35 μg/L, while the content of characteristic aroma components in osmanthus black tea scented with osmanthus hydrolat was only 226.60 μg/L. It can be found that the content of seven characteristic aromas in the collected osmanthus hydrolat can reach up to 5.73 mg/L. How to make more effective use of osmanthus hydrolat deserves more in-depth study.

4. Conclusions

In this study, the effects of different temperatures and times on the aroma of *O. fragrans* hydrolat were investigated under a vacuum environment. The aroma characteristics of the collected hydrolat and *O. fragrans* and *O. fragrans* residue were analyzed, respectively. The characteristic aroma components in *O. fragrans* were dihydro-*β*-ionone, nonanal, *β*-cyclocitral, *β*-ionone, benzaldehyde, *α*-ionone, and 6-methyl-5-hepten-2-one. The key differential aroma components of *O. fragrans* hydrolat were trans-linalool oxide furanoid, *β*-ionone, *α*-ionone, dihydro-*β*-ionone, phenylacetaldehyde, benzaldehyde, nonanal, methyl benzoate, and jasmonone. The hydrolat collected at 40 °C for 5 h and 50 °C for 5 h contained rich *O. fragrans* main aroma substances. Further analysis showed that the main aroma substances in the hydrolat of *O. fragrans* were higher than their corresponding *O. fragrans* residues. Therefore, this method can effectively collect floral hydrolat with the aroma characteristics of *O. fragrans*. Under the conditions of 50 °C for 5 h, the amount of hydrolat was the highest, and the concentration of the main characteristic substances of *O. fragrans* was highest with an obvious *O. fragrans* fragrance. Subsequently, the obtained osmanthus hydrolat was used to perfume black tea. Through the analysis of volatile components and the sensory, physical, and chemical quality, it was concluded that black tea billet adsorbs the osmanthus aroma in the hydrolat, and that this effectively improves the original aroma quality of black tea and gives it a rich osmanthus aroma. Unlike the low yield of flower essential oil, the hydrolat collected under these conditions has the advantages of natural flower fragrance, high yield, and low cost. These results provide insights for the development of other flower fragrance hydrolats, and can be applied to the development and production of other products.

CRediT authorship contribution statement

Xin Meng: Writing – original draft, Methodology, Data curation. **Fang Wang:** Validation. **Chao-Hong Fu:** Resources. **Lin Zeng:** Writing – review & editing. **Zhen-Hua Chen:** Validation. **Qizhen Du:** Writing – review & editing. **Zhi-Hui Feng:** Investigation. **Jun-Feng Yin:** Supervision. **Yong-Quan Xu:** Writing – review & editing, Project administration, Funding acquisition.

Declaration of competing interest

C-HF was employed by Pan'an ecological agriculture development Co., LTD. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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

Data availability

Data will be made available on request.

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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.2024.101564.

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