Temporal Variation of the Non-Volatile Compounds and Key Odorants in Xinyang Maojian Green Teas during the Spring and Autumn Seasons

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Abstract: Xinyang Maojian (XYMJ) green tea is one of the top ten teas in China, and the consumers prefer spring tea due to its umami taste and pleasurable aroma. However, the knowledge about temporal variation of the volatile compounds in XYMJ green teas harvested during different seasons is very limited. In the present work, the main non-volatile compounds that endowed the taste and volatile compounds responsible for the aroma in XYMJ green teas harvested during the spring and autumn seasons were determined. The average contents of free amino acids (FAA) were significantly higher and gradually declined in the spring teas, whereas the caffeine was significantly lower and gradually increased in the spring teas. A total of 39 volatile compounds of six chemical classes were detected in XYMJ green teas, and they displayed various change trends during the spring and autumn seasons, among which 15 volatile compounds were identified as the key odorants based on odor activity value (OAV). The highest OAV of 2195.05 was calculated for the violet-like smelling trans-β-ionone followed by decanal, nonanal, dimethyl sulfide, linalool, geraniol and naphthalene. The OAVs of geraniol, (Z)-3-hexenyl hexanoate, heptanal, benzaldehyde and hexanal in XYMJ spring teas were higher than XYMJ autumn teas. The hierarchical clustering analysis indicated that XYMJ green teas were divided into three clusters and the quality of XYMJ green teas changed greatly within spring season. Harvest season is a crucial factor affecting the flavor quality of XYMJ green teas.

Keywords: Xinyang Maojian (XYMJ) green tea; non-volatile compounds; volatile compounds; key odors; harvest season

1. Introduction

Tea is one of the most popular non-alcoholic beverages worldwide, and more than three billion people in more than 160 countries are fond of tea drinking due to its pleasant taste, attractive aroma and beneficial health properties [1–3]. The tender leaves of *Camellia sinensis* are used to produce tea, which were divided into six types including green, white, yellow, oolong, black and dark teas according to the processing methods and sensory qualities [4]. Green tea is minimally oxidized and non-fermented, the primary steps of green tea processing following harvest include post-harvest spreading, fixing, rolling, shaping and drying. Fixing is the key process step to determine the green tea quality. In China, green tea is always divided into the “spring tea”, “summer tea” and “autumn tea” according to the harvest season [5]. Generally, it is recognized that the quality of green tea harvested in spring is superior to that plucked in summer and autumn. Spring teas contain...
higher contents of umami-tasting free amino acids but lower levels of astringent-tasting flavonoids, whereas this phenomenon is converse for the summer and autumn teas [5,6]. Within the spring season, the quality of green tea changes greatly due to the fluctuation of metabolites in tender shoots [7]. Tea consumers always choose the harvest season as an important concern for purchasing green tea. Moreover, the spring green teas have a much higher price than the summer and autumn green teas [8].

The organoleptic quality and character of tea is determined by its taste, aroma, infusion color and appearance. Although aroma compounds account for less than 0.03% in tea dry weight, the score evaluating coefficient of aroma is 25% for the green tea sensory evaluation, which is just only below the taste (30%). There are more than 260 volatile compounds that have been identified in green tea, and about 30 of these identified volatile compounds contribute to the characteristic green tea aroma [9,10]. Parts of the volatile compounds come from the fresh tea leaves, while the other parts appear during the tea processing [9]. There are many factors that have effects on the tea aroma quality, such as cultivars, natural environment, elevation, processing technology and storage methods [1,11–14]. Among these factors, the harvest season is one of the important factors for tea aroma quality. However, the temporal variation of aroma compounds in spring and autumn green tea has been scarcely reported so far.

Grown and produced primarily in Henan province, Xinyang Maojian (XYMJ) green tea is one of the top ten teas in China, and it is distinguished for its umami taste and refreshing aroma [15]. Our previous studies showed that the quality and chemical constituents of XMYJ green teas were greatly affected by cultivars, elevation and production region, whereas the identified metabolites between manual and mechanical XMYJ teas were not significantly different [16,17]. However, the previous reports of XMYJ green teas mainly focused on the non-volatile chemical constituents [16,17]. The understanding on the temporal variation of the volatile compounds in XMYJ green teas is very limited. Headspace solid-phase microextraction (HS-SPME) combined with gas chromatography-mass spectrometry (GC-MS) has been widely applied to determine volatile compounds in teas [4,11,18–20]. In the present work, HS-SPME/GC-MS combined with odor activity value (OAV) method was adopted to identify the key odors responsible for XMYJ green teas during the spring and autumn seasons.

2. Materials and Methods

2.1. Samples and Chemicals

XYMJ green tea samples were collected from the core tea-producing area in Shihe County, Xinyang, Henan province of China. According to the XMYJ green tea processing procedures (spreading → fixing → rolling → drying), the fresh tea leaves with one leaf and one bud of *Camellia sinensis* cv. *Quntizhong* were randomly plucked on 24 March (XYMJ01), 3 April (XYMJ02), 10 April (XYMJ03), 20 April (XYMJ04), 15 September (XYMJ05), 26 September (XYMJ06), 8 October (XYMJ07) and 15 October (XYMJ08), 2020. The plucked fresh tea leaves were processed into XMYJ green teas at the corresponding sampling day. The former 4 samples are XMYJ spring teas, and the later 4 samples are XMYJ autumn teas. Due to the high production cost and low profit, XMYJ summer teas are hardly processed by the tea-farmers. Therefore, the XMYJ summer tea samples were not prepared in order to fit the actual situation of XMYJ green teas production. All tea samples for different production dates were collected by the quartering sampling method and packed into the aluminum foil bags, respectively, which were stored in the dark at −40 °C for further analysis.

(−)-Epigallocatechin gallate (EGCG, ≥99%), (−)-gallic acid (GA, ≥99%), (−)-catechin (C, ≥99%), (−)-epicatechin (EC, ≥99%), (−)-gallocatechin (GC, ≥99%), (−)-epicatechin gallate (ECG, ≥99%), (−)-gallocatechin gallate (GCG, ≥99%), (−)-epigallocatechin (EGC, ≥99%), glutamic acid and caffeine were purchased from Yuanye Bio-Technology Co., Ltd. (Shanghai, China). Analytical reagent grade Na₂HPO₄·12H₂O, KH₂PO₄ and ninhydrin were obtained from Beijing Chemical Works (Beijing, China). The SPME manual holder and the 50/30 μm divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS)
fiber were purchased from Supelco (Bellefonte, PA, USA). HPLC-grade acetonitrile, acetic acid and methyl alcohol were purchased from Tedia Company (Fairfield, OH, USA). Ethyl decanoate was purchased from J&K Scientific (Beijing, China). n-Alkanes (C5–C40) were purchased from Sigma-Aldrich (Shanghai, China). Ultrapure water was obtained from a Milli-Q water purification system (Merck Millipore, Billerica, MA, USA).

2.2. Equipment and Apparatus

The 7890A gas chromatograph and 5975C mass spectrometer (Agilent Technologies, Santa Clara, CA, USA), Waters e2695 HPLC system (Waters Corporation, Milford, MA, USA), TU-1901 ultraviolet-visible (UV-Vis) spectrophotometer (Puxi Technologies, Beijing, China), HHS-type thermostat water bath (Shanghai Boxun Industry & Commerce Co., Ltd., Shanghai, China), and Milli-Q water purifier (Billerica, MA, USA).

2.3. Analysis of Catechins, Caffeine, and Free Amino Acids

Determination of catechins and caffeine in XYMJ green teas was carried out by HPLC. Briefly, the ground XYMJ green tea sample of 0.200 g was added 5.0 mL of 70% methanol and incubated at 70 °C for 10 min while stirring twice (every 5 min). After cooling to room temperature, the sample was centrifuged at 1400 × g for 10 min. Next, the supernatant was transferred into a new 10 mL volumetric flask. Subsequently, we repeated the extraction and combined all the supernatants together. The total supernatant volume was up to 10 mL with 70% methanol. The extracts were further filtered through a 0.22-µm Millipore filter [ANPEL Laboratory Technologies (Shanghai) Inc., Shanghai, China]. The gradient was from solvent A to solvent B, and the linear gradient condition of the mobile phase was 0–10 min, 100% A; 10–25 min, 100–68% A; 25–35 min, 68% A; 35–45 min, 68–100% A; and 45–60 min, 100% A. The flow rate was 1 mL/min, and the detection was performed at 278 nm. Free amino acids content in XYMJ green tea samples were determined by the National Standard of P. R. China (GB/T 8314-2013)—Tea-Determination of free amino acids content.

2.4. Analysis of Tea Volatiles

According to the methodology for sensory evaluation of tea (GB/T 23776-2018—Methodology for Sensory Evaluation of Tea), 3.0 g XYMJ green tea samples were precisely weighted and placed into the 100 mL vial sealed with silicone septa and infused with 30 mL boiling water, and then 20.0 µL internal standard solution (ethyl caprate, 71 µg/g in anhydrous ethanol) was added immediately. The vial was placed in the 50 °C water bath to equilibrate for 10 min, and the SPME fiber was exposed for 50 min to the headspace while the sample was maintained at 50 °C. The fiber was removed and inserted immediately into the gas chromatography injector port for thermal desorption at 240 °C for 3 min. The gas chromatography apparatus was used to collect data.

The samples were analyzed by GC-MS (Agilent Technologies, Santa Clara, CA, USA) equipped with a HB-5MS column (30 m × 0.32 mm × 0.25 µm). Helium (>99.99%) was chosen as the carrier at a constant flow velocity of 1.0 mL/min. The column temperature was programmed as follows: 50 °C (hold 5 min) was increased to 180 °C at 3 °C/min, the column was held at 180 °C for 2 min, after which the temperature was increased 10 °C/min until it reached 250 °C (hold 3 min). The mass spectrum in the electron impact mode was generated at 70 eV; temperatures for injector and ion source were 240 °C and 230 °C, respectively. The chromatograms were recorded by monitoring the total ion currents in the 50–600 mass range. The volatile compounds were tentatively identified by comparing the National Institute of Standards and Technology (NIST) library (14.L) (a mass spectrum fitness of >90% was used as a criterion), retention indices (RIs) and retention times. RI values were calculated using n-alkanes (C5–C40) as the external references under identical experimental conditions. The concentrations of the volatiles were calculated in µg/L based on internal standard solution.
2.5. Odor Activity Value (OAV) Calculation

Odor activity value (OAV) is often applied to evaluate the contributions of aroma compounds. OAV was calculated using the equation $OAV = C/OT$, where $C$ was the concentration of the volatile compound and $OT$ was its odor threshold in water. Generally, compounds with $OAV \geq 1$ are regarded as potential contributors to the aroma profile [21].

2.6. Statistical Analysis

All the results were carried out in triplicate for the analytical determination. The analysis of significant differences between the samples was determined by one-way ANOVA (Duncan’s multiple range tests) using SPSS 20.0 (SPSS Inc., Chicago, IL, USA). The principal coordinate analysis (PCoA) and the hierarchical cluster analysis (HCA) of MS data were based on Bray–Curtis dissimilarities using vegan package in R (version 4.1).

3. Results and Discussion

3.1. Variation of Non-Volatile Compounds in XYMJ Green Teas

The non-volatile compounds endowed the taste of XYMJ green teas, which varied with the season (Table S1). The average contents of free amino acids (FAA), gallic acid (GA) and epicatechin gallate (ECG) were significantly higher in spring teas, while the average contents of caffeine (CAF) and catechin (C) were significantly lower in spring teas (Table 1).

As for epicatechin (EC), epigallocatechin (EGC) and epigallocatechin gallate (EGCG), there were no significant differences between XYMJ spring teas and autumn teas. The XYMJ spring teas has higher content of non-galloylated catechins, including EGC and EC, a finding consistent with the previous report [5]. During the spring season, FAA responsible for the umami taste gradually declined from 2.007% to 1.658%, the bitter caffeine and astringent EGCG increased from 3.317% to 3.738% and 4.527% to 6.790%, respectively (Table S1). The degrees of catechin galloylation were designed as ECG/(EC + ECG) and EGC/(EGC + EGCG), which were found to be inversely correlated to the cultivation altitude for oolong tea [22]. Although the degrees of catechin galloylation varied in XYMJ spring teas or autumn teas, there were no significant differences in XYMJ teas between spring and autumn season. Sweet aftertaste is a positive term to describe tea infusions and is often observed after XYMJ green tea drinking, which is attributed to the hydrolysis of galloylated catechins [3]. The non-volatile compounds were chosen as variables to perform the principal coordinate analysis (PCoA, Figure 1A) and hierarchical clustering analysis (HCA, Figure 1C). The clustering results of PCoA were evaluated by PERMANOVA in vegan and the results showed that the 8 XYMJ green teas were divided into three clusters (PERMANOVA $R^2 = 71.0\%$, $p < 0.001$). Cluster-1 consisted of the earlier spring teas XYMJ01 and XYMJ02, and Cluster-2 consisted of the later spring teas XYMJ03 and XYMJ04. The four XYMJ spring teas were clearly discriminated, while the four XYMJ autumn teas consisted of Cluster-3, indicating that the quality of XYMJ spring teas changed greatly within a single spring season.

Table 1. Contents of non-volatile compounds in XYMJ green teas (%).

| Compounds | XYMJ Spring Teas | | XYMJ Autumn Teas | |
|-----------|-----------------|-----------------|-----------------|------------------|
|           | Range | Average       | Range | Average       |
| FAA       | 1.658–2.007 | 1.826 ± 0.147 a | 1.619–1.750 | 1.691 ± 0.060 b |
| CAF       | 3.317–3.738 | 3.477 ± 0.197 b | 3.559–3.694 | 3.608 ± 0.062 a |
| GA        | 0.055–0.060 | 0.057 ± 0.003 a | 0.044–0.053 | 0.047 ± 0.004 b |
| EGC       | 1.979–2.466 | 2.178 ± 0.284 | 1.748–2.362 | 1.991 ± 0.274 |
| C         | 0.077–0.104 | 0.090 ± 0.011 b | 0.091–0.126 | 0.104 ± 0.015 a |
| EGCG      | 4.527–6.790 | 5.712 ± 1.107 | 5.724–5.871 | 5.820 ± 0.065 |
| EC        | 0.517–0.703 | 0.623 ± 0.093 | 0.531–0.696 | 0.604 ± 0.076 |
| ECG       | 1.681–1.893 | 1.793 ± 0.101 a | 1.620–1.770 | 1.703 ± 0.072 b |

Note: All data are expressed as mean ± S.D. ($n = 3$). Different letters in the same row indicate significant differences between mean values ($p < 0.05$, ANOVA, Duncan test).
3.2. Variation of Volatile Compounds in XYMJ Green Teas

The volatile compounds in XYMJ green teas were successfully determined by the HS-SPME and GC-MS. A total of 39 volatile compounds were tentatively identified, mainly including alcohols, aldehydes, esters, ketones, hydrocarbons and other compounds. Various change trends of the volatile compounds in XYMJ green teas were shown in the heat map (Figure S1), and the contents of identified volatile compounds in XYMJ green teas are listed in Table 2.

The dominant identified alcohol compounds in XYMJ green teas were geraniol, linalool, cis-nerolidol, δ-cadinol, 1-octen-3-ol and 3-decyn-2-ol. The total content of alcohol compounds decreased gradually in the spring season, while increased in the later autumn season. As shown in Figure 2, the contents of 1-octen-3-ol, 3-decyn-2-ol and linalool varied similarly in the spring season, and 1-octen-3-ol, 3-decyn-2-ol and δ-cadinol varied similarly in the autumn season. Among the identified alcohol compounds, terpenic alcohols, such as linalool, geraniol, cis-nerolidol and δ-cadinol, are the key aroma compounds for XYMJ green teas. The variation of geraniol displayed the same tendency with the total terpenic alcohols. The variation of linalool, geraniol and δ-cadinol presented increased tendency in the later autumn season, while cis-nerolidol decreased in the autumn season. The total content of terpenic alcohols decreased gradually in the spring season, while it increased in the later autumn season. Released from their corresponding glycoside precursors, the floral-fruity terpenic alcohols contribute significantly to the tea aroma due to their high contents and low thresholds [23].
Table 2. Contents of volatile compounds tentatively identified in XYMJ green teas (µg/L).

| No. | Retention Time (min) | CAS No. | Volatile Compounds | XYMJ Spring Teas Range | Average | XYMJ Autumn Teas Range | Average |
|-----|----------------------|---------|--------------------|------------------------|---------|------------------------|---------|
| 1   | 1.873                | 75-18-3 | dimethyl sulfide   | 0.57~3.98 2.66 b        | 8.05~11.95 9.06 a |
| 2   | 4.916                | 66-25-1 | hexanal            | 5.41~15.04 9.01 a       | 2.77~6.16 4.03 b |
| 3   | 8.688                | 111-71-7| heptanal           | 2.81~17.20 7.18 a       | 1.33~3.24 2.65 b |
| 4   | 11.359               | 100-52-7| benzaldehyde      | 5.25~10.60 7.08 a       | 3.90~5.59 4.91 b |
| 5   | 12.383               | 26001-58-1| 1-octen-3-ol | 1.07~3.62 2.04         | 1.06~3.40 2.08 |
| 6   | 12.631               | 585-25-1| 2,3-octanedione    | 2.04~4.46 2.64          | 3.35~3.90 3.61 |
| 7   | 12.774               | 110-93-0| (±)-β-pinene      | 0.0~5.04 1.92           | 0.0~0.99 0.49 |
| 8   | 12.869               | 66-25-1 | 6-methyl-5-heptene-2-one | 1.31~3.90 2.46     | 0.6~2.8 1.85 |
| 9   | 13.412               | 28973-97-9| (6Z)-farnesene | 0.0~5.33 2.04          | 1.22~5.78 3.18 b |
| 10  | 14.241               | 13466-78-9| 3-carene       | 1.44~6.40 4.36          | 4.34~7.75 6.39 |
| 11  | 16.921               | 127-91-3| 4-carene           | 0.0~3.56 0.89           | / 0.0 |
| 12  | 17.717               | 34995-77-2| Linalool oxide   | 0.0~5.33 2.69          | 2.07~3.22 2.81 |
| 13  | 18.34                | 874-41-9| 1,3-dimethyl-4-ethylbenzene | 0.0~3.43 1.58     | 0.0~0.54 0.17 |
| 14  | 18.545               | 124-19-6| nonanal            | 24.20~51.62 34.51       | 22.56~32.80 27.41 |
| 15  | 18.912               | 60-12-8 | phenylethyl alcohol | 0.0~3.27 2.30         | 0.88~4.25 2.56 |
| 16  | 21.875               | 39028-58-5| Linalool oxide   | 0.0~3.56 1.21          | 0.0~7.77 3.81 |
| 17  | 22.112               | 91-20-3 | napthalene         | 2.76~9.39 4.53          | 3.57~6.25 4.57 |
| 18  | 22.545               | 53398-84-8| trans-3-hexenyl butyrate | 6.67~23.70 14.69      | 5.85~16.20 9.08 |
| 19  | 22.783               | 5889-27-5| limonene           | 13.37~26.56 19.01 a     | 9.64~16.64 11.47 b |
| 20  | 23.412               | 13466-78-9| 3-carene       | 4.73~6.00 5.30          | 3.50~8.61 6.29 |
| 21  | 24.021               | 432-25-7| β-cyclocitrail    | 2.31~3.39 2.87          | 3.38~14.72 9.61 |
| 22  | 24.116               | 29050-33-7| (+)-4-carene   | 0.0~3.56 0.89           | / 0.0 |
| 23  | 24.679               | 629-62-9| pentadecane       | 6.67~22.02 13.04        | 7.27~14.07 9.26 |
| 24  | 25.731               | 34995-77-2| Linalool oxide   | 0.0~3.56 1.21          | 0.0~7.77 3.81 |
| 25  | 27.208               | 874-41-9| 1,3-dimethyl-4-ethylbenzene | 0.0~3.43 1.58     | 0.0~0.54 0.17 |
| 26  | 27.336               | 124-19-6| nonanal            | 24.20~51.62 34.51       | 22.56~32.80 27.41 |
| 27  | 29.831               | 91-20-3 | napthalene         | 2.76~9.39 4.53          | 3.57~6.25 4.57 |
| 28  | 31.283               | 13466-78-9| 3-carene       | 4.73~6.00 5.30          | 3.50~8.61 6.29 |
| 29  | 32.348               | 6378-65-0| hexanoic acid, hexyl ester | 2.96~11.08 7.17 a     | 2.15~3.76 3.29 b |
| 30  | 34.198               | 3879-26-3| cis-geranylacetone | 2.63~8.67 5.36 b       | 5.12~11.45 8.89 a |
| 31  | 35.522               | 142-50-7| trans-β-ionone    | 5.90~10.97 8.35         | 2.93~12.81 7.44 |
| 32  | 36.083               | 19435-97-3| δ-cadinol       | 13.80~19.73 17.58       | 12.30~15.22 13.15 |
| 33  | 37.552               | 629-92-5| nonadecane        | 6.67~22.02 13.04        | 7.27~14.07 9.26 |
| 34  | 38.957               | 483-76-1| δ-cadinol         | 6.67~22.02 13.04        | 7.27~14.07 9.26 |
| 35  | 40.792               | 34995-77-2| Linalool oxide   | 0.0~3.56 1.21          | 0.0~7.77 3.81 |
| 36  | 41.484               | 19435-97-3| δ-cadinol       | 6.67~22.02 13.04        | 7.27~14.07 9.26 |

Note: "/" Not detected; different letters in the same row indicate significant differences between mean values (p < 0.05, ANOVA, Duncan test).

Due to their relatively low odor threshold values, the aldehydes have been speculated to play important roles on the formation of clean aroma [24]. As shown in Figure 3, the content of aliphatic aldehydes, including hexanal, heptanal, nonanal and decanal, were much higher than the other aldehydes. During the spring season, the hexanal decreased from 15.04 µg/L to 5.41 µg/L, while the β-cyclocitrail increased from 2.31 µg/L to 3.39 µg/L. The aliphatic aldehydes and benzaldehyde presented a similar tendency in the autumn season. Hexanal usually represented green and grassy notes, β-cyclocitrail represented fruity aroma notes [25], the decrease of hexanal and the increase of β-cyclocitrail would contribute to the good XYMJ green tea aroma formation. The contents of hexanal and heptanal in XYMJ spring teas were higher (p < 0.05) than that in XYMJ autumn teas, while the contents of nonanal and decanal had no significant differences between the XYMJ spring and autumn teas.
Figure 2. Various change trends of the alcohols in XYMJ green teas. All data are expressed as mean ± S.D. (n = 3).

Figure 3. Various change trends of the aldehydes in XYMJ green teas. All data are expressed as mean ± S.D. (n = 3).
(Z)-3-Hexenyl hexanoate was the highest in XYMJ green teas followed by methyl 2-methylpentanoate, methyl salicylate, trans-3-hexenyl butyrate, cis-3-hexenyl-α-methylbutyrate and hexanoic acid, hexyl ester (Figure 4). (Z)-3-Hexenyl hexanoate was identified to endow fresh and clean odor to green teas [26]. With a pleasant fruity aroma, the content of methyl 2-methylvalerate ranged from 9.68 µg/L to 28.90 µg/L in XYMJ green teas, which was also found in bead-shaped green tea [18]. Liberated from its glycosides and giving a minty odor, methyl salicylate was recognized as an important volatile compound for the tea aroma formation [23]. (Z)-3-Hexenyl butyrate was ranged from 5.85 µg/L to 23.70 µg/L in XYMJ green teas, which has a fruity odor [27]. These esters had fresh and fruity aroma, which would be responsible for the clean aroma notes of the XYMJ green teas.

![Graph of various change trends of the esters in XYMJ green teas](image)

The variation of hydrocarbons in XYMJ green teas is shown in Figure 5. It is generally considered that saturated hydrocarbons, such as pentadecane, nonadecane and dodecane, have no or minor contribution to the tea flavor [28]. Naphthalene has pungent naphthyl odors [19], limonene was reported to give citrus flavor in tea leaves [29], δ-cadinene imparts spicy and woody aroma in tea leaves [30], and α-cubebene has an herby and waxy flavor [31]. δ-cadinene and α-cubebene presented the similar tendency during the spring and autumn seasons. Unsaturated hydrocarbons, including naphthalene, limonene, δ-cadinene and α-cubebene, would play an important role on the XYMJ green teas flavor formation.
During the spring season, trans-β-ionone (woody, violet) and cis-geranylacetone (floral, hay-like) increased gradually from 5.90 µg/L to 10.97 µg/L and 2.63 µg/L to 8.67 µg/L, respectively. The average content of cis-geranylacetone was higher ($p < 0.05$) in the autumn season than that in the spring season. It was reported that 2,3-octanedione had green and broccoli-like odor [32]. It is reported that dimethyl sulfide is a key volatile compound in the fresh green tea [33]. The aparagus-like dimethyl sulfide in XYMJ spring teas is significantly lower than that in XYMJ autumn teas. The animal-like indole in XYMJ spring teas was higher ($p < 0.05$) than that in XYMJ autumn teas. Indole is a common aroma-active compound in green tea [23], it was reported that the diluted indole had sweet and floral odor and strengthened the overall green tea odor over specific content ranges [34].

The identified common volatile compounds were used as variables to accomplish the PCoA (Figure 1B) and HCA (Figure 1D). The results showed that the 8 XYMJ green teas were divided into three clusters (PERMANOVA $R^2 = 49.5\%$, $p < 0.001$). Cluster-1 consisted of the earlier spring teas XYMJ01 and XYMJ02, Cluster-2 consisted of the later spring teas (XYMJ03, XYMJ04) and XYMJ08, and the other three XYMJ autumn teas made up Cluster-3. Different from choosing the non-volatile compounds as variables, the XYMJ08 was divided into Cluster-2. However, the results could also reveal that production seasons have effects on the aroma quality of XYMJ green teas.

### 3.3. Key Odorants in XYMJ Green Teas

The contribution of a particular volatile compound to the global aroma of XYMJ green teas depends on not only its content, but also on its odor activity value (OAV). It is generally considered that the compounds with an OAV ≥ 1 are great contributors to the aroma characteristics [21]. The higher the OAV of compounds is, the greater the contribution is. As listed in Table 3, a total of 15 volatile compounds with mean OAV > 1 were considered as the key odorants in the XYMJ green teas. The highest OAV of 2195.05 was calculated for the violet-like smelling trans-β-ionone followed by decanal (OAV = 57.94),
nonanal (OAV = 30.96), dimethyl sulfide (OAV = 19.53), linalool (OAV = 15.81), geraniol (14.37) and naphthalene (OAV = 10.34). The OAV of geraniol in XYMJ spring teas was higher ($p < 0.05$) than XYMJ autumn teas. (Z)-3-Hexenyl hexanoate, 1-octen-3-ol, benzaldehyde, cis-nerolidol, heptanal, hexanal, $\beta$-cyclocitrinal and 2,3-octanedione presented OAVs (10 < OAV > 1). Compared to the XYMJ spring teas, the OAVs of (Z)-3-hexenyl hexanoate, benzaldehyde, heptanal and hexanal were lower ($p < 0.05$) in XYMJ autumn teas. Thus, these compounds mentioned above were considered to be the major contributors to the aroma of the XYMJ green teas.

Mainly derived from the thermal degradation of $\beta$-carotene during the green tea manufacturing process, $\beta$-ionone with low odor threshold was considered the most important key odor based on the OAV calculation. $\beta$-Ionone is an essential volatile compound for the tea aroma formation and has been identified as a significant contributor to the tea aroma, including green tea [18,19,35], white tea [36,37], yellow tea [38], oolong tea [39,40], black tea [41,42] and dark tea [43–45]. The aldehydes, derived from the lipid oxidation, play an important role on the entire odor [24]. The OAVs of floral-fruity aldehydes (nonanal and decanal) were approximately 20–40 times higher than that of grassy or chestnut-like aldehydes (hexanal and heptanal), which would be helpful to the aroma quality of XYMJ green teas.

Dimethyl sulfide is one of the common aroma compounds in green tea, and it could be formed from its precursor methyl methionine sulfoxide salt upon the thermal decomposition [23]. It has been confirmed by the aroma recombination experiment that dimethyl sulfide had an important contribution to cooked corn-like aroma of green teas made by tea cultivar ‘Zhonghuang 1’ [20]. Dimethyl sulfide showed a high flavor dilution factor in high-grade matcha and had some connection with the quality of matcha [46]. Among the alcohol compounds, terpenic alcohols, including linalool, geraniol and cis-nerolidol, are the dominant odorants for the XYMJ green teas. Due to their high OAVs and floral-fruity aroma notes, the terpenic alcohols are beneficial to the aroma of the excellent-quality green tea. With pungent naphthyl odor, naphthalene was identified as the key odorants responsible for the chestnut-like aroma quality of green teas [19]. (Z)-3-hexenyl hexanoate was found to be not only abundant in its content, but also prominent in OAV (2.62). Formed from linoleic acid, 1-octen-3-ol (OAV = 2.06) presents a mushroom-like odor and was also identified as one of the key aroma compounds of Longjing tea [23,46]. Benzaldehyde (OAV = 2.00) is described as almond-like smell and $\beta$-cyclocitrinal (OAV = 1.25) contributes a fruity aroma.

### Table 3. The key odorants responsible for the XYMJ green tea aroma (OAV > 1).

| Odor-Active Compounds | Odor Characteristics ¹ | Odor Threshold ² (µg/L) | XYMJ Spring Teas Range (OAV) | XYMJ Autumn Teas Range (OAV) | Average (OAV) |
|-----------------------|------------------------|--------------------------|-----------------------------|-------------------------------|---------------|
| Dimethyl sulfide      | sweet, chestnut-like,  | 0.30                     | 1.90–13.25                  | 26.84–39.84                   | 19.53         |
|                       | cabbage-like           |                          |                             |                               |               |
| Hexanal               | green grass, tallow,   | 4.50                     | 1.20–3.34                   | 0.62–1.37                     | 1.45          |
|                       | fat                    |                          |                             |                               |               |
| Heptanal              | chestnut-like, citrus, | 3.00                     | 0.94–5.73                   | 0.44–1.08                     | 1.64          |
|                       | rancid                 |                          |                             |                               |               |
| Benzaldehyde          | almond-like smell      | 2.03                     | 1.75–3.53                   | 1.30–2.03                     | 2.00          |
| 1-Octen-3-ol          | mushroom               | 3.62                     | 1.07–3.40                   | 1.06–3.62                     | 2.06          |
| 2,3-Octanedione        | green flavor, broccoli-like | 2.52                  | 0.58–1.77                   | 1.33–1.55                     | 1.24          |
| Linalool              | floral, citrus, clean | 6.00                     | 12.95–17.20                 | 14.38–19.29                   | 15.81         |
| Nonanal               | fatty, green, sweet    | 1.00                     | 24.20–51.62                 | 22.56–32.80                   | 30.96         |
|                       | orange-like            |                          |                             |                               |               |
| Naphthalene           | tar, camphoric and     | 0.44                     | 6.28–21.33                  | 8.11–14.21                    | 10.34         |
|                       | greasy odor            |                          |                             |                               |               |
| Decanal               | floral-fatty odor,     | 0.10                     | 47.28–59.99                 | 35.02–86.06                   | 57.94         |
|                       | citrus                  |                          |                             |                               |               |
| β-cyclocitrinal       | citrus, lemon          | 5.00                     | 0.46–0.68                   | 0.68–2.94                     | 1.25          |
| Geraniol              | rose-like odor         | 7.50                     | 14.89–25.42                 | 3.89–18.14                    | 14.37         |
| (Z)-3-Hexenyl         | tender, fresh and      | 16.00                    | 1.31–5.23                   | 1.30–3.12                     | 2.62          |
| hexanoate             | clean                   |                          |                             |                               |               |
| trans-β-Ionone        | violet, sweet, floral  | 0.007                    | 1972.05–2819.05             | 1756.73–2174.90               | 2195.05       |
| cis-Nerolidol         | slight neroli-like,    | 10.00                    | 0.16–6.77                   | 1.23–2.23                     | 1.67          |

¹ Odor characteristics was referred from the website [47] and the literatures [18–20,32]. ² Odor threshold values were according to the reported references [18,19].
3.4. Comparison of Key Odorants in XYMJ and Other Green Teas

The identified key odorants in the XYMJ green teas had been reported in other green teas. Table 4 listed the top 10 key odorants in other green teas reported by previous studies. Compared to the other green teas, the differences are the OAVs of the key odorants and their potential contributions to the tea aroma. The various aroma-types, such as chestnut-like, cooker corn-like, orchid-like and clean aroma, might be resulted from the differences of the OAVs for volatile compounds in the green teas [18–20,48]. Table 4 also showed that the key odorants in the XYMJ green teas and other green teas almost belong to endogenous biosynthesis volatiles, including fatty acid derived volatiles (FADVs), amino acid derived volatiles (AADVs), volatile terpenes (VTs) and carotenoid derived volatiles (CDVs) [49,50]. It is reported that most FADVs presented a fresh or green odor. Seven of the fifteen key odors in the XYMJ green teas are FADVs, which may contribute to the formation of the refreshing or green aroma quality of XYMJ green teas. With pleasurable floral-fruity flavors, the VTs would form the superior aroma quality of XYMJ green teas. The Strecker aldehydes, including 2-methylpropanal, 2-methylbutanal, 3-methylbutanal, methional and phenylacetaldehyde, were formed by a reaction of their parent amino acids with α-dicarbonyl compounds during the green tea processing [23,51]. Belonging to the AADVs, these Strecker aldehydes were identified for contributing to the green tea aroma formation [23,51].

Table 4. The top 10 key odorants identified in the XYMJ and other green teas.

| Green Teas          | Top 10 Key Odorants Identified in Green Teas                                                                 |
|---------------------|---------------------------------------------------------------------------------------------------------------|
| Chestnut-like green tea [19] | trans-β-ionone (1617) d, 1-methylnaphthalene (241.6) c, 3-methylbutanal (105.2) b, nonanal (99.79) a, benzeneacetaldehyde (77.28) b, decanal (60.80) a, linalool (41.64) c, (E)-3-penten-2-one (22.34) a, heptanal (17.22) a, octanal (15.16) a |
| Fangping green tea [20] | dimethyl sulfide (1195.21) e, linalool (80.27) c, octanal (36.36) a, 1-nonanal (27.91) a, β-ionone (20.24) d, pentanal (15.58) a, 2-methylbutyraldehyde (12.87) b, benzaldehyde (11.98) b, ethyl acetate (10.98) c, α-ionone (6.72) d |
| Longjing tea [46] | β-ionone (20,406) d, 1-methylnaphthalene (7275) c, naphthalene (2347) c, 2,4-nonadienal (2022) c, (E)-2-nonenedial (1354) c, decanal (1100 a, 1-octen-3-ol (507) a, hexanal (417) a, (E,E)-3,5-octadien-2-one (397) a, 2-methylbutanal (390) b |
| Jingshan green tea [51] | dimethyl sulfide (458) c, (E,E)-2,4-heptadienal (46) a, 1-hexen-3-one (19) a, geraniol (17) c, methanethiol (14) b, (E,Z)-2,6-nonadienal (13) a, 3-methylnonane-2,4-dione (13) a, (Z)-1,5-octadien-3-one (12) a, (Z)-4-heptenal (11) a, linalool (11) c |
| Longjing tea [52] | dimethyl sulfide (440) c, (E,E)-2,4-heptadienal (50) a, methylpropanal (32) b, 3-methylbutanal (28) b, 3-methylnonane-2,4-dione (27) a, 1-hexen-3-one (22) a, (Z)-1,5-octadien-3-one (21) a, β-ionone (16) d, (Z)-4-heptenal (15) a, methanethiol (12) b |
| XYMJ green tea | trans-β-ionone (2195.05) d, decanal (57.94) a, nonanal (30.96) a, dimethyl sulfide (19.53) c, linalool (15.81) c, geraniol (14.37) c, naphthalene (10.34) c, (Z)-3-hexyl hexanoate (2.62) a, octan-3-ol (2.06) a, benzaldehyde (2.00) b |

Note: a: fatty acid derived volatiles (FADVs), b: amino acid derived volatiles (AADVs), c: volatile terpenes (VTs), d: carotenoid derived volatiles (CDVs) and e: others. The numbers in the brackets were the OAVs reported in the literatures.

4. Conclusions

In this work, non-volatile compounds endowed the taste and volatile compounds responsible for the aroma in XYMJ green tea harvested during the spring and autumn season were determined. The changes of the flavor compounds in XYMJ green teas are
analyzed and discussed. It is found that the flavor compounds differ between the spring and autumn tea samples and they showed various change trends. The average contents of FAA were significantly higher and gradually declined in the spring teas, whereas the caffeine was significantly lower and gradually increased in the spring teas. A total of 39 volatile compounds were determined by the HS-SPME combined GC-MS. The contents and types of volatile compounds displayed various change trends during the spring and autumn season, which confirms that the XYMJ green tea aroma formation is dynamic. There were 15 volatile compounds identified as the key odorants based on OAV, including trans-β-ionone, nonanal, dimethyl sulfide, linalool, geraniol, naphthalene, (Z)-3-hexenyl hexanoate, 1-octen-3-ol, benzaldehyde, cis-nerolidol, heptanal, hexanal, β-cyclocitral and 2,3-octanedione. The OAVs of geraniol, (Z)-3-hexenyl hexanoate, heptanal, benzaldehyde and hexanal in XYMJ spring teas were higher than XYMJ autumn teas. The XYMJ green teas harvested during the spring and autumn seasons have their key odorants and corresponding precursors, which were mainly derived from fatty acids, amino acids, glycosides and carotenoids. The types and OVAs of volatile compounds are different from the other green teas, which endow the unique aroma quality of the XYMJ green tea. The non-volatile and volatile compounds were chosen as the variables, respectively, to perform the hierarchical clustering analysis, and the results indicated that harvest season had important effects on the flavor quality of XYMJ green teas. The obtained findings of this study give a better understanding on the flavor compounds of XYMJ green teas harvested during the spring and autumn seasons, providing a scientific basis for the tea flavor formation and the quality control of XYMJ green tea production.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/agronomy12051085/s1, Table S1: Contents of free amino acids (FAA), caffeine (CAF) and catechins in XYMJ green teas (%); Figure S1: Heat map of the contents of volatile compounds in XYMJ green teas.

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