

Comprehensive Evaluation of Quality of *Camellia semiserrata* Seed Oils from Various Harvest Dates

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Abstract: The ripening degree of camellia fruit is one of the key factors affecting the quality of camellia seed oil. In this study, taking *Camellia semiserrata* as the research object, the oil content, physicochemical indexes, nutritional indexes, fatty acid composition, and volatile compounds of camellia seed oils from various harvest dates (from September to October) were determined. The results showed that with the increase of the ripening degree of camellia fruit, the oil content of camellia seed increased at first and then decreased and reached the highest (58.74%) on September 30, while the acid value, peroxide value, β -sitosterol, α -tocopherol, and polyphenols of camellia seed oil showed a downward trend. Among them, the highest contents of β -sitosterol, α -tocopherol, and polyphenols were observed on September 2, which were 6881.60, 311.34, and 78.08 mg/kg, respectively. In terms of the fatty acid composition of camellia seed oils, the content of oleic acid increased at first and then decreased, the content of linoleic acid and palmitic acid decreased gradually, while the content of stearic acid increased gradually. A total of 37 volatile compounds were identified in different samples, including 12 aldehydes, 5 ketones, 12 alcohols, 2 acids, 5 esters, and 1 other. With the increase of the ripening degree, the concentration of aldehydes and alcohols increased at first and then decreased, the concentration of ketones and esters decreased gradually, but the concentration of acid compounds had no obvious rule. In addition, the camellia seed oils from various harvest dates were classified and comprehensively evaluated by principal component analysis and grey relation analysis. The results showed that different camellia seed oils could be divided into three groups, and the comprehensive score of camellia seed oils on September 30 was the highest. In general, this work can provide theoretical guidance for the harvest date of *Camellia semiserrata*.

Key words: *Camellia semiserrata* seed oil, harvest date, quality index, fatty acid composition, volatile compound

1 Introduction

Camellia oleifera Abel. is a woody oil crop endemic to China, which is known as the four major woody oil crops in the world together with olive, palm, and coconut¹. *Camellia semiserrata* is a special camellia variety, which has the characteristics of fast growth, large tree body, high yield, and long high yield period. Compared with *Camellia oleifera*, it has larger fruit, higher seed oil content, and lighter oil color. Moreover, *Camellia semiserrata* has a long flowering period and red or pink flowers, so it is a very ornamental garden tree species. Due to the high economic, social, ecological, and ornamental value of *Camellia semiserrata*, it is of great significance to the development of *Camellia semiserrata* resources².

Camellia seed oil (CSO) is a vegetable oil extracted from

camellia seed, which is popular because it is rich in mono-unsaturated fatty acids and active functional components (sterol, VE, polyphenols, squalene, etc.)³. The ripening degree of oil fruit is one of the key factors affecting the comprehensive quality of vegetable oil⁴. During the ripening of oil fruit, the oil content gradually accumulates under the regulation of the oil synthesis pathway. When studying the Huashuo and Huajin *Camellia oleifera*, Zhang *et al.* found that the oil content of camellia seeds increased gradually with the increase of ripening degree of camellia fruit⁵. The fatty acid composition of vegetable oils also varies with ripening degree. Previous studies have shown that with the increase of ripening degree of olive fruit, the unsaturated fatty acids content in olive oil increased significantly, while the saturated fatty acids content decreased

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significantly⁶⁾. However, palm oil showed different rules that the contents of saturated fatty acids and monounsaturated fatty acids increased during ripening, while polyunsaturated fatty acids content decreased during ripening⁷⁾. In terms of active functional components, a higher content of them can often be observed in the early harvest periods of oil fruit. For example, the content of phenolic compounds in palm oil decreased significantly with the increase of ripening degree⁸⁾. Huang *et al.* also found that the content of total phenols and total flavonoids in coratina olive oil in earlier harvest periods was higher⁹⁾. In addition, affected by the lipoxygenase pathway, fatty acid degradation, and microbial action, the flavor profile of vegetable oil often changes significantly with the change of ripening degree of oil fruit. There have been many studies about the effect of ripening degree on olive oil flavor were reported^{10–12)}.

At present, the studies of the CSO quality are mostly focused on the processing methods^{13, 14)}, but there are few studies on the harvest date. Our research group studied the changes of quality indexes of *Camellia oleifera* (Cenruan 2 and Cenruan 3) seed oil from different harvest dates (from August to November in 2013 and 2014) and obtained the best harvest dates of Cenruan 2 and Cenruan 3 were November 15 and October 26, respectively. However, there is no study on the effect of the ripening degree of camellia fruit on the comprehensive quality of *Camellia semiserrata* seed oil. Variety is an important factor affecting the harvest date of camellia fruit. However, the study about the harvest date of *Camellia semiserrata* has not been reported. Therefore, in this study, the oil content, physicochemical indexes, nutritional indexes, fatty acid composition, and volatile compounds of *Camellia semiserrata* seed oils from various harvest dates were determined, to reveal the change rule of each quality index during the ripening process of camellia fruit. Additionally, the quality of different CSOs was comprehensively evaluated by principal component analysis (PCA) and grey relation analysis (GRA), to obtain the best harvest date of *Camellia semiserrata* fruit and provide comprehensive reference information for the growers of this camellia variety.

2 Materials and Methods

2.1 Materials

Camellia semiserrata fruits were harvested from the Zhangmutou Forest Farm (22° 48' -22° 48' N, 113° 53' -114° 53' E; Dongguan, Guangdong, China). This region has a south subtropical monsoon climate and is characterized by high temperature and rainy weather. The average annual temperature is 21.8°C and the average annual rainfall is 1790 mm. The rainy season is mainly from April to September, the average annual relative humidity is 79%, and the average frost-free period is 350 days. Additionally, this region is a gently hilly area with an altitude of between 100 m and 400 m. According to the previous experience, this study designed several harvest dates for *Camellia semiserrata* fruits in September and October. The specific harvesting dates were shown in Table 1. The healthy and undamaged camellia seeds were dried to a moisture content of 4%-7%. Crude oils were obtained by pressing extraction using a household screw press (ZYJ-904, Belston Co., Ltd., Berlin, Germany). Subsequently, the crude oils were centrifuged for 15 min at 6790 g to obtain the CSO samples.

2.2 Chemicals

Fatty acid methyl ester standard mixture and n-alkanes (C8-C30) were HPLC grade and purchased from Sigma-Aldrich (St. Louis, MO, U.S.A.). β -Sitosterol, α -tocopherol, and gallic acid were HPLC grade and purchased from Yuanye Biotechnology Co., Ltd. (Shanghai, China). Folin-Ciocalteu reagent (analytical grade) and O-dichlorobenzene (HPLC grade) were purchased from Macklin Biochemical Co., Ltd. (Shanghai, China). The other chemicals were purchased from Guangzhou Chemical Reagent Factory (Guangzhou, China).

2.3 Oil content

According to GB5009.6-2016¹⁵⁾, the Soxhlet extraction method was used to determine the total oil content in camellia seeds of different harvest dates.

2.4 Physicochemical indexes

The determination of acid value (AV) and peroxide value (POV) of CSOs were according to GB 5009.229-2016¹⁶⁾ and GB 5009.227-2016¹⁷⁾, respectively.

Table 1 Harvest dates and codes of *Camellia semiserrata* fruits.

Harvesting date (September)	code	Harvesting date (October)	code
2021.9.2	D1	2021.10.10	D6
2021.9.9	D2	2021.10.20	D7
2021.9.16	D3	2021.10.30	D8
2021.9.23	D4		
2021.9.30	D5		

2.5 Nutritional indexes

2.5.1 β -sitosterol

The β -sitosterol content in CSOs was determined by the ultraviolet spectrophotometry method. Determination: 1g CSO was weighed, and the volume was fixed to 10 mL by chloroform. Subsequently, a 2 mL sample solution was mixed with 8 mL acetic anhydride and 5 drops of concentrated sulfuric acid. After 45 min, the absorbance was measured at 662 nm by a UV-5200 spectrophotometer (Yuanxi instrument Co., Ltd., Shanghai, China). Standard curve drawing: A standard curve was drawn using different concentrations of β -sitosterol and the linear regression equation was obtained as $Y = 2.4102X + 0.0033$ ($R^2 = 0.999$). The β -sitosterol content in CSOs was calculated according to the linear regression equation.

2.5.2 Polyphenols

The polyphenols content in CSOs was determined by Folin-Ciocalteu method¹⁸). Firstly, polyphenols were extracted from CSO (3.0 g) with 5 mL 80% methanol three times. Determination: 3 mL Folin-Ciocalteu reagent was added to 1 mL sample solution. After 10 min, 2 mL of 7.5% sodium carbonate was added, and distilled water was added to bring the total volume to 10 mL. After a dark reaction of 15 min, the absorbance was measured at 765 nm. Standard curve drawing: A standard curve was drawn using different concentrations of gallic acid and the linear regression equation was obtained as $Y = 0.017X - 0.0104$ ($R^2 = 0.999$). The polyphenols content in CSOs was calculated according to the linear regression equation.

2.5.3 α -Tocopherol

The α -tocopherol content in CSOs was determined by the ultraviolet spectrophotometry method. Determination: 1g CSO was weighed, and the volume was fixed to 10 mL by anhydrous ethanol. Then, 1 mL sample solution was mixed with 1 mL ferric chloride solution and 2.5 mL 2,2-bipyridine solution. After a dark reaction of 10 min, 0.5 mL phosphoric acid solution was added, and anhydrous ethanol was added to bring the total volume to 10 mL. Subsequently, the absorbance of samples was measured at 520 nm. Standard curve drawing: A standard curve was drawn using different concentrations of α -tocopherol and the linear regression equation was obtained as $Y = 4.962X - 0.0067$ ($R^2 = 0.996$). The α -tocopherol content in CSOs was calculated according to the linear regression equation.

2.6 Fatty acid composition analysis

According to GB 5009.168-2016¹⁹), the fatty acid composition of CSO samples was analyzed by gas chromatography (7890B, Agilent Technologies, USA) equipped with a DB-23 column (60 m $\times$ 0.25 mm $\times$ 0.15 μ m). Fatty acid methyl esterification treatment: the oil sample (60 mg) was thoroughly mixed with 4 mL isooctane and 200 μ L 2mol/L potassium hydroxide-methanol solution. The mixture was thoroughly vortexed for 30 s. Subsequently, 1 g sodium bi-

sulfate was added to the solution to neutralize the excess potassium hydroxide. After the salt was precipitated, the supernatant was used for analysis by gas chromatography.

Gas chromatographic conditions: the injection volume was 1 μ L at a split ratio of 25:1 and the carrier gas was nitrogen; the initial oven temperature was 50°C and held for 1 min, then increased to 150°C with 25°C/min and held for 4 min, then increased to 180°C with 2°C/min and held for 4 min, then increased to 200°C with 3°C/min and held for 4 min, and finally increased to 230°C with 15°C/min and held for 5 min. Additionally, the injector and detector temperatures were 250°C and 280°C, respectively. The fatty acid composition was identified by comparison of retention times to the standard of fatty acid methyl ester. The quantitative analysis was performed by using the area normalization method.

2.7 Volatile compounds analysis

The volatile compounds in CSOs were identified by headspace-solid phase microextraction-gas chromatography-mass spectrometry (HS-SPME-GC-MS). The specific determination steps and conditions were the same as those previously studied by our research group²⁰). Briefly, quantitative analysis of volatile compounds was performed by using O-dichlorobenzene as the internal standard. In addition, retention indices (RI) of volatile compounds were calculated using n-alkanes (C8-C30) as the external standard. Qualitative analysis of volatile compounds was carried out by comparing the calculated RI with the reference RI.

2.8 Statistical analysis

One-way analysis of variance (ANOVA) was performed by SPSS 26 (IBM Watson Analytics, Beijing, China). Origin 2021 (Origin Lab Corporation, Northampton, MA) was used to perform PCA and draw figures. Microsoft Excel 16.0 was used to perform GRA.

The GRA referred to the method of Nie *et al.*²¹). The first step was to set the reference sequence and carry out non-quantitative processing of the sample data; the second step was to calculate the absolute difference between each sample sequence and the reference sequence, and to obtain the maximum and minimum sample absolute difference; the third step was to calculate the grey relation coefficient; the fourth step was to calculate the grey relation degree of each sample.

The specific calculation formula of the absolute difference was as follows:

$$\Delta_i(k) = |X_0(k) - X_i(k)|$$

$\Delta_i(k)$: absolute difference; $X_0(k)$: reference sequence; $X_i(k)$: sample sequences; i : the number of samples; k : the number of indexes.

The specific calculation formula of the grey relation coefficient was as follows:

$$\xi_i(k) = \frac{\min \Delta_i(k) + \rho \max \Delta_i(k)}{\Delta_i(k) + \rho \max \Delta_i(k)}$$

$\xi_i(k)$: correlation coefficient; $\min \Delta_i(k)$: minimum absolute difference; $\max \Delta_i(k)$: maximum absolute difference; ρ : resolution coefficient, $\rho = 0.5$.

The specific calculation formula of the grey relation degree was as follows:

$$R_k = \frac{1}{n} \sum_{k=1}^n \xi_i(k)$$

R_k : grey relation degree; n : total number of indexes.

3 Results and Discussion

3.1 Basic quality indexes of *Camellia semiserrata* seed oils from various harvest dates

3.1.1 Oil content

Camellia semiserrata is a camellia variety with high oil

content. The oil content of camellia seeds is often used to evaluate the harvest date of camellia fruit. Fig. 1A shows that at different harvest dates, the oil content of camellia seeds ranged between 47.11% and 58.74, with a trend of increasing and then decreasing. Specifically, it increased over time between D1 (September 2) and D5 (September 30) (from 47.11% to 53.35%) and reached its highest value on D5 (30 September). This is probably because the soluble sugar and starch content of the seeds are consumed during the ripening of the camellia fruit, thus providing energy to promote oil accumulation⁵⁾. The oil content of camellia seeds then decreased, but there was no significant difference in the oil content of camellia seeds harvested on D6 (October 10), D7 (October 20), and D8 (October 30). In addition, based on data on CSO content, D1-D4 can be classified as early maturity stage, D5 as full maturity stage, and D6-D8 as late maturity stage. In conclusion, the best harvest date to obtain camellia fruit with a high oil content

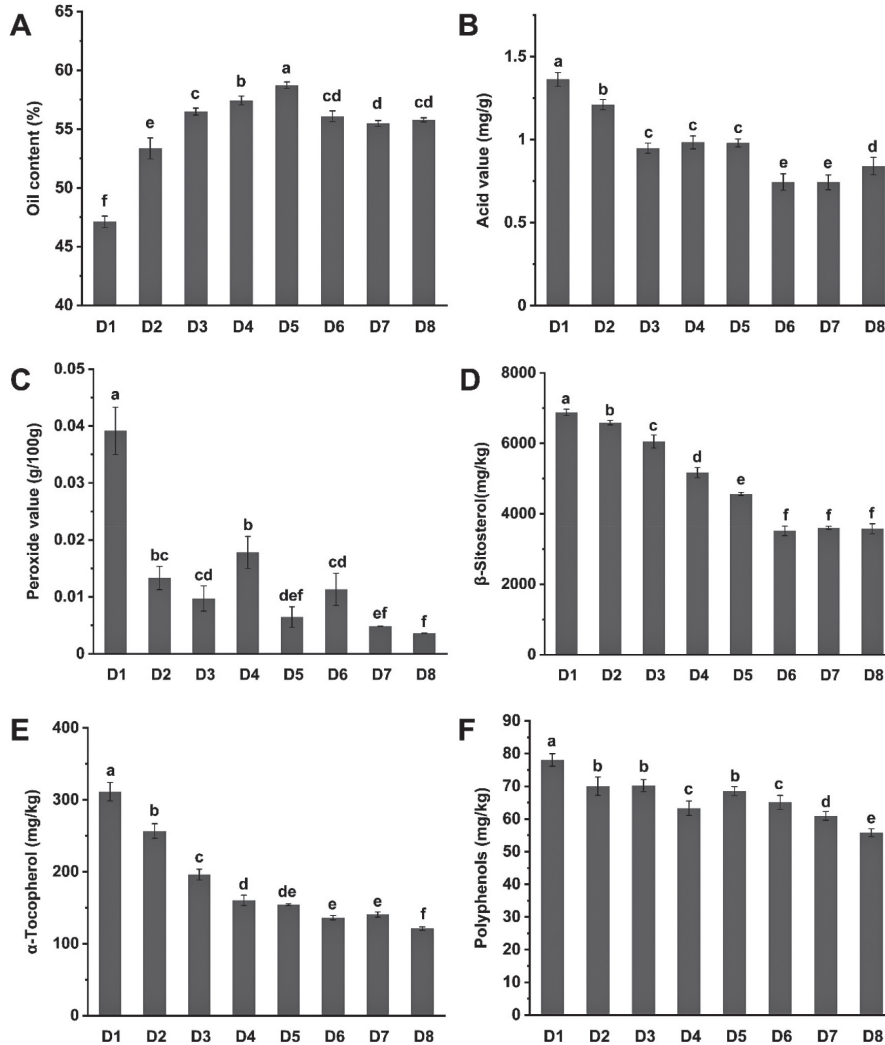


Fig. 1 The oil content, physicochemical indexes, and nutritional indexes of *Camellia semiserrata* seed oils from various harvest dates.

should be around September 30.

3.1.2 Physicochemical indexes

Acid value and peroxide value are important physicochemical indexes to evaluate the quality of CSO. According to the Chinese national standard GB/T11765-2018²²⁾, the acid value of camellia seed crude oil should be less than or equal to 4 mg/g, and the peroxide value should be less than or equal to 0.25 g/100 g. As shown in **Figs. 1B** and **1C**, the acid and peroxide values of the CSO harvested on different dates were in accordance with national standards. In addition, harvest date had a significant effect on the CSO acid and peroxide values, as shown by their progressive decrease with increasing harvest date, during the period D1-D8. This result is similar to the study by Lv *et al.* who showed that the free acidity and peroxide value of Longnan olive oil decreased with the increase of ripening degree of olive fruit⁴⁾. The CSO on D1 had the highest acid value and peroxide value, which were 1.36 mg/g and 0.039 g/100 g, respectively, while the CSO on D7 had the lowest acid value (0.74 mg/g) and the lowest peroxide value (0.0036 g/100 g) on D8. The lower the acid and peroxide values, the higher the oil quality. Therefore, a later harvest date is favorable for improving the physicochemical quality of CSO.

3.1.3 Nutrition indexes

To explore the effect of harvest date on the nutritional indexes of CSO, the contents of β -sitosterol, tocopherol, and polyphenol of CSO were determined. As shown in **Fig. 1D**, the harvest date had a significant effect on the β -sitosterol content of CSO, and the β -sitosterol content decreased with the increase of ripening degree of camellia fruit. The β -sitosterol content of CSO from various harvest dates was between 3516.00 mg/kg and 6881.60 mg/kg, and the CSO of D1 had the highest β -sitosterol content. During D1 to D6, the β -sitosterol content of CSO decreased significantly. However, the β -sitosterol content of CSO did not change significantly during D6 to D8. Sönmez *et al.* determined the β -sitosterol content of Memecik and Ayvalık olive oil from different harvest dates and found that the β -sitosterol content decreased with the increase of fruit maturity²³⁾. **Figure 1E** showed the α -tocopherol content of CSO from various harvest dates. Like the change of β -sitosterol, the α -tocopherol content of CSO was also decreased with the increase of ripening degree of camellia fruit. The α -tocopherol content of CSO of D1 was the highest (311.34 mg/kg), while that of D8 was the lowest (120.11 mg/kg). The α -tocopherol content of CSO decreased rapidly during D1 to D4 and relatively slowly during D4 to D8. Similarly, the polyphenols content of CSO on the earlier harvest dates was higher than that of CSO on later harvest dates (**Fig. 1F**). During D1-D8, the polyphenols content of CSO decreased significantly, from 78.08 mg/kg to 55.79 mg/kg. Similar results had been found in coratina olive oil that the total phenol content decreased with the increase of olive fruit ripening degree⁹⁾.

In short, the harvest date had a great effect on the nutritional indexes of CSO, and the nutrient content decreased with the increase of the ripening degree of camellia fruit. This may be because various physiological metabolic processes consumed these nutrients or the precursors that synthesized these nutrients during the ripening of camellia fruit, resulting in the decrease of nutrient content in CSO. For example, the metabolism of sugars, lipids, and amino acids would consume phosphoenolpyruvate and erythritose-4-phosphate during the ripening, which were precursors of phenolic compounds⁴⁾. Furthermore, the activity of the enzyme was also an important factor affecting the nutrient content. Previous studies have reported that the activities of β -glucosidase, polyphenol oxidase, and peroxidase also affected the content of phenolic compounds¹⁸⁾. Before the full ripening of camellia fruit (D1-D5), the nutrient content of CSO decreased rapidly, while after the full ripening of camellia fruit (D5-D8), the nutrient content of CSO changed slowly. This may be related to the metabolic rate of various physiological metabolic processes during the camellia fruit ripening. If the harvest date of camellia fruit was evaluated by nutritional indexes, the CSO with higher nutrient content could be obtained on the earlier harvest dates.

3.2 Fatty acid composition and volatile compounds of *Camellia semiserrata* seed oils from various harvest dates

Analysis of the data on the basic indexes of CSO from different harvest dates revealed that there was that there were no significant differences between D6, D7, and D8. Therefore, only the D7 group was selected for the determination of the fatty acid composition and volatile compounds of CSO in the following experiments.

3.2.1 Fatty acid composition

The fatty acids are not only vital components of CSO, but also the key quality parameter to evaluate the quality and authenticity of CSO²⁴⁾. The fatty acid composition of *Camellia semiserrata* seed oil from various harvest dates is shown in **Table 2**. A total of 16 fatty acids were identified in different CSOs, including 8 saturated fatty acids (SAFA), 5 monounsaturated fatty acids (MUFA), and 3 polyunsaturated fatty acids (PUFA). The SAFA content of CSOs from various harvest dates was between 10.251% and 11.199%. Except for stearic acid, the contents of the other 7 SAFAs showed a downward trend with the increase of ripening degree of camellia fruit. On the contrary, the stearic acid content increased significantly with the increase of the ripening degree of camellia fruit. During D1-D7, the palmitic acid content decreased from 8.201% to 7.214%, while the stearic acid content increased from 2.691% to 3.150%. The MUFA content of CSOs from various harvest dates was between 79.730% and 81.400%. Oleic acid was the most important MUFA in CSOs from various harvest dates, and

Table 2 The fatty acid composition of *Camellia semiserrata* seed oils from various harvest dates.

Fatty acid composition ^a	Content (%) ^b					
	D1	D2	D3	D4	D5	D7
Myristic acid (C14:0)	0.073 ± 0.001 a	0.071 ± 0.001 a	0.061 ± 0.001 b	0.045 ± 0.001 c	0.047 ± 0.001 c	0.038 ± 0.000 d
Pentadecanoic acid (C15:0)	0.008 ± 0.000 a	0.007 ± 0.000 b	0.008 ± 0.000 a	—	—	—
Palmitic acid (C16:0)	8.201 ± 0.005 a	8.172 ± 0.007 b	8.017 ± 0.005 c	7.241 ± 0.002 e	7.281 ± 0.016 d	7.214 ± 0.012 f
Palmitoleic acid (C16:1n7)	0.099 ± 0.001 a	0.099 ± 0.000 a	0.093 ± 0.002 b	0.067 ± 0.002 c	0.067 ± 0.002 c	0.058 ± 0.000 d
Margaric acid (C17:0)	0.065 ± 0.001 a	0.064 ± 0.000 a	0.065 ± 0.002 a	0.048 ± 0.001 c	0.056 ± 0.000 b	0.055 ± 0.000 b
Stearic acid (C18:0)	2.691 ± 0.002 d	2.651 ± 0.003 e	2.617 ± 0.003 f	2.793 ± 0.002 c	2.852 ± 0.022 b	3.150 ± 0.004 a
Oleic acid (18:1n9c)	79.016 ± 0.010 e	78.992 ± 0.033 e	79.671 ± 0.011 d	80.744 ± 0.008 a	80.003 ± 0.047 c	80.241 ± 0.026 b
Linoleic acid (18:2n6c)	8.852 ± 0.003 b	8.956 ± 0.017 a	8.463 ± 0.007 d	8.173 ± 0.015 f	8.818 ± 0.024 c	8.385 ± 0.023 e
Arachidic acid (C20:0)	0.069 ± 0.000 a	0.062 ± 0.001 b	0.067 ± 0.001 a	0.056 ± 0.001 d	0.057 ± 0.000 d	0.060 ± 0.002 c
Eicosenic acid (C20:1)	0.544 ± 0.001 b	0.551 ± 0.008 b	0.574 ± 0.002 a	0.522 ± 0.003 cd	0.516 ± 0.004 d	0.526 ± 0.005 c
linolenic acid (C18:3n3)	0.204 ± 0.001 a	0.204 ± 0.001 a	0.190 ± 0.003 b	0.148 ± 0.004 c	0.143 ± 0.002 d	0.139 ± 0.002 d
Behenic acid (C22:0)	0.030 ± 0.001 a	0.025 ± 0.001 b	0.024 ± 0.000 b	0.021 ± 0.000 c	0.020 ± 0.001 c	0.017 ± 0.000 d
Erucic acid (C22:1n9)	0.028 ± 0.000 a	0.029 ± 0.001 a	0.030 ± 0.001 a	0.024 ± 0.000 b	0.021 ± 0.001 b	0.022 ± 0.001 b
Arachidonic acid (C20:4n6)	—	—	—	0.024 ± 0.000 b	0.027 ± 0.002 a	0.008 ± 0.000 c
Tetracosanoic acid (C24:0)	0.058 ± 0.001 a	0.053 ± 0.001 b	0.055 ± 0.000 b	0.044 ± 0.002 c	0.044 ± 0.001 c	0.043 ± 0.001 c
Tetracosenoic acid (C24:1n9)	0.054 ± 0.000 b	0.056 ± 0.000 a	0.057 ± 0.000 a	0.041 ± 0.001 c	0.039 ± 0.001 d	0.034 ± 0.001 e
SAFA	11.199 ± 0.006 a	11.108 ± 0.010 b	10.918 ± 0.001 c	10.251 ± 0.005 f	10.360 ± 0.023 e	10.581 ± 0.009 d
MUFA	79.743 ± 0.009 e	79.730 ± 0.026 e	80.427 ± 0.011 d	81.400 ± 0.010 a	80.649 ± 0.044 c	80.884 ± 0.024 b
PUFA	9.057 ± 0.003 b	9.161 ± 0.018 a	8.654 ± 0.01 d	8.347 ± 0.015 f	8.989 ± 0.027 c	8.533 ± 0.023 e

^a SAFA: saturated fatty acids; MUFA: monounsaturated fatty acids; PUFA: polyunsaturated fatty acids.

^b Different letters indicated that the significant differences ($p < 0.05$) in the same row. All values are mean ± standard deviation of three replicates.

its content was between 79.016% and 80.744%. The oleic acid content increased at first and then decreased with the increase of the ripening degree of camellia fruit. Additionally, the oleic acid content reached the highest on D4. Similarly, the changing trend of arachidonic acid was the same as that of oleic acid, and arachidonic acid content reached the highest on D3. The content of the other three MUFAs (palmitoleic acid, erucic acid, tetracosenoic acid) decreased gradually with the increase of ripening degree of

camellia fruit. The PUFA content of CSOs from various harvest dates was between 8.347% and 9.161%. Linoleic acid (8.173%-8.956%) and linolenic acid (0.139%-0.204%) were the main PUFAs, and their content decreased gradually with the increase of ripening degree of camellia fruit. Moreover, it was worth noting that pentadecanoic acid was only detected in CSOs of the earlier harvesting dates (D1, D2, D3), while arachidonic acid was only detected in CSOs of the later harvesting dates (D4, D5, D7).

In a word, the harvest date significantly affected the fatty acid composition of CSO. Oleic acid, linoleic acid, palmitic acid, and stearic acid were the main fatty acids of CSOs from various harvest dates. During the ripening of camellia fruit, the SAFA and PUFA content of CSO decreased gradually, while the MUFA content of CSO increased at first and then decreased. Ye *et al.* studied the fatty acid composition of camellia oleifera seed oil at different maturities and the change trends of SAFA, PUFA, and MUFA content of CSO were consistent with the results of this study²⁵⁾. The synthesis and change of these fatty acids were mainly affected by two enzyme systems, including acetyl-CoA carboxylase and fatty acid synthase complex²⁵⁾. Under the action of these enzyme systems, SAFAs may be converted into unsaturated fatty acids, and PUFAs may be converted into MUFAs.

3.2.2 Volatile compounds

Flavor, as the sensory quality index of CSO, is an important factor affecting consumers' choices. To understand the effect of the ripening degree of camellia fruit on the CSO flavor, the volatile compounds of CSOs from various harvest dates were identified by HS-SPME-GC-MS. The results are shown in Table 3. A total of 37 volatile compounds were identified, including 12 aldehydes, 5 ketones, 12 alcohols, 2 acids, 5 esters, and 1 other. Moreover, the concentrations of different volatile compound types were calculated, and the results are shown in Table 4. Combining Tables 3 and 4, it can be seen that the composition and concentration of volatile compounds in CSOs from various harvest dates showed significant differences. Then, based on different volatile compound types, the changes of CSO flavor during the camellia fruit ripening process were analyzed.

Aldehydes are an important part of the CSO flavor, which usually contribute greatly to the flavor due to the lower odor threshold. The concentration of aldehydes in CSOs from various harvest dates was between 1315.08 µg/kg and 6806.63 µg/kg. During D1-D7, the concentration of aldehydes increased at first and then decreased with the increase of the ripening degree of camellia fruit. The lower concentration of aldehydes was observed in the early and late ripening stages, while the highest was observed in the fully ripening stage (D5). Previous studies had shown that the concentration of aldehydes in Hojiblanca olive oil increased at first and then decreased with the increase of olive fruit maturity, which was consistent with this study²⁶⁾. Hexanal was a typical volatile compound in CSO, which was mainly characterized by green odor and was synthesized by the lipoxygenase pathway with linoleic acid as the precursor²⁷⁾. It was observed that the hexanal concentration reached the highest on D5 (4751.45 µg/kg), which may be because the lipoxygenase activity of fully ripening camellia fruit was higher than that in the early and late ripening stages. In addition, the concentrations of heptanal,

octanal, (*E*)-2-octenal, decanal, and (*E*)-2-nonenal also reached the highest on D5, and these aldehydes mainly contributed to green and fatty odors. The above aldehydes were mainly produced through the degradation pathway of fatty acids. Among them, heptanal and (*E*)-2-octenal were the degradation products of linoleic acid, while octanal, decanal and (*E*)-2-nonenal were the degradation products of oleic acid^{27, 28)}. Except for these fatty aldehydes, the concentrations of benzaldehyde and benzeneacetaldehyde decreased gradually with the increase of ripening degree of camellia fruit, which were typical degradation products of phenylalanine and mainly contributed greatly to floral and honey odors²⁹⁾.

The concentration of ketones in CSOs from various harvest dates was between 543.57 µg/kg and 1240.06 µg/kg. The concentration of ketones on D1 and D2 were significantly higher than that at other harvest dates, which was mainly reflected in the higher concentrations of acetoin, 1-hydroxy-2-propanone, and acetophenone on D1 and D2. Among them, acetoin contributed to sweet and fatty odors, while 1-hydroxy-2-propanone and acetophenone contributed to sweet and pungent odors.

Alcohols were the second-largest volatile compounds in CSOs from various harvest dates, and the concentration of alcohols was between 503.78 µg/kg and 2003.15 µg/kg. Similar to aldehydes, the concentration of alcohols reached the highest when at the fully ripening stage of camellia fruit (D5). Caporaso *et al.* study the volatile compounds of Spanish olive oil and found that the concentration of alcohols in the fully ripening stage was higher than that in the early and late ripening stages³⁰⁾. This was consistent with our study. A total of 7 alcohols reached the highest concentration on D5, including 1-pentanol, 1-hexanol, 1-heptanol, 1-octanol, propylene glycol, α -terpineol, and benzyl alcohol. Among them, 1-hexanol (herbal, green) was mainly formed by the lipoxygenase pathway, and its concentration depended on the activity of alcohol dehydrogenase³¹⁾. 1-Pentanol (fermented, fusel), 1-heptanol (green), and 1-octanol (green, waxy) were formed by the degradation of fatty acids. Additionally, benzyl alcohol (floral, fruity) was the degradation product of phenylalanine²⁹⁾. The above alcohols made the CSO at full ripening stage have strong green, fermented and floral odors.

The concentration of acids in CSOs from various harvest dates was between 83.97 µg/kg and 367.76 µg/kg, which increased at first and then decreased with the increase of ripening degree of camellia fruit and reached the highest on D4. Only acetic acid and 3-methyl-butanoic acid were identified, which were the main contribution to the sour odor.

The concentration of esters in CSO from various harvest dates was between 1118.96 µg/kg and 2983.48 µg/kg, which decreased significantly with the increase of ripening degree of camellia fruit. Interestingly, the change of esters concen-

Table 3 The fatty acid composition of *Camellia semiserrata* seed oils from various harvest dates.

No.	Volatile compound	RT	RI ^a	RI ^b	Concentration (µg/kg) ^c						
					D1	D2	D3	D4	D5	D7	
Aldehyde											
1	Hexanal	6.855	1079	1083	150.91 ± 52.85c	225.87 ± 92.81c	648.4 ± 21.85b	640.13 ± 55.08b	4751.45 ± 210.88a	710.37 ± 216.86b	
2	Heptanal	9.636	1181	1183	25.98 ± 1.2c	22.39 ± 4.32c	50.66 ± 11.38b	16.18 ± 0.16c	279.8 ± 6.24a	20.17 ± 8.28c	
3	Octanal	12.563	1285	1289	65.23 ± 20.19c	87.01 ± 22.07bc	104.59 ± 24.17b	33.78 ± 8.78d	403.4 ± 4.79a	62.21 ± 10.87cd	
4	Nonanal	15.419	1389	1392	956 ± 231.35bc	1026.61 ± 188.75b	1379.96 ± 301.37a	229.1 ± 54.78e	663.92 ± 51.84cd	401.34 ± 37.61de	
5	(E)-2-Octenal	16.321	1424	1425	12.69 ± 3.01bc	12.12 ± 3.37bc	18.01 ± 1.73b	9.51 ± 5.58c	30.99 ± 4.55a	14.36 ± 2.58bc	
6	Methional	16.939	1448	1453	98.3 ± 17.75a	88.97 ± 7.58ab	67.96 ± 21.88c	17.79 ± 2.13d	97.51 ± 9.63a	—	
7	Decanal	18.156	1494	1496	175.22 ± 3.31ab	147.6 ± 102.43b	165.57 ± 129.81ab	90.28 ± 6.08b	309.76 ± 84.15a	111.53 ± 59.12b	
8	Benzaldehyde	18.656	1515	1515	713.58 ± 40.96a	533.27 ± 109.29b	333.12 ± 32.03c	274.62 ± 16.53cd	205.44 ± 17.99d	104.47 ± 9.95e	
9	(E)-2-Nonenal	19.064	1531	1532	10.01 ± 1.51b	8.76 ± 0.76b	7.26 ± 2.96b	3.69 ± 0.25c	14.38 ± 1.69a	—	
10	Benzeneacetaldehyde	21.569	1635	1646	177.84 ± 14.11a	187.44 ± 14.5a	44.7 ± 0.9b	—	13.68 ± 3.24c	31.53 ± 4.32b	
11	2-Dodecenal	21.666	1639	—	—	—	—	—	27.15 ± 5.83a	—	
12	2-Undecenal	24.145	1747	—	—	—	—	—	9.15 ± 1.64a	—	
Ketone											
13	Acetoin	12.373	1278	1280	254.44 ± 50bc	643.99 ± 88.92a	163.16 ± 6.87d	281.75 ± 19.48bc	245.83 ± 4.49bc	419.1 ± 33.64b	
14	1-Hydroxy-2-propanone	12.755	1292	1295	339.07 ± 33.56a	364.86 ± 40.29a	—	—	—	—	
15	2-Nonanone	15.312	1385	1386	—	—	—	—	10.53 ± 4.87a	—	
16	5-Hydroxy-4-octanone	16.443	1428	—	—	—	7.13 ± 1.52a	6.41 ± 0.58a	—	7.8 ± 2.05a	
17	Acetophenone	21.759	1643	1649	646.55 ± 35a	681.67 ± 67.64a	381.74 ± 38.4c	458.77 ± 16.63b	287.2 ± 8.7d	241.83 ± 1.19d	
Alcohol											
18	3-Methyl-1-butanol	10.408	1208	1210	89.67 ± 33.97d	116.91 ± 13.19cd	124.78 ± 19.55cd	203.57 ± 20.77b	153.98 ± 38.26c	570.47 ± 30.85a	
19	1-Pentanol	11.603	1251	1252	—	—	—	101.44 ± 12.53b	701.87 ± 40.22a	146.57 ± 73.45b	
20	1-Hexanol	14.436	1353	1356	—	—	—	20.49 ± 3.83b	103.6 ± 5.79a	—	
21	1-Heptanol	17.153	1456	1458	—	—	—	61.63 ± 2.46b	344.89 ± 42.2a	34.26 ± 6.44b	
22	3-Pentanol	19.164	1535	—	—	—	—	—	—	8.35 ± 2.65a	
23	1-Octanol	19.719	1558	1561	39.5 ± 14.84c	55.79 ± 7.06b	43.85 ± 5.82bc	13.35 ± 1.87d	135.53 ± 2.78a	36.24 ± 5.28c	
24	2,3-Butanediol	20.159	1576	1576	408.18 ± 111.11a	515.84 ± 61.97a	178.13 ± 41.82b	184.66 ± 2.81b	405.44 ± 19.24a	492.16 ± 52.45a	
25	Propylene Glycol	20.485	1589	1594	16.9 ± 5.29a	22.22 ± 0.18a	8.38 ± 1.08c	6.81 ± 1.28c	16.72 ± 2.17a	18.09 ± 7.84a	
26	Terpinen-4-ol	20.749	1600	1600	90.01 ± 11.87b	125.61 ± 41.52a	63.42 ± 7.21bc	50.03 ± 0.67c	44.14 ± 1.77c	36.21 ± 1.79c	
27	α-Terpineol	22.972	1695	1697	16.52 ± 0.83ab	24.72 ± 8.68a	15.67 ± 3.7ab	14.6 ± 1.26ab	24.21 ± 10.65a	10.31 ± 5.16b	
28	Benzyl alcohol	26.833	1871	1877	11.61 ± 5.16b	10.1 ± 6.32b	7.74 ± 1.39b	8.7 ± 0.35b	23.86 ± 0.95a	6.76 ± 0.06b	
29	Phenylethyl Alcohol	27.571	1906	1912	104.51 ± 10.4a	117.91 ± 16.25a	61.81 ± 8.75c	53.59 ± 7.05c	48.89 ± 4.24c	85.47 ± 9.16b	
Acid											
30	Acetic acid	17.585	1472	1449	194.4 ± 33.94b	121.95 ± 15.88c	271.53 ± 19.95a	207.41 ± 3.81b	262.27 ± 28.08a	83.97 ± 2.87d	
31	3-Methyl-butanolic acid	23.945	1738	—	86.18 ± 7.26b	64.33 ± 18.05bc	—	160.35 ± 81.16a	—	—	
Ester											
32	Ethyl tiglate	11.121	1234	1232	—	—	—	19.11 ± 1.27a	—	—	
33	2-Methylpropyl 2-hydroxypropanoate	14.425	1353	—	48.28 ± 9.5a	56.67 ± 17.69a	—	—	—	79.14 ± 35.6a	
34	Butyrolactone	21.169	1618	1623	274.02 ± 26.77b	338.47 ± 60.59b	104.36 ± 1.19c	70.12 ± 5.53c	106.36 ± 5.84c	550.94 ± 151.18a	
35	Hexanoic acid, ethenyl ester	22.033	1654	—	—	—	—	—	54.83 ± 6.38a	—	
36	Methyl cinnamate	30.831	2069	2073	2661.17 ± 227.03a	2480.36 ± 627.02a	1898.2 ± 336.01b	1825.18 ± 264.49b	1057.13 ± 52.61c	488.87 ± 76.86c	
Other											
37	2-Pentyl-furan	10.981	1229	1231	—	—	—	10.81 ± 0.88b	42.08 ± 0.99a	—	

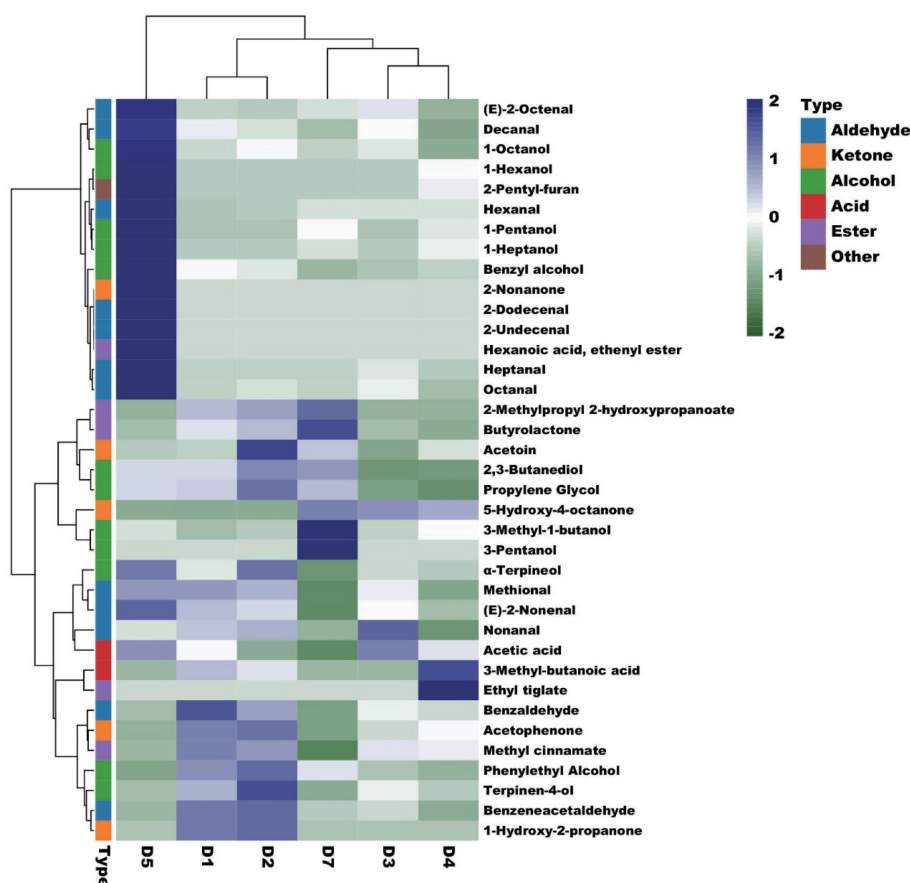
"—" Not detected

^a Retention index calculated on DB-Wax metal capillary column using n-alkanes C7-C30 as external standard.^b Retention index reference value from NIST Standard Reference Database Number 69 (<https://webbook.nist.gov/chemistry/>).^c Different letters indicated that the significant differences ($p < 0.05$) in the same row. All values are mean ± standard deviation of three replicates.

Table 4 The concentrations of different volatile compound types of *Camellia semiserrata* seed oils from various harvest dates.

Compound types	Concentration ($\mu\text{g/kg}$) ^a					
	D1	D2	D3	D4	D5	D7
Aldehyde	2385.77 $\pm$ 309.71 b	2340.05 $\pm$ 93.56 b	2820.24 $\pm$ 468.38 b	1315.08 $\pm$ 124.12 c	6806.63 $\pm$ 250.19 a	1455.98 $\pm$ 208.83 c
Ketone	1240.06 $\pm$ 114.75 b	1690.52 $\pm$ 113.21 a	552.02 $\pm$ 36.47 d	746.94 $\pm$ 36.49 c	543.57 $\pm$ 0.83 d	668.74 $\pm$ 35.41 cd
Alcohol	776.9 $\pm$ 166.39 d	989.09 $\pm$ 50.62 c	503.78 $\pm$ 37.59 c	718.86 $\pm$ 45.59 d	2003.15 $\pm$ 103.41 a	1444.91 $\pm$ 129.81 b
Acid	280.58 $\pm$ 39.37 b	186.28 $\pm$ 18.59 c	271.53 $\pm$ 19.95 b	367.76 $\pm$ 84.7 a	262.27 $\pm$ 28.08 b	83.97 $\pm$ 2.87 d
Ester	2983.48 $\pm$ 258.66 a	2875.51 $\pm$ 561.3 a	2002.56 $\pm$ 337.1 b	1914.41 $\pm$ 261.03 b	1218.32 $\pm$ 48.87 c	1118.96 $\pm$ 199.17 c

^a Different letters indicated that the significant differences ($p < 0.05$) in the same row. All values are mean $\pm$ standard deviation of three replicates.

**Fig. 2** Cluster heat map of *Camellia semiserrata* seed oils from various harvest dates.

tration was mainly reflected in the change of methyl cinnamate concentration. During D1-D7, the methyl cinnamate concentration decreased significantly from 2661.17 $\mu\text{g/kg}$ to 488.87 $\mu\text{g/kg}$. Methyl cinnamate mainly provided sweet and balsam odors.

To further distinguish the flavor profile of CSOs from various harvest dates, a cluster heat map (Fig. 2) was drawn based on the concentration of 37 volatile compounds. It could be seen from Fig. 2 that CSOs from various harvest dates could be divided into four clusters: cluster 1 (D1 and D2), cluster 2 (D3 and D4), cluster 3 (D5), and cluster 4 (D7). Blue in the heatmap represented high concentration,

while green represented low concentration. It could be observed that more compounds had higher concentrations on D5, and these compounds were mainly aldehydes and alcohols. As for D1 and D2, there were also many compounds showing relatively high concentrations, mainly aldehydes, alcohols, ketones, and esters. In addition, some alcohols, esters, and ketones showed relatively high concentrations on D7. However, most compounds had relatively low concentrations on D3 and D4. In a word, the harvest date was an important factor affecting the CSO flavor, and the flavor intensity of the CSO on D5 (full ripening stage) was the highest.

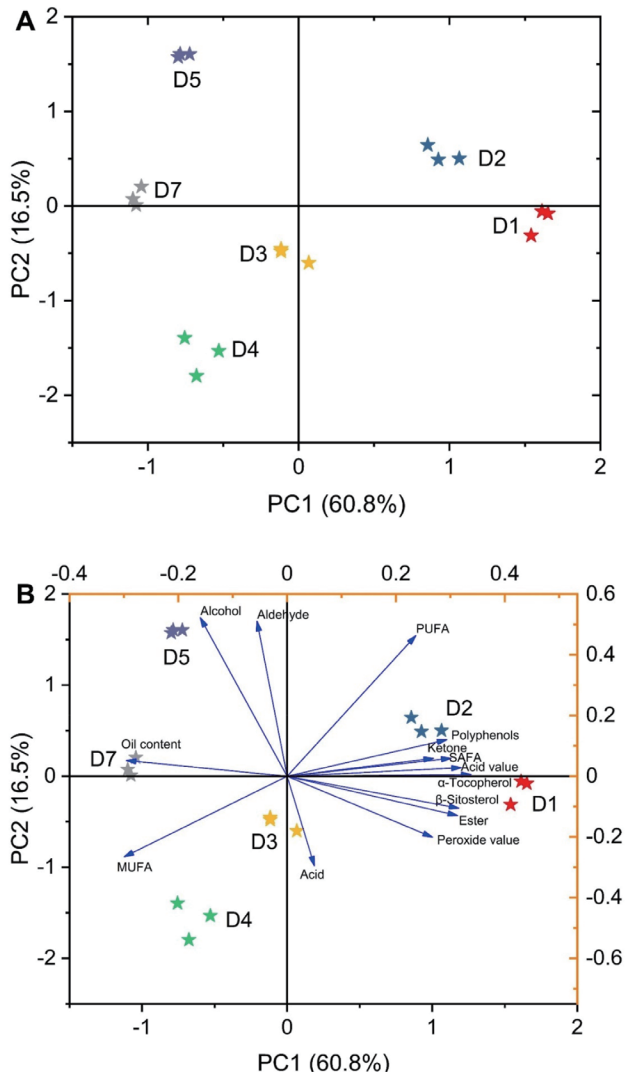


Fig. 3 Principal component analysis of *Camellia semiserrata* seed oils from various harvest dates. (A) Score plot, (B) biplot.

3.3 Comprehensive evaluation of quality of *Camellia semiserrata* seed oils from various harvest dates

3.3.1 Classification of *Camellia semiserrata* seed oils from various harvest dates based on PCA

Principal component analysis (PCA) is often used to comprehensively evaluate and distinguish different samples³²⁾. To fully understand the overall quality differences of CSOs from various harvest dates, the PCA model was established based on the data of oil content, physicochemical indexes, nutritional indexes, fatty acid indexes (SAFA, MUFA, PUFA), and volatile compound indexes (aldehydes, ketones, alcohols, acids, esters). Figure 3A was the PCA score plot of CSOs from various harvest dates. PC1 and PC2 explained 60.8% and 16.5% of the total variables respectively, which could better reflect the information of the different samples. By observing the PCA score

plot, it could be found that D1 and D2 were located on the right side of the PC1 axis, D3 and D4 were located in the third quadrant, while D5 and D7 were located in the second quadrant. This meant that CSOs from various harvest dates were divided into three groups, and there were significant differences in the comprehensive quality of CSOs in different groups.

To screen the characteristic indexes causing the comprehensive quality difference of CSOs, the PCA biplot was drawn combined with the score plot and loading plot (Fig. 3B). The indexes close to the sample point in the figure were the characteristic indexes of the sample. As shown in Fig. 3B, the characteristic indexes of CSOs of D1 and D2 included polyphenols, α -tocopherol, β -sitosterol, acid value, peroxide value, SAFA, PUFA, ketones, and esters. The characteristic indexes of CSOs of D3 and D4 were MUFA and acids. Oil content, alcohols, and aldehydes were the characteristic indexes of CSOs of D5 and D7.

3.3.2 Comprehensive quality ranking of *Camellia semiserrata* seed oils from various harvest dates based on GRA

The quality indexes of CSO showed different change rules during the camellia fruit ripening. Therefore, to objectively select the best harvest date of *Camellia semiserrata* fruit, the grey relation analysis (GRA) was used to rank the comprehensive quality of CSOs from various harvest dates. Considering that the values of different indexes represent different meanings, the index values of the reference sequence were set as follows: the maximum value of oil content, the minimum physicochemical indexes, the maximum value of nutritional indexes, the average value of fatty acid indexes, the maximum value of volatile compound indexes (Table S1). The dimensionless processing result of the sample sequences was shown in Table S2. Based on the dimensionless data, the grey relation coefficient and grey relation degree of each sample were calculated, and the results were shown in Tables 5 and 6, respectively. Higher correlation degree indicated better the comprehensive quality. Therefore, the order of comprehensive quality of CSOs from various harvest dates was D5 > D1 > D3 > D2 > D7 > D4. All in all, the D5 (September 30) was the best harvest date of *Camellia semiserrata* fruit.

4 Conclusions

The harvest date had a significant effect on the oil content, physicochemical indexes, nutritional indexes, fatty acid composition, and volatile compounds of *Camellia semiserrata* seed oil. With the increase of ripening degree, the oil content of camellia seed increased at first and then decreased, while the physicochemical indexes (acid value and peroxide value) and nutritional indexes (the contents of β -sitosterol, α -tocopherol, and polyphenols) of

Table 5 The grey relation coefficient and grey relation degree of *Camellia semiserrata* seed oils from various harvest dates.

Indexes	Grey relation coefficient					
	D1	D2	D3	D4	D5	D7
Oil content	0.333	0.519	0.721	0.817	1.000	0.641
β -Sitosterol	1.000	0.861	0.686	0.514	0.438	0.355
α -Tocopherol	1.000	0.657	0.477	0.410	0.401	0.381
Polyphenols	1.000	0.537	0.543	0.387	0.495	0.353
Acid value	0.343	0.409	0.611	0.574	0.577	1.000
Peroxide value	0.335	0.672	0.780	0.572	0.915	1.000
SAFA	0.526	0.580	0.739	0.515	0.578	0.768
MUFA	0.538	0.534	0.949	0.478	0.828	0.673
PUFA	0.613	0.532	0.756	0.488	0.679	0.622
Aldehyde	0.433	0.430	0.458	0.380	1.000	0.387
Ketone	0.619	0.999	0.391	0.437	0.389	0.417
Alcohol	0.417	0.464	0.369	0.406	1.000	0.611
Acid	0.620	0.439	0.596	0.998	0.574	0.334
Ester	1.000	0.914	0.540	0.518	0.394	0.381

Table 6 The grey relation degree of *Camellia semiserrata* seed oils from various harvest dates.

Sample	Grey relation degree	Rank
D1	0.627	2
D2	0.611	4
D3	0.615	3
D4	0.535	6
D5	0.662	1
D7	0.566	5

CSO decreased gradually. Among them, the oil content reached the highest (58.74%) on September 30, while the contents of β -sitosterol, α -tocopherol and polyphenols reached the highest on September 2, which were 6881.60, 311.34 and 78.08 mg/kg, respectively. Oleic acid, linoleic acid, palmitic acid, and stearic acid were the main fatty acids of CSOs from various harvest dates. During the ripening of camellia fruit, the SAFA and PUFA content decreased gradually, while the MUFA content increased at first and then decreased. In terms of volatile compounds, the concentrations of alcohols and aldehydes reached the highest in the fully ripening stage (September 30), which were 6806.63 and 2003.15 μ g/kg. The concentrations of ketones and esters were higher in the early ripening stage. Among them, the highest concentration of ketones (1690.52 μ g/kg) was observed on September 9, and the highest concentration of esters (2983.48 μ g/kg) was observed on September 2. The CSOs from various harvest

dates were divided into three groups by PCA: group 1 (D1 and D2), group 2 (D3 and D4), group 3 (D5 and D7). Furthermore, through the comprehensive quality ranking of GRA, it was concluded the best harvest date of *Camellia semiserrata* fruit was September 30. This study systematically revealed the effect of harvest date on the comprehensive quality of *Camellia semiserrata* seed oil and provide theoretical guidance for growers' breeding and harvesting work.

Author Contributions

Junhua He: Writing-original draft, Writing-review & editing, Software, Instrumental analysis, Data collection. Xuehui Wu: Conceptualization, Methodology, Writing-review & editing, Project administration, Fund applicant. Jun Liu: Investigation, Validation, Visualization, Supervision. Yongfang Huang: Resources, Formal analysis, Methodology. Jianfeng Zhang: Investigation, Resources, Supervision.

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Conflict of Interest

The authors declare that they have no conflict of interest.

Supporting Information

This material is available free of charge via the Internet at doi: 10.5650/jos.ess22114

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