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Abstract: Bleaching is a necessary step in the production of refined camellia oil (Camellia oleifera Abel), since crude oil is a dark brown colour, due to pigments extracted from the seed coat during pressing, that is unacceptable to consumers. In order to understand the quality change and oxidative state of camellia oil in the bleaching step, measurements of various quality parameters, i.e. peroxide value (POV), free fatty acid (FFA) and UV absorbance, and volatile profile of crude and bleached oils were carried out. The results showed that FFA, K270, and K232 increased, whereas POV decreased, with increase of the activated earth dosage of 0-4% and of bleaching time from 0 to 40 min at 110 -°C. As the amount of activated earth was increased from 0% to 4% with bleaching at 110 -°C for 30 min, various classes of volatile compounds increased in concentration: aldehydes (23.7 Î¼g/g), alcohols (13.2 Î¼g/g), esters (8.0 Î¼g/g), alkenes (2.0 Î¼g/g) and ketones (1.9 Î¼g/g). Likewise when bleaching was carried out at 110 -°C with 3% activated earth, and the bleaching time varied between 0 and 40 min, concentrations of volatile compounds also increased; aldehydes (27.7 Î¼g/g), alcohols (18.2 Î¼g/g), esters (7.3 Î¼g/g), ketones (3.2 Î¼g/g) and alkenes (0.6 Î¼g/g). These findings indicate that hydroperoxides in the oil were decomposed into lower molecular weight products in the process of bleaching and that the extent of this decomposition can be controlled by time and amount of activated earth.
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Summary

Bleaching is a necessary step in the production of refined camellia oil (*Camellia oleifera* Abel), since crude oil is a dark brown colour, due to pigments extracted from the seed coat during pressing, that is unacceptable to consumers. In order to understand the quality change and oxidative state of camellia oil in the bleaching step, measurements of various quality parameters, i.e. peroxide value (POV), free fatty acid (FFA) and UV absorbance, and volatile profile of crude and bleached oils were carried out. The results showed that FFA, K_{270}, and K_{232} increased, whereas POV decreased, with increase of the activated earth dosage of 0-4% and of bleaching time from 0 to 40 min at 110 °C. As the amount of activated earth was increased from 0% to 4% with bleaching at 110 °C for 30 min, various classes of volatile compounds increased in concentration: aldehydes (23.7 μg/g), alcohols (13.2 μg/g), esters (8.0 μg/g), alkenes (2.0 μg/g) and ketones (1.9 μg/g). Likewise when bleaching was carried out at 110 °C with 3% activated earth, and the bleaching time varied between 0 and 40 min, concentrations of volatile compounds also increased; aldehydes (27.7 μg/g), alcohols (18.2 μg/g), esters (7.3 μg/g), ketones (3.2 μg/g) and alkenes (0.6 μg/g). These findings indicate that hydroperoxides in the oil were decomposed into lower molecular weight products in the process of bleaching and that the extent of this decomposition can be controlled by time and amount of activated earth.

1 Introduction

Seeds of camellia (*Camellia oleifera* Abel, Theaceae) have been utilized in China for more than 1000 years [1]. The extracted camellia oil is a favorite cooking oil in south China, especially in Hunan and Jiangxi provinces, where more than 50% of the vegetable cooking oil is from camellia [1, 2]. Camellia oil has several favourable characteristics including a high oleic acid content typically exceeding 80% with low polyunsaturated content and good phenolic content, which mean that rancidity during processing will be minimised and phenols will provide stability on storage [3]. In the Chinese camellia oil industry, the seed is mainly processed by hydraulic press for crude oil in the local production area [4]. The hydraulic pressed crude oil is also used as the main material for refining to meet the standard of the Chinese government, i.e. GB-11765-2003 [5]. The bleaching technology of camellia oil has been studied many times [6, 7, 8]. However, the effect of bleaching conditions using activated earth was mostly determined by classical analytical methods such as free fatty acids, peroxide value and carbonyl value, which are not enough for a full understanding of oxidative processes during bleaching. To get detailed information such as individual deterioration products of hydroperoxides, headspace solid-phase microextraction (SPME) has been adopted [9, 10].

In the present study, the changes of quality of camellia oil in the course of bleaching using activated earth are investigated, in order to establish a scientific background for the process used for oil refining. To monitor the oxidative states of bleached oil free fatty acid, peroxide value, and UV absorbance were determined. Changes in concentration of the volatile compounds were performed by gas chromatography-mass spectrometry (GC-MS) coupled with SPME.

2 Materials and methods

2.1 Materials

Crude camellia oil was a commercial sample obtained from Hunan, P.R. China. Activated earth was purchased from the Second Chemical Engineering Company of Ezhou, Hubei, P.R.China. Hexanal, octanal, nonanol were purchased from Fluka Co., USA, and heptanal, nonanal were abtain from Alfa Co, UK. Hexyl acetate, ethyl acetate, pentanol, butyl acetate, hexanol, benzaldehyde, heptanol, 2-octanone, octanol were analytical grade reagents from Top Chem Co., LTD, Shenzhen, China..

2.2 Laboratory scale refining

Laboratory scale refining was designed to duplicate commercial practices in China. Conventional water degumming was used to remove phospholipids. The crude oil was heated to 80 °C and mixed with 3% soft water pre-heated to 80 °C, and then stirred for 30 min. Centrifugation at 3000 rpm for 20 min separated the water and oil [11]. Alkali refining was performed by the method reported by Pang *et al*. [8]. The water-degummed oil (100 g) was stirred at 60-70 rpm for about 20 min while 10% NaOH was added. The temperature was then increased by 1 °C/min and the stirring rate was reduced to 30-40 rpm for 10-15 min until the temperature reached 55-65 °C. The oil was left to stand 8 h. Next, the neutralised oil was heated to 78-80 °C and mixed with 6 - 10% soft water (6-10 mL) that had been heated to 83-85 °C. The mixture was cooled and left to stand for 1 h to separate oil and water. The procedure was repeated 3-6 times until the separated water was clear [8]. The oil was bleached at 110 °C and 10 kPa residual pressure for 0-40 min with 0-4% of activated bleaching earth [10], that met standard HG/T 2569-1994 with free acid (expressed as % H_2SO_4) ≤0.20; moisture (%) ≤8.0 and activity ≥220. The samples were cooled and stored at –18 °C until analysis.

2.3 Fatty acid profile

The fatty acid profile was determined as fatty acid methyl esters by gas chromatography. The methyl esters were prepared and analysed as previously

described [3] by addition of methanolic potassium hydroxide (0.2 M) to 0.1 g oil in 2 ml n-heptane. The mixture was shaken vigorously and allowed to stand and separate. An aliquot (1 ml) of the heptane phase was removed and analysed by gas chromatography using flame ionization detection [3].

2.4 Volatile profiles

The volatile compounds from different oils were extracted using SPME with a polydimethylsiloxane/carbowax/divinylbenzene fiber (PDMS/CAR/DVB, 50/30μm, Supelco) as follows. Oil (1 g) was placed in a 15 mL reactivial (Supelco) and equilibrated at 40 °C for 15 min. The SPME needle was inserted through the septum and left in the headspace at the same temperature with stirring of 100 rpm for 25 min. The fiber was retracted into the needle and immediately transferred into the gas chromatograph. A PerkinElmer Clarus 500 gas chromatograph with a PE-5 column (30 m × 0.25 mm i.d. × 0.25 μm film) was used. The column was temperature programmed from an initial temperature of 40 °C for 5 min, increased at 4 °C /min to 100 °C, and then increased at 3 °C /min to 250 °C with a final isothermal period of 10 min. The fiber was desorbed for 3 min using a splitless injection port at 260 °C. The flow rate of nitrogen carrier gas was 0.67 mL min^{-1} and the precolumn pressure of nitrogen was at 69.95 kPa. Chromatograms were routinely monitored by an FID detector which was maintained at 280 °C. For identification, volatiles were analyzed by gas chromatography-mass spectrometry by thermal desorption in the injection port of a Finnigan Trace GC Ultra gas chromatograph (The Electron Corporation, USA) coupled with a Finnigan Trace DSQ quadruple mass spectrometer using the same chromatographic conditions as before. Electron impact ionization (EI) was used for MS. The electron multiplier voltage for MS was 1400 V, and filament emission current was 100 μA. The ion source temperature was maintained at 230 °C. The temperature of the transfer line, interfacing the GC and MS, was set at 260 °C. Mass spectral scan time from *m/z* 35 to 450 was 0.8 s.

The concentrations of volatile compounds that showed in 2.1 were calculated from the relative regression equations. The concentrations of other aldehydes, esters, alcohols and ketones were calculated as equivalents of hexanal, ethyl acetate , octanol , 2-octanone, respectively.

2.5 UV absorbance

UV absorption was determined using the International Olive Oil Council method COI/T20/Doc. No. 19 [12].

2.6 Free fatty acids (FFA)

Free fatty acids were determined by a method of the American Oilseed Chemists' Society (Aa 6-38) [13].

2.7 Peroxide value

Peroxide value was determined using the International Union of Pure and Applied Chemistry method 2-50 [14].

2.8 Statistical Analyses

Significant differences were performed using T-test. Data analysis was done using SPSS13.0 (SPSS Inc., Chicago). All analyses were performed in at least duplicate.

3 Results and discussion

The Chinese standard GB 11765-2003 specifies that the colour of refined oil (Lovibond cuvette, 25.4 mm) must be Yellow $\leq$35, Red $\leq$3.0 for Grade 2 and Yellow $\leq$35, Red $\leq$2.0 for Grade 1 classification. The hydraulic pressed crude oil used for

this study was Yellow =35, Red =4.7 (Lovibond cuvette, 25.4 mm). After bleaching using 4% earth at 110 °C for 40 min, the colour was Yellow =35, Red =0.6 (Lovibond cuvette, 25.4 mm). The crude oil conformed to the typical camellia oil fatty acid profile with palmitic acid, 9.1%; stearic acid 2.0%; oleic acid 79.7%; linoleic acid 8.7%; linolenic acid 0.3% and eicosaenoic acid 0.3%.

In the bleaching step, the peroxide value decreased significantly with the increase in the amount of activated earth and time (Fig. 1 and Fig. 2, $p<0.01$). Oxidation of oil was accelerated and hydroperoxides were further decomposed into lower molecular weight products in the process of bleaching due to the great surface area of activated earth [15]. Meanwhile, free fatty acid had a tendency to increase with increase of bleaching time and amount of activated earth. A former study also found these changes in camellia oil refining [6]. However, compared with corn and soybean oil, the peroxide value and free fatty acid content of camellia oil did not change dramatically [16, 17]. The greater stability of camellia oil compared with corn and soybean oil during bleaching can be attributed to the favourable fatty acid profile of camellia oil.

Although there is no prescribed Chinese standard for UV absorbance, it is well accepted that UV absorbance is used as an indicator of the oil quality and level of oxidation for olive oil since K_{232} provides an indicator of the content of diene conjugated bonds, while K_{270} reflects triene concentration [18]. Both K_{232} and K_{270} increased significantly ($p<0.001$) as the earth dosage and bleaching time increased (Fig. 3, Fig. 4). There were linear correlations between K_{232}, K_{270} and the amount of earth, or bleaching time (data not shown).

The measurement of volatile compounds by GC provides an alternative monitoring technique of the changes induced by bleaching. The increase in total area count of the GC-MS chromatograms of volatiles with increasing amounts of activated earth is shown in Fig 5. This trend was also found in concentrations of most individual

volatiles (Table 1) with the exception of 6-camphenone, limonene and decanoic acid. Benzaldehyde, for example, showed a 10-fold increase in concentration as the amount of activated earth increased from 0% to 4%. New compounds such as mycene, 1,5-heptadiene-3,4-diol, ε-caprolactone, terpinolen, 2-amino octanoic acid and (*E*,*Z*)-2,4-decadienal were formed during bleaching, particularly after the amount of activated earth increased above 2%. The formation of (*E*,*Z*)-2,4-decadienal, and the increase in concentration of (*E*,*E*)-2,4-decadienal and 2,6-dimethyl-5,7-octadien-2-ol at higher levels of activated earth is consistent with the increase in K_{232} (Fig. 3) – an indicator of conjugated dienes. Due to the complex fatty acid composition of camellia oil a great variety of hydroperoxides were present in the crude oil (POV was 16.0 meq/kg in the degummed oil). The activated earth decomposed the hydroperoxides and numerous volatile substances were formed or increased in concentration in the bleached oil as the level of activated earth increased. This is also consistent with the decrease in POV shown in Fig. 1.

Fig. 6 clearly showed changes of different types of volatiles with the change of amount of activated earth used in bleaching. In absolute terms, aldehydes had the greatest increase in concentration (23.7 μg/g) as the amount of activated earth was increased, followed by alcohols (13.2 μg/g), esters (8.0 μg/g), alkenes (2.0μg/g) and ketones (1.9 μg/g). In relative terms, esters showed the largest increase in concentration (300%), followed by aldehydes (172%), alcohols (170%), ketones (155%) and alkenes (95%). The large increase in aldehyde concentration is consistent with the activated earth decomposing hydroperoxides (as discussed above). The large relative increase in ester concentration may be due to activated earth catalyzing esterification reactions.

For examining the effect of bleaching time on oil quality and volatile compound formation, 3% activated earth was used and the degummed oils were bleached at 110 °C for 10, 20, 30, 40 min. Total area of volatiles increased with the bleaching time

($p<0.01$, Fig. 7) , while the number of peaks increased from 37 into 41. The concentrations of most individual compounds, except decanoic acid, 6-camphenone and 5,7-diethyl-5,6-decadien-3-yne, also increased with the bleaching time (Table 2). Once again, benzaldehyde showed the largest increase in concentration with bleaching time, from 0.14 μg/g before bleaching to 2.04 μg/g after 40 min of bleaching. Most of the volatiles, e.g. pentanol, hexanol, octanal, hexyl acetate and decanal, had a linear relation between concentration and bleaching time with R^2 of 0.97-0.99. The concentrations of different classes of volatiles, namely, aldehydes, alcohols, esters, alkenes and ketones, increased with bleaching time (Fig. 8) similar to the result from increase in amount of activated earth (Fig. 6). Among these volatile compounds, aldehydes and alcohols were dominant fractions. At 40 min of bleaching, 2,5-dimethylpyrazine, which is responsible for a "roasty" aroma note, was detected. The formation of pyrazines in the course of the Maillard reaction required a minimum reaction time and temperature of 50 min and 100 °C for pumpkin seed [19, 20]. Due to the higher reaction temperature of 110 °C, a slightly shorter time of 40 min to detect 2,5-dimethylpyrazine may be expected.

4 Conclusion

Bleaching is a necessary step in the production of refined camellia oil to meet the specifications for colour contained in Chinese legislation. This study sheds light on the chemical changes the oil undergoes under different conditions of bleaching with activated earth. Both bleaching time and amount of activated earth affect gross oil parameters such as POV, FFA, K_{232} and K_{270}. A decrease in POV during bleaching is desirable, yet the downside of this is an increase in K_{232} and K_{270} generated through the decomposition of hydroperoxides. Consistent with this, aldehydes, especially conjugated dienes and trienes increase with increasing amounts of activated earth and temperature. Interestingly, 2,5-dimethylpyrazine, which would impart a positive sensory note to the oil, is formed during bleaching at 110 °C. However, it is likely that

this would be lost during subsequent deodorizing, made necessary by the generation of undesirable aldehydes in the bleaching step.

References

[1] J. M. Ruter. Nursery production of tea oil camellia under different light levels. In *Trends in New Crops and New Uses*. Eds. J. Janick and A. Whipkey, ASHS Press, Alexandria, VA., 2002, pp 222-224

[2] L. Tang, E. Bayer, R. Zhuang. Obtain, properties and utilization of Chinese teaseed oil. *Fett Wissenschaft Technologie-Fat Science Technology,* 1993,**95**, 23-27.

[3] H. Zhong, D. Bedgood, A. Bishop, P. Prenzler, K. Robards. Endogenous biophenol, fatty acid and volatile profiles of selected oils. *Food Chem.*, 2007, **100**, 1544-1551.

[4] H. Y. Zhong, C. N. Wan, B. X. Xie. The present status and development tendency of utilization and processing in camellia oil in China. *China Forestry Science and Technology*, 2001, **4**, 6-9

[5] General Administration of Quality Supervision, Inspection and Quarantine of People's Republic of China. Chinese National Standard, Oil-tea camellia oil, Chinese Standard Publishing House, 2003, GB-11765-2003.

[6] H. Y. Zhong, C. N. Wan, B. X. Xie, M. Y. Zhao. The bleaching technique and colour determination of oil-tea camellia seed oil. *Journal of Central South Forestry University*, 2000, **4** ,25-29

[7] S. Z. Lu, H. Zen, X. Q. Cai. The refining technology of camellia oil. *Guangxi Forestry Science*, 2003, 4, 182-184.

[8] W. S. Pang, X. R. Mao. The investigation of refining technology of camellia oil. *China Forestry Science and Technology*, 2004, 1, 54-55.

[9] F. Doleschall, Z. S. Kemény, K. Recseg, et al. A new analytical method to monitor lipid peroxidation during bleaching. *Eur.J.Lipid Sci.Technol*., 2002, **104**, 14 ~ 18.

[10] F. Doleschall, Z. S. Kemény, K. Recseg, K. Kövári. Monitoring of lipid degradation products by solid-phase microextraction. *J. Microcolumn Separations*, 2001, **13**, 215 ~ 220.

[11] H. Y. Zhong, Y. Q. Zhang, H. Z. Sun, Z. H. Li. Hydrated degumming technique for camellia oil. *Nonwood Forest Research*, 2004, **1**, 29-31

[12] IOOC Method of analysis: Spectrophotometric investigation in the ultraviolet. COI/T20/Doc. no. 19/Rev.1, 2001.

[13] AOCS. Official methods and recommended practices of the American Oil Chemists' Society. 5th edition. AOCS Press, Champoign, Illinois, 1998, p115.

[14] IUPAC. Standard methods for the analysis of oils, fats and derivatives, 7th revised edition, Blackwell Scientific Publications, London 1992.

[15] H. B. W. Patterson. Bleaching and purifying fats and oils. Theory and Practice, AOCS Press, llinois (USA), 1992, pp. 69 ~ 71.

[16] S. Y. Man, Y. H. Hu. Effect of bleaching on the oil quality. *China Oils and Fats*, 2001, **6**, 31-33.

[17] G. Y. Lou, Q. Zhao, X. F. Wang. The bleaching technology of salad corn oil. *China oils and Fats*, 1998, **4**, 36-37.

[18] R. J. Mailer, J. Ayton, D. Conlan. Comparison and evaluation of the quality of thirty-eight commercial Australian and New Zealand olive oils. *Adv. Hort. Sci*., 2002, **16**, 259-266.

[19] J. Maga, C. E. Sizer. Pyrazines in foods. *CRC Critical Reviews in Food Technology*, 1973, **4**, 39-115.

[20] B. Siegmund, M. Murkovic. Changes in chemical composition of pumpkin seeds during the roasting process for production of pumpkin seed oil. (Part 2: volatile compounds). *Food Chem.*, 2004, **84**, 367-374.

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Figure Captions

Fig. 1. Effect of amount of active earth on the peroxide value and acid value of camellia oil [neutralised oil with 0% activated earth after heating at 110 °C for 30 min].

Fig. 2. Effect of bleaching time on the peroxide value and acid value of camellia oil [bleached oil with 3% activated earth and no heating].

Fig.3. Effect of amount of activated earth on K_{232} value and K_{270} values [bleached at 110 °C for 30 min].

Fig. 4. Effect of bleaching time on K_{232} value and K_{270} values of camellia oils [bleached at 110 °C with 3% activated earth].

Fig. 5. Effect of amount of active earth on total peak area of volatile compounds in GC-FID chromatograms [bleached at 110 °C for 30 min].

Fig. 6. Effect of dosage of activated earth on amount of different volatile variety of camellia oils [bleached at 110 °C for 30 min].

Fig. 7. Effect of bleaching time on total peak area of volatile compounds in GC-FID chromatograms [bleached at 110 °C with 3% activated earth].

Fig. 8. Effect of bleaching time on amount of different classes of volatile compounds of camellia oils [bleached at 110 °C, with 3% activated earth].

Tab. 1 Effect of amount of active earth on volatile compounds of camellia oil (μg/g) bleached at 110 °C for 30 min.

No	Compounds	Amount of activated earth (%)				
		0	1	2	3	4
1	Ethyl acetate	2.16±0.03	3.16±0.08	4.68±0.26	6.75±0.60	8.68±0.34
2	Hexane	2.35±0.15	3.26±0.23	4.82±0.32	6.05±0.62	7.49±0.35
3	2-Methyl-1-butanol	2.65±0.06	3.59±0.36	4.65±0.66	6.15±0.78	6.54±0.59
4	Pentanol	1.56±0.14	2.44±0.21	3.25±0.37	4.10±0.32	5.14±0.41
5	Hexanal	4.18±0.01	4.40±0.03	7.71±0.02	8.83±0.04	11.21±1.37
6	Hexanol	0.90±0.08	1.01±0.05	1.18±0.21	1.35±0.05	1.07±0.02
7	Styrene	0.68±0.06	0.79±0.06	1.03±0.26	1.23±0.15	1.33±0.03
8	Heptanal	0.64±0.22	0.85±0.74	2.05±0.01	2.55±0.13	3.19±0.11
9	α-Pinene	0.07±0.00	0.29±0.02	0.31±0.03	0.46±0.19	0.60±0.07
10	(*E*)-2-heptenal	0.06±0.01	0.07±0.00	0.12±0.01	0.18±0.02	0.25±0.04
11	Benzaldehyde	0.16±0.01	0.33±0.03	0.46±0.01	0.80±0.04	1.69±0.03
12	Heptanol	0.31±0.02	0.39±0.02	0.53±0.02	0.87±0.09	1.11±0.03
13	Myrcene	ND	ND	0.31±0.04	0.35±0.07	0.47±0.11
14	Ethyl caproate	0.11±0.00	0.18±0.00	0.21±0.02	0.27±0.01	0.40±0.01
15	2-Octanone	1.78±0.03	1.95±0.40	2.17±0.09	2.09±0.11	2.71±0.03
16	Octanal	0.88±0.01	0.82±0.02	1.22±0.02	1.80±0.05	4.50±0.26
17	Hexyl acetate	0.38±0.00	0.60±0.10	0.62±0.10	0.97±0.08	1.35±0.07
18	Limonene	0.13±0.03	0.20±0.05	0.14±0.02	0.07±0.00	0.10±0.01
19	1,5-Heptadiene-3,4-diol	ND	ND	0.21±0.01	0.21±0.00	0.20±0.02
20	ε-Caprolactone	ND	ND	ND	0.06±0.00	0.18±0.02
21	(*E*)-2-octenal	0.59±0.01	0.82±0.10	1.19±0.08	1.40±0.06	1.76±0.12
22	Octanol	0.70±0.09	0.76±0.07	0.77±0.08	1.13±0.14	1.77±0.11
23	Terpinolene	ND	ND	0.13±0.00	0.14±0.02	0.17±0.01
24	2-Nonanone	0.08±0.01	0.10±0.01	0.14±0.02	0.19±0.00	0.24±0.02
25	Nonanal	4.73±0.35	5.01±0.11	6.89±0.32	7.79±0.41	9.13±0.49
26	2-phenylethanol	0.56±0.11	0.62±0.06	0.73±0.04	1.06±0.04	1.24±0.05
27	2-Hexylfuran	0.28±0.02	0.35±0.04	0.50±0.03	0.56±0.02	0.60±0.00
28	(*E*)-2-nonenal	0.77±0.05	0.76±0.05	1.06±0.04	1.12±0.03	1.20±0.06
29	(*Z*)-2-nonenal	0.15±0.06	0.23±0.02	0.30±0.03	0.38±0.03	0.41±0.04
30	2-Decanone	0.07±0.00	0.07±0.02	0.09±0.01	0.11±0.03	0.12±0.02
31	Decanal	0.99±0.10	0.92±0.04	1.22±0.04	1.24±0.03	1.29±0.04
32	Decanol	0.62±0.03	1.39±0.04	1.86±0.03	1.88±0.06	2.63±0.08
33	2-Amino octanoic acid	ND	ND	0.58±0.02	0.63±0.06	0.78±0.07
34	(*E*)-2-decenal	0.13±0.02	0.27±0.04	0.26±0.03	0.30±0.04	0.29±0.07
35	2-Undecenal	0.37±0.01	0.54±0.02	1.06±0.01	1.16±0.08	1.90±0.03
36	Decanoic acid	0.19±0.02	0.57±0.01	0.44±0.06	0.29±0.09	0.15±0.04

37	(*E*,*Z*)-2,4-decadienal	ND	ND	ND	0.12±0.01	0.24±0.01
38	(*E*,E)-2,4-decadienal	0.24±0.02	0.25±0.03	0.33±0.01	0.35±0.04	0.41±0.02
39	2,6-Dimethyl-5,7-octadien-2-ol	0.45±0.08	0.48±0.04	0.70±0.02	0.75±0.06	0.79±0.01
40	5,7-Diethyl-5,6-decadien-3-yne	0.47±0.08	0.45±0.01	0.41±0.01	0.40±0.00	0.37±0.03
41	6-Camphenone	0.16±0.03	0.06±0.00	0.07±0.01	0.07±0.00	ND
42	α-Muurolene	0.32±0.04	0.30±0.02	0.32±0.04	0.39±0.03	0.39±0.04

ND: not detected.

Tab. 2 Effect of bleaching time on volatile compounds of camellia oil (μg/g) bleached at 110 °C with 3% activated earth.

No	Compounds	Time of bleaching（min）				
		0	10	20	30	40
1	Ethyl acetate	2.45±0.13	6.23±0.23	7.04±0.44	7.25±0.89	8.49±0.66
2	Hexane	2.14±0.13	3.05±0.06	3.15±0.12	5.59±0.70	8.49±0.35
3	2-Methyl-1-butanol	2.65±0.06	3.36±0.12	4.56±0.74	6.22±1.48	8.53±0.86
4	Pentanol	1.26±0.09	2.51±0.17	3.59±0.20	4.56±0.52	6.74±0.26
5	Hexanal	3.84±0.14	6.49±0.05	7.26±0.11	8.15±0.29	11.96±1.26
6	Hexanol	0.84±0.03	1.07±0.18	1.30±0.00	1.46±0.06	1.65±0.04
7	Styrene	0.55±0.02	0.75±0.13	0.93±0.14	1.33±0.03	1.59±0.40
8	Heptanal	0.78±0.10	1.27±0.34	1.72±0.25	1.73±0.20	1.93±0.13
9	2,5-dimethylpyrazine	ND	ND	ND	ND	0.40±0.01
10	α-Pinene	0.08±0.01	0.21±0.04	0.33±0.09	0.46±0.18	1.11±0.37
11	(*E*)-2-heptenal	0.06±0.00	0.06±0.01	0.09±0.01	0.16±0.02	0.20±0.04
12	Benzaldehyde	0.14±0.00	0.29±0.01	0.64±0.01	1.25±0.26	2.04±0.28
13	Heptanol	0.35±0.03	0.42±0.03	0.97±0.10	1.06±0.23	1.28±0.08
14	Myrcene	ND	ND	0.25±0.03	0.35±0.06	0.47±0.10
15	Ethyl caproate	0.10±0.01	0.19±0.01	0.30±0.02	0.34±0.04	0.36±0.01
16	2-Octanone	1.56±0.05	2.08±0.12	2.03±0.06	2.04±0.13	2.09±0.01
17	Octanal	0.85±0.01	1.16±0.01	2.17±0.33	3.21±0.49	4.22±0.50
18	Hexyl acetate	0.36±0.02	0.46±0.01	0.73±0.15	1.02±0.07	1.25±0.01
19	Limonene	0.12±0.00	0.15±0.01	0.19±0.00	0.20±0.09	0.22±0.05
20	1,5-Heptadiene-3,4-diol	ND	ND	0.18±0.02	0.30±0.09	0.41±0.02
21	ε-Caprolactone	ND	ND	0.05±0.01	0.07±0.00	0.13±0.02
22	(*E*)-2-octenal	0.52±0.03	1.28±0.01	1.34±0.07	1.62±0.09	1.71±0.16
23	Octanol	0.65±0.02	1.00±0.10	1.25±0.32	1.70±0.16	2.48±0.17
24	Terpinolene	ND	0.22±0.01	0.26±0.05	0.28±0.03	0.52±0.01
25	2-Nonanone	0.08±0.00	0.08±0.01	0.13±0.04	0.17±0.02	0.30±0.02
26	Nonanal	5.10±0.26	6.65±0.22	7.94±0.08	9.98±0.13	13.54±0.57
27	2-phenylethanol	0.51±0.03	0.77±0.01	1.05±0.54	1.06±0.09	1.17±0.40
28	2-Hexylfuran	0.23±0.02	0.51±0.04	0.49±0.03	0.56±0.01	0.53±0.04
29	(*E*)-2-nonenal	0.70±0.03	0.79±0.19	0.85±0.16	0.89±0.06	0.97±0.16
30	(*Z*)-2-nonenal	0.13±0.00	0.27±0.05	0.31±0.01	0.41±0.02	0.56±0.20
31	2-Decanone	0.07±0.00	0.08±0.00	0.09±0.02	0.10±0.05	0.10±0.04
32	Decanal	0.93±0.08	1.02±0.12	1.15±0.05	1.21±0.08	1.36±0.05
33	Decanol	0.60±0.03	0.67±0.05	1.29±0.49	1.66±0.14	2.27±0.25
34	(*E*)-2-decenal	0.13±0.01	0.19±0.02	0.30±0.04	0.33±0.01	0.34±0.18
35	2-Undecenal	0.36±0.02	0.74±0.17	1.43±0.32	1.74±0.12	2.04±0.06
36	Decanoic acid	0.18±0.02	0.49±0.04	0.34±0.03	0.19±0.03	0.34±0.00
37	(*E,Z*)-2,4-decadienal	ND	ND	ND	0.14±0.02	0.27±0.00
38	(*E,E*)-2,4-decadienal	0.23±0.03	0.26±0.06	0.31±0.06	0.31±0.06	0.32±0.07

39	2,6-Dimethyl-5,7-octadien-2-ol	0.39±0.06	0.60±0.03	0.69±0.11	0.72±0.03	0.88±0.31
40	5,7-Diethyl-5,6-decadien-3-yne	0.49±0.06	0.47±0.08	0.42±0.04	0.40±0.03	0.37±0.02
41	6-Camphenone	0.15±0.02	0.07±0.01	0.04±0.01	0.04±0.01	ND
42	α-Muurolene	0.28±0.03	0.31±0.02	0.33±0.01	0.25±0.02	0.29±0.03

ND: not detected.