Physicochemical properties, flavor intensity and oxidative stability of different camellia oils

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Abstract: Comparison and analysis of physicochemical properties and oxidative stability of 10 brands of camellia oils were conducted in this study. Results showed that more attention should be paid to the iodine (IV), saponification (SV) and peroxide (PV) values of camellia oils during sampling inspection as they were more likely to be out the range of quality standards. Regression analysis between physicochemical indices and oxidative stability showed individual index could not affect the stability of camellia oils significantly ($p>0.05$). However, very high correlations were found between physical indices such as optical rotation (OR) and turbidity ($R=-0.929$), turbidity and color ($R=-0.930$). High correlations were found between chemical indices such as IV and moisture and volatile matter (MVM) ($R=-0.853$), IV and PV ($R=0.831$), MVM and PV ($R=-0.809$). Package with nitrogen could retard the oxidation of camellia oil. These results may be useful for rapid evaluation, differentiation and quality improvement of camellia oils.

Keywords: camellia oil, optical rotation, regression analysis, quality indices, sensory evaluation
DOI: 10.25165/j.ijabe.20181105.3656

Citation: Jiang X F, Wu S M, Yuan D, Lamu C J, Wang Q, Zhang Z H. Physicochemical properties, flavor intensity and oxidative stability of different camellia oils. Int J Agric & Biol Eng, 2018; 11(5): 230–235.

1 Introduction

Camellia tea seeds, originated from China, have a long history of cultivation and utilization for over 2300 years. In some countries such as China, India, Sri Lanka, Indonesia and Japan, camellia oil has been accepted as an edible oil, and thousands of tons of camellia oil are produced annually[1]. The physicochemical properties of camellia oil are similar to those of olive oil, thus it is considered as "eastern olive oil" and "the king of cooking oil"[2].

Camellia oil has been considered as a kind of high quality edible oil because of its high contents of oleic acid (exceeding 80% of its fatty acid compositions) and lipid accompanying compounds (Vitamins A, B and E)[2,3]. It can be used to reduce the blood pressure, to postpone the process of aging, and to be a rich source of emollients for skin care[4]. In Chinese herbal medicine, camellia oil is also considered as a superior nutritional dietary supplement that benefits the digestion[2].

Nowadays, more and more commercial camellia oils are sold in China, and their prices are much higher than other common edible oils such as soybean and rapeseed oils. Therefore, adulterating camellia oil with other cheaper oils could be very lucrative in food industry. Till now, many new methods have been introduced to investigate the adulteration of camellia oils. Hai et al.[5] tried to use electronic nose to detect the maize oil adulteration in camellia oil, and the accuracy of prediction for camellia oil was 83.6%. Wang et al.[6] reported to use fiber optic diffuse reflectance near infrared spectroscopy (NIR) and attenuated total reflectance infrared spectroscopy (MIR) to determine the authentication of camellia oil. Weng et al.[7] revealed a high correlation ($R^2=0.994$) between the Raman intensity ratio (ν_{1656}/ν_{1439}) and the percentage of camellia oil. Li et al.[2] applied NIR spectroscopy with multivariate calibration models to discriminate pure camellia oil.

The prerequisite of the camellia oil discrimination is based on the characteristic data from original products. Until now, little research has focused on the difference of the physicochemical properties among camellia oils, and the stability of these camellia oils are unknown. In order to differentiate camellia oils from different companies, comprehensive study of the basic information for the camellia oils is necessary. The objective of this study is to compare and analyze the characteristic indices of different camellia oils, and try to evaluate the correlations between physicochemical properties, flavor intensity and oxidative stability of these camellia oil products. The present data are helpful to differentiate camellia oils and might be further used for authentication of the camellia oil products.

2 Materials and methods

2.1 Materials

Ten different camellia oil samples were kindly provided by ten different oil companies in China. Sample oils were packed in sealed glass bottles and sent to the laboratory as soom as possible after the production day. Two oil bottles were obtained from each company, and stored in darkness at 4°C prior to analysis. All

Received date: 2017-07-24 **Accepted date:** 2018-04-17
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chemicals and reagents of analytical grade were purchased from Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China).

2.2 Determination of physical indices

The turbidity of the camellia oils was determined using a turbidimeter (Model WGZ-200, Xinrui Instrument Co., Shanghai, China)[8]. The results were reported in nephelometric turbidity units (NTU).

The optical rotation (OR) of the oil samples was measured using an automatic polarimeter (Model WZZ-2B, Precision Instrument Co., Shanghai, China). Measurements were made at 20°C using light with a wave length of 589.44 nm.

The color of the oil samples was measured by Lovibond method (GB/T 22460-2008, National Standard of the People's Republic of China, 2008), and the type of cuvette used to determine color is 25.4 mm.

The relative density (RD) of the oil samples was determined in accordance with GB/T 5526-1985. The refractive index (RI) of the oil samples were determined according to the AOCS methods[9].

2.3 Determination of flavor intensity

The panel was composed of 12 trained members between 18 and 30 years old. All panelists had previously received training in descriptive sensory analysis (> 20 h) and had experience in sensory profiles of various food samples. These panelists were trained for an additional 2 h to identify the flavor of the oil samples. Quantitative descriptive analysis was applied to monitor the intensity of the flavor by using a scale of 0 (none) - 4 (very strong). The sensory experiments were performed in duplicate and the mean values and standard deviations were calculated.

2.4 Determination of chemical indices

The moisture and volatile matter (MVM), iodine value (IV), acid value (AV), peroxide value (PV), carbonyl group value (CGV), saponification value (SV) and unsaponifiable matter (USM) of the camellia oil samples were determined according to the AOCS methods[9].

2.5 Determination of oxidative stability

Analysis of the oxidative stability of the oil samples was modified based on the method of Tan et al.[10] using a differential scanning calorimeter (DSC) instrument (Model DSC 2910, TA Instruments, New Castle, USA). Indium was used to calibrate the instrument, and an empty open aluminium pan was used as reference. Oil samples (5.0±0.5 mg) were weighed into open aluminium pans and placed in the equipment's sample chamber. The temperature of the calorimeter program was increased from 30°C to 200°C at a rate of 20°C/min, and then equilibrated at 200°C for 8 min. Oxygen (purity>99.8%) was passed through the sample enclosure at 20 mL/min. The oxidation of the sample was observed as a sharp increase in the energy flow due to the exothermic nature of the oxidation reactions[11]. The induction period (IP) value of the oil samples was determined using the intersection of the extrapolated baseline and the tangent line.

2.6 Statistical analysis

Unless otherwise stated, all sample preparation and instrumental analyses were performed in duplicate. One-way ANOVA was carried out by using Tukey adjustment to determine the significant difference between different oil samples. Significant differences were declared at $p \leq 0.05$. Multiple regression analysis between individual variables and IP values were performed using SPSS 15.0 software (IBM SPSS Statistics, USA).

3 Results and discussion

3.1 Physical properties and flavor intensity

The physical characteristic indices (RI, RD, color, turbidity and OR) of the ten camellia oils are given in Table 1. The RI is often used as a criterion for oil purity[12]. The RI of the ten camellia oil samples varied from 1.4607-1.4620, which was a little lower than other reported vegetable oils such as olive (1.468-1.471), rapeseed (1.465-1.469), sunflower (1.467-1.469) and pumpkin (1.466-1.474) oils[13]. Sample 1 had significant higher RI than the other nine samples ($p<0.05$), while sample 2-10 had no significant differences ($p>0.05$). In general, all the RI of the ten oil samples could meet the requirement of the camellia oil standard (GB 11756-2003).

Table 1 Refractive index (RI), relative density (RD), color, turbidity and optical rotation (OR) of the ten camellia oils in China

Samples	RI (n^{40})	RD (d_{20}^{20})	Color [a] (yellow unit)	Turbidity /NTU	OR/(°)
S1	1.4620±0.0001^{A}	0.914±0.000^{AB}	0.6	0.96±0.01^{A}	0.176±0.013^{D}
S2	1.4607±0.0006^{B}	0.913±0.000^{A}	7.2	3.62±0.01^{B}	0.124±0.003^{BC}
S3	1.4608±0.0001^{B}	0.915±0.001^{BC}	0.3	0.24±0.00^{C}	0.099±0.001^{AB}
S4	1.4608±0.0006^{B}	0.915±0.001^{BC}	0.6	0.36±0.01^{D}	0.090±0.002^{A}
S5	1.4608±0.0000^{B}	0.916±0.001^{C}	1	0.55±0.01^{E}	0.115±0.001^{ABC}
S6	1.4608±0.0000^{B}	0.915±0.001^{BC}	0.6	0.43±0.01^{D}	0.067±0.007^{A}
S7	1.4610±0.0000^{B}	0.915±0.001^{BC}	1.2	2.56±0.06^{F}	0.449±0.031^{E}
S8	1.4610±0.0000^{B}	0.916±0.001^{C}	0.6	0.02±0.01^{G}	0.143±0.001^{C}
S9	1.4610±0.0000^{B}	0.914±0.000^{AB}	1.0	0.56±0.00^{E}	0.120±0.002^{AB}
S10	1.4610±0.0000^{B}	0.914±0.000^{AB}	2.0	0.96±0.01^{A}	0.190±0.002^{D}
Requirement GB 11756-2003	1.460-1.464	0.912-0.922	-	-	-

Note: [a] The type of cuvette used to determine color is 25.4 mm.

The letters A, B, C, D, E, F and G represent the differences among different oil samples, the same letter indicates no significant difference ($p>0.05$), different letters indicate a significant difference ($p<0.05$).

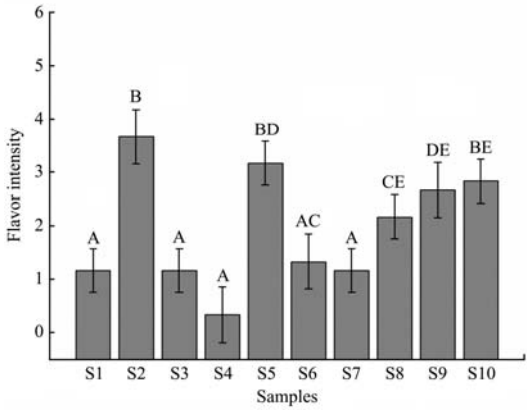
The RD of the ten camellia oils ranged from 0.913-0.916 g/cm^3 (Table 1). It has been reported that the RD of the oil decreased with the molecular weight, yet increased with the unsaturation level[14]. In our study, though the RD of the ten camellia oils could vary to some extent, all of them could meet the requirement of the camellia oil standard (GB 11756-2003). In addition, the RD of the ten camellia oils was within the range of other reported vegetable oils such as canola (0.911 g/cm^3), soybean (0.914 g/cm^3), sunflower (0.916 g/cm^3) and palm (0.918 g/cm^3) oils[15].

The turbidity of the ten camellia oils ranged from 0.02-3.62 NTU (Table 1), and sample 2 (3.62 NTU) and sample 7 (2.56 NTU) had significantly higher turbidity than the other eight oil samples ($p<0.05$), which indicated more impurities were contained in these two oil samples. It has been reported that different oil extraction

method could affect the oil turbidity significantly, and the turbidity for olive oils could vary from 0.6 to 455.1 NTU[16]. The difference in the turbidity for the ten camellia oils might be attributed to the difference in oil processing methods.

The optical rotation (OR) is a phenomenon peculiar to certain kinds of organic molecules which have the property of rotating a plane of polarized light passing through them, and the intensity of the OR is determined by both the types and amounts of the organic molecules present in the oil[17]. The OR values of the ten camellia oils ranged from 0.067°-0.449° (Table 1), which were significantly lower than the sesame oil (1.44°, 25°C)[18]. Sample 7 had significantly higher OR value (0.449°) than the other oil samples ($p<0.05$), while sample 6 had the lowest OR value (0.067°). The differences in OR values indicated that the compositions and contents of the organic molecules in camellia oils could vary greatly.

The color of the camellia oils was described by using yellow unit (Table 1), and sample 2 had the deepest color (yellow unit 7.2). It meant that sample 2 contained more pigments than the other nine oil samples. Figure 1 shows the flavor intensity of the ten camellia oils determined by sensory evaluation. Sample 2 had the strongest flavor (intensity 3.67), while sample 4 had the lightest flavor (intensity 0.33).



Note: The letters A, B, C, D and E represent the differences in flavor intensity among different oil samples: the same letter indicates no significant difference ($p>0.05$), different letters indicate a significant difference ($p<0.05$).

Figure 1 Flavor intensity of the ten camellia oils determined by sensory evaluation

3.2 Chemical properties

The chemical indices (IV, SV, USM, MVM, AV, PV and CGV) of the ten camellia oils are given in Table 2. The IV is a parameter that indicates the unsaturation of the oils, and higher IV means higher unsaturation[19]. Sample 5 had significantly lower IV (74.5±6.7 g/100 g) than the other oil samples ($p<0.05$), while the IV of sample 8 (98.6±1.5 g/100 g) and sample 9 (94.9±8.7 g/100 g) were significantly higher ($p<0.05$). Though the IV for the ten camellia oils (74.5-98.6 g/100 g) was within the range of other reported vegetables oils such as olive (75-94 g/100 g), rapeseed (94-120 g/100 g), sunflower (118-145 g/100 g) and pumpkin (116-133 g/100 g) oils[13], the IV for samples 5, 8 and 9 could not meet the requirement of the camellia oil standard (GB 11756-2003), which might be attributed to the deterioration of these oils.

The SV is a measure of the alkali-reactive groups in oils and it is useful to predict the type of glycerides in an oil sample. The glycerides with short-chain fatty acids usually have higher SV than those with long-chain fatty acids[12]. The SV of the ten camellia oils ranged from 190.3-194.0 mg KOH/g (Table 2), which was within the range of the reported vegetable oils such as olive (184-196 mg KOH/g), sunflower (188-194 mg KOH/g) and pumpkin (174-197 mg KOH/g) oils[13]. In addition, there was no significant difference in SV among the ten camellia oils ($p>0.05$). However, six out of ten of the studied oils had lower SV than the requirement of the camellia oil standard (GB 11756-2003), which might be attributed to the impurity (such as water, long chain fatty alcohols and sterols) contained in these oil samples.

The USM values of the ten camellia oils varied greatly from 2.6-9.2 g/kg (Table 2), which indicated that the amounts of long chain fatty alcohols, sterols and pigments contained in these camellia oils differed significantly[20]. Higher USM value usually means higher contents of hydrocarbons, sterols, triterpenols, carotenoids and tocopherols[12]. However, all the USM values of the ten oil samples could meet the requirement of the camellia oil standard (GB 11756-2003).

All the MVM values (0.01%-0.14%) of the ten oil samples could meet the requirement of the camellia oil standard (Table 2), and sample 2 had significantly higher MVM value than the other oil samples ($p<0.05$), which might affect its stability during storage[21].

Table 2 Iodine value (IV), saponification value (SV), unsaponifiable matter (USM), volatile matter (MVM), acid value (AV), peroxide value (PV) and carbonyl group value (CGV) of the ten camellia oils in China

Samples	IV/g·(100 g)$^{-1}$	SV (KOH)/mg·g^{-1}	USM/g·kg^{-1}	MVM/%	AV/mg KOH·g^{-1}	PV/mmol·kg^{-1}	CGV/meq·kg^{-1}
S1	86.8±9.5^{AB}	190.3±1.4^{A}	3.3±0.2^{A}	0.08±0.01^{A}	0.20±0.03^{A}	10.0±0.1^{A}	38.1±1.3^{AB}
S2	83.7±3.6^{AB}	192.8±1.7^{A}	8.1±0.1^{B}	0.14±0.01^{B}	1.12±0.01^{B}	16.2±0.1^{B}	51.6±5.8^{AB}
S3	82.4±2.9^{AB}	193.0±0.7^{A}	2.6±0.2^{C}	0.04±0.01^{CD}	0.15±0.02^{A}	6.7±0.3^{C}	38.2±7.9^{AB}
S4	83.2±1.1^{AB}	190.6±3.4^{A}	3.3±0.1^{A}	0.01±0.00^{C}	0.18±0.00^{A}	5.4±0.2^{D}	46.3±2.9^{AB}
S5	74.5±6.7^{A}	193.1±0.6^{A}	5.7±0.3^{D}	0.01±0.00^{C}	0.18±0.01^{A}	9.6±0.1^{A}	54.5±0.8^{A}
S6	88.4±0.7^{AB}	193.0±2.5^{A}	3.1±0.1^{A}	0.06±0.00^{AD}	0.16±0.04^{A}	6.1±0.0^{E}	55.3±2.5^{A}
S7	83.3±3.9^{AB}	192.8±2.1^{A}	5.9±0.1^{D}	0.08±0.01^{A}	0.20±0.02^{A}	8.5±0.2^{F}	31.0±3.5^{B}
S8	98.6±1.5^{B}	194.0±2.3^{A}	6.4±0.0^{E}	0.08±0.02^{A}	0.17±0.03^{A}	6.2±0.1^{E}	23.3±0.9^{C}
S9	94.9±8.7^{B}	190.5±1.8^{A}	9.2±0.2^{F}	0.04±0.01^{CD}	0.18±0.03^{A}	6.1±0.2^{E}	45.5±2.9^{AB}
S10	88.3±1.3^{AB}	191.7±1.3^{A}	7.5±0.1^{G}	0.06±0.02^{AD}	0.19±0.02^{A}	9.6±0.1^{A}	74.0±1.5^{D}
Requirement GB11765-2003	83-89	193-196	≤15	≤0.15	≤2.5	≤7.5	-

Note: The letters A, B, C, D, E, F and G represent the differences among different oil samples, the same letter indicates no significant difference ($p>0.05$), different letters indicate a significant difference ($p<0.05$).

The AV is a parameter that depends on the amount of free fatty acids present in the oil[22]. The results of AV showed that sample 2 had more free fatty acids than the other oil samples ($p<0.05$), which may be relevant to the different refining technique of the oil. However, all the AV of the ten oil samples could meet the requirement of the camellia oil standard (GB 11756-2003).

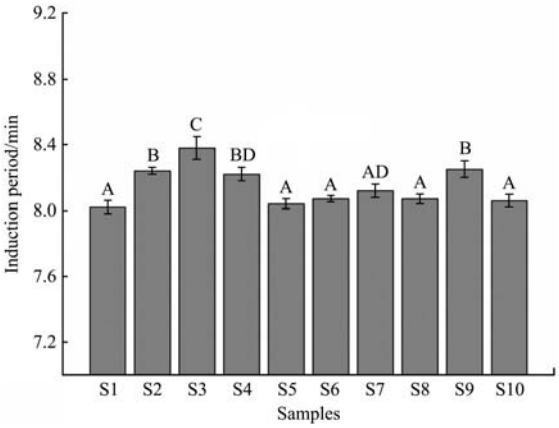
The PV indicates the level of the formed hydroperoxides, which are the primary oxidation products of the oils[23]. The PV

of the ten camellia oils ranged from 5.4-16.2 mmol/kg (Table 2), which was significantly higher than the refined soybean (0 mmol/kg)[24] and rapeseed (0.74 mmol/kg) oils[25]. Our results showed that only half of the ten oil samples could meet the requirement of China (GB 11756-2003), which indicated that camellia oils were liable to be oxidized.

The CGV characterizes the secondary decomposition products such as aldehydes and ketones. These compounds are more stable than hydroperoxides, and the CGV seems to be a good index of oxidative changes in lipids[26]. The CGV of the ten camellia oils ranged from 23.3-74.0 meq/kg. Sample 10 had significantly higher CGV (74.0 meq/kg) than the other nine samples, while the CGV of sample 8 (23.3 meq/kg) was significantly lower ($p<0.05$). It indicated that the formation of the secondary oxidation products varied greatly among different camellia oils.

3. 3 Oxidative stability

Figure 2 shows the IP values of the ten oil samples determined by DSC. The IP value is an indicator that predicts the oxidative stability of the oil during long-time storage, and higher IP value means higher stability of the oil[27]. In general, the IP values of the ten samples ranged from 8.06-8.38 min, and sample 3 had significantly higher IP than the other oil samples ($p<0.05$). Figure 3 shows the DSC diagram of the camellia oil (sample 8) under the treatments of oxygen and nitrogen. The IP values for the oxygen and nitrogen treatments were 8.07±0.03 min and 8.77±0.10 min, respectively. These results indicated that oxygen could promote the oxidation process of the camellia oil, while nitrogen could retard its oxidation.



Note: The letters A, B, C and D represent the differences in IP values among different oil samples, the same letter indicates no significant difference ($p>0.05$), different letters indicate a significant difference ($p<0.05$).

Figure 2 Induction period (IP) values of the ten camellia oil

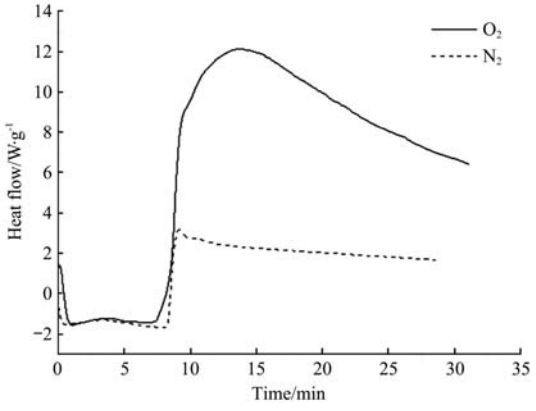


Figure 3 DSC diagram of the camellia oil (Sample 8) under the treatments of oxygen and nitrogen

3.4 Correlations between physical indices, flavor intensity and oxidative stability

Table 3 shows the regression analysis for the relationship between IP (dependent variable), physical indices and flavor intensity (independent variables) in camellia oils. The R^2 value of the model is 0.491, which meant that the independent variables explain 49.1% of the variability of the dependent variable. The p value of the model is 0.794 (>0.05), which implied that the regression model was not quite good fit for the data. All the p values (0.270-0.935) of the physical indices (RI, RD, turbidity, OR and color) and flavor intensity were more than 0.05, indicating that none of the individual index had significant effect on the oxidative stability of the camellia oils.

Very high correlations were found between OR and turbidity ($R=-0.929$) and between turbidity and color ($R=-0.930$). The values of OR, turbidity and color for the ten camellia oils revealed that oil samples with higher turbidity usually had deeper color and higher optical rotation (Table 1). In addition, medium correlation ($R=-0.726$) was found between color and flavor intensity (Table 3). It should be noted that oil samples with deeper color (such as samples 2, 5, 9 and 10) usually had higher flavor intensity (Table 1), which might be attributed to the difference in refining process, and more volatiles are remained in these oil samples.

Table 3 Multiple regression analysis for the relationship between IP (dependent variable), physical indices and flavor intensity (independent variables) in camellia oils

Model[a]	Partial regression coefficient	Standard deviation	Standard coefficient	t	p	
Constant	406.847	294.400		1.382	0.261	
RI	–225.077	166.777	–0.718	–1.350	0.270	
RD	–76.255	–0.620	–0.620	–0.881	0.443	
T	0.021	0.204	0.204	–0.089	0.935	
OR	–0.230	–0.215	–0.215	–0.184	0.866	
Color	–0.025	–0.437	–0.437	–0.190	0.862	
Flavor	–0.029	–0.269	–0.269	–0.384	0.726	
Correlations[b]	Flavor	OR	RI	RD	Turbidity	Color
Flavor	1	–0.502	–0.155	–0.096	0.595	–0.726
OR	–0.502	1	–0.161	–0.047	–0.929	0.835
RI	–0.155	–0.161	1	0.545	0.103	0.185
RD	–0.096	–0.047	0.545	1	–0.057	0.242
T	0.595	–0.929	0.103	–0.057	1	–0.930
Color	–0.726	0.835	0.185	0.242	–0.930	1

Note: [a] R^2 and p values of the model are 0.491 and 0.794, respectively.
[b] IP is dependent variable in the model.

3.5 Correlations between chemical indices and oxidative stability

Table 4 shows the regression analysis for the relationship between IP (dependent variable) and chemical indices (independent variables) in camellia oils. The R^2 value of the model is 0.501, which meant that the independent variables explain 50.1% of the variability of the dependent variable. The p value of the model is 0.911 (>0.05), which revealed that the regression model could not fit for the data well. In addition, the results of p values (0.329-0.998) for these chemical indices (IV, SV, USM, MVM, AV, PV and CGV) showed that individual chemical index could not affect the oxidative stability of the camellia oils significantly.

It has been reported that oil stability could be affected by the

water content and unsaturation of the oil[10,28], and PV presents the primary oxidation products contained in the oil[23]. Our study revealed that there existed high correlations between IV and MVM ($R=-0.853$), between IV and PV ($R=0.831$) and between MVM and PV ($R=-0.809$) (Table 4).

Table 4 Regression analysis for the relationship between IP (dependent variable) and chemical indices (independent variables) in camellia oils

Model[a]	Partial regression coefficient	Standard deviation	Standard coefficient	*t*	*p*		
Constant	11.942	10.823		1.103	0.385		
IV	–0.009	0.023	–0.510	–0.385	0.737		
SV	–0.014	0.051	–0.159	–0.278	0.807		
USM	0.009	0.041	0.186	0.228	0.841		
MVM	–0.017	5.043	–0.006	–0.003	0.998		
AV	0.540	0.421	1.377	1.281	0.329		
PV	–0.052	0.072	–1.427	–0.719	0.547		
CGV	–0.001	0.005	–0.160	–0.253	0.824		
Correlations[b]	CGV	AV	SV	IV	USM	MVM	PV
CGV	1	0.024	0.050	–0.198	0.023	0.401	–0.385
AV	0.024	1	–0.008	0.041	–0.124	–0.187	–0.330
SV	0.050	–0.008	1	0.398	–0.255	–0.404	0.339
IV	–0.198	0.041	0.398	1	–0.727	–0.853	0.831
USM	0.023	–0.124	–0.255	–0.727	1	0.570	–0.595
MVM	0.401	–0.187	–0.404	–0.853	0.570	1	–0.809
PV	–0.385	–0.330	0.339	0.831	–0.595	–0.809	1

Note: [a] R^2 and *p* values of the model are 0.501 and 0.911, respectively.
[b] IP is dependent variable in the model.

4 Conclusions

This study reports the characteristic indices of camellia oils from different companies, and evaluates the correlations between physicochemical properties, flavor intensity and oxidative stability of these camellia oils. The results revealed that the physicochemical properties of camellia oils from different companies could vary greatly, and none of the individual index could affect the oxidative stability of the camellia oils significantly. The results of PV and IP indicated that camellia oils were liable to be oxidized and nitrogen could be an effective way to retard their oxidation. Furthermore, more attention should be paid to the SV and IV indices of camellia oils during quality control, because these two indices were more likely to be out the range of quality standards. In general, this study provides basic information of the camellia oils and it might be further used for authentication of the camellia oil products.

Acknowledgments

This study was supported by the National Natural Science Foundation of China (Grant No. 31471668 and Grant No. 31671958), SJTU Agri-X Funding (Grant No. Agri-X2015007), Risk Assessment Laboratory (Hangzhou) Funding (Grant No.2015FXPG03), and China Postdoctoral Science Foundation (Grant No. 2015M580333).

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