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Flavor Characteristics of Lapsang Souchong and Smoked Lapsang Souchong, a Special Chinese Black Tea with Pine Smoking Process

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The major volatile constituents of lapsang souchong, smoked lapsang souchong, and smoked souchong, a group of special black teas in China, were analyzed with GC and GC-MS analyses. Forty-nine constituents were identified. Longifolene and α-terpineol were the most abundant compounds in the aroma. Due to its special production process, the compounds longifolene, longicyclene, guaiacol, 4-methylguaiacol, 4-ethylguaiacol, etc., were identified only in this kind of black tea. The aroma constituents of tea origin decreased during the smoking process, whereas the pine terpenoids and the thermal pyrolysis products of the pine wood increased markedly. The pine material for smoking was also analyzed for the volatile constituents showing apparent selectivity of the tea leaves to absorb. There were apparent differences among the tea samples in the contents of aroma constituents. The characteristics of the aroma of the teas are discussed with their processes and the features of sensory qualities.

KEYWORDS: Tea; *Camellia sinensis*; lapsang souchong; *Pinus taiwanensis*; pine smoke; volatile constituent; aroma

INTRODUCTION

Lapsang souchong (Zhengshan Xiaozhong) is a special black tea produced in the Wuyi (Bohea) Mountain area in southeastern China from the young shoots of the local tea species (*Camellia sinensis* var. *sinensis* cv. Bohea). It is said that the production began in the middle of 15th century, and lapsang souchong is known to be the origin of black tea. It is the grade of black tea native to China, produced only in the heartland of the National Wuyi Mountain Nature Preservation Zone. Wuyi Mountain has been proved to have been the cradle of black tea in the world. The word "Bohea" meant the black tea (lapsang souchong) produced in the Wuyi area in the early world tea trade; for example, a literature source published in 1750 recorded that "the Europeans contracted their first acquaintance with the green tea, then Bohea took its place" (*1*). In the beginning of 18th century Bohea (black tea) was the commonly used kind of tea in Boston (*2*). The exportation of Wuyi black tea to Europe and America was much earlier than the cultivation of tea trees and black tea manufacturing in foreign countries (*3*). Although nowadays the CTC and orthodox black teas are produced in large quantities in China and abroad and are well consumed in the world, lapsang souchong is still produced in the Wuyi Mountain area with its traditional outstanding quality characteristics.

The quality of lapsang souchong is superior to that of other black teas. Its taste is rich, giving a harmony feeling, and its aroma is sweet and fruity plus a special flavor of pine. The quality is decided by the excellent environment of the Wuyi Mountain with a natural forest system, which has been designated as a national nature preservation zone and an area of the "Man and Biosphere" project by UNESCO. The traditional black teas, lapsang souchong and smoked lapsang souchong, are produced in the Wuyi Mountain area. Smoked lapsang souchong is processed from lapsang souchong through the heating of pine branches (smoking). In addition, there is another black tea called smoked souchong, which is processed from congou black tea (leaf tea) through the smoking process. Such products are mainly exported to Europe, having a good reputation there for the special smoked flavor.

Here we report the flavor of lapsang souchong, smoked lapsang souchong, and smoked souchong and discuss the effects of the special processes compared to common black tea manufacturing.

MATERIALS AND METHODS

Materials. The three samples, lapsang souchong, smoked lapsang souchong, and smoked souchong, were products of the Yuanxun Tea Factory of the National Wuyi Mountain Nature Preservation Zone in

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Wuyishan, Fujian province, China. All tea samples were manufactured in May and June of 2004 according to the standard processes. The pine sample (*Pinus taiwanensis* Hayata) used in the smoking process of the tea was collected in the same region, that is, Tongmu village of Wuyi Mountain. The pine sample was the joint parts of the wood branches, naturally air-dried. The bark was cut off before analysis. The standard chemicals of aroma were purchased from Aldrich and Fluka.

Analyses of the Volatile Constituents of Tea Samples. Ten grams of tea sample was mixed with 500 mL of deionized water in a 1 L flask, and then the volatile constituents were extracted according to the simultaneous distillation and extraction (SDE) method using a modified Likens−Nickerson apparatus. Forty milliliters of redistilled ethyl ether was used as the extraction solvent, and the extraction was continued for 1.5 h after the sample began boiling in the flask. The resulting ether extract was dried over anhydrous sodium sulfate for 2 h. After filtration, the ether extract was concentrated at 45 °C. Twenty micrograms of ethyl decanoate (5 mg/mL in ethyl acetate) was added as an internal standard for GC quantitative analysis. A 5.00 μL sample concentrate was injected into a Kechuang GC9800 gas chromatograph equipped with a PEG-20M capillary column, 30 m $\times$ 0.25 mm i.d., and a flame ionization detector. The temperature program was maintained at 60 °C for 10 min and then risen to 180 °C at a rate of 3 °C/min and then maintained at that temperature for 30 min.

The aroma concentrates were also analyzed with GC-MS on an Agilent 6890 gas chromatograph connected with an HP 5973 mass spectrometer and a database NIST98. The column was an HP Innowax, 30 m $\times$ 0.25 mm i.d. The temperature program was maintained at 60 °C for 2 min and then risen to 240 °C at a rate of 5 °C/min. The mass conditions were EI 70 eV and scan from 35 to 400 amu.

The aroma constituents were identified with the Kovats indices and the mass spectra compared with those of authentic compounds and the literature.

Analysis of the Volatile Composition of Oleoresins in Pine Material. The pine material for smoking tea was finely cut, and 5 g was subjected to SDE treatment with 40 mL of ethyl ether as mentioned above. The aroma concentrate was analyzed with GC and GC-MS as mentioned above under the same conditions except that the column for GC-MS was an HP DB-17 and the temperature program was maintained at 60 °C for 2 min and then risen to 240 °C at a rate of 20 °C/min.

RESULTS AND DISCUSSION

Manufacturing of Tea. The manufacturing processes are quite different from those for other black teas and produce the unique flavor. The processes for these three black teas are as follows.

Manufacture of Lapsang Souchong. The process includes the following steps: plucking, solar withering, indoor withering, rolling, fermentation, fixing, second rolling, and drying. The operations are quite different from those for congou (or orthodox) black tea. The plucking standard is one bud and two leaves or sometimes one bud and three leaves in May and June. Solar withering involves spreading the leaves on a bamboo sheet under sunshine to reduce the moisture content, which is not done in the procedures for other black teas. Indoor withering is performed at ∼60 °C above the heating room, where the floor boards are wood pieces laid with slits of 5 cm to let the hot air flow through from the heating room. Such warm withering is necessary because the humidity of the air in the Wuyi Mountain area is quite high (80−86%). The tea leaves absorb the burning pine smoke in the hot air. The rolling is similar to that done for other black tea, but the fermentation is performed in bamboo baskets to maintain the temperature. The fixing step involves heating the fermented leaves by pan firing. The leaves are then subjected to a second rolling for making the shape when they are still hot. Then the leaves are dried in the heating room. For this unique drying process the leaves are put on bamboo sieves and placed 1.5 m from the ground, where there is a pipe to introduce the hot air and smoke flow from a stove beneath the ground. Pine wood (log) of the local species (*P. taiwanensis*) is used as the fuel. When the tea leaves are dried enough and have simultaneously absorbed the pine smoke, they are packed as the product lapsang souchong (*4*, *5*).

Manufacture of Smoked Lapsang Souchong. Smoked lapsang souchong is processed from lapsang souchong through a special smoking process. The process is performed in a hole placed with a well burning charcoal on the bottom and covered with pine material on the charcoal. The pine material is the joint parts of pine wood without bark and the outer part of the xylem. Such material contains a high level of oleoresin and gives high-quality of smoke. The tea is sprayed with water and then spread in a bamboo basket mounted over the hole. The volatile constituents and the thermal pyrolysis products of the pine material rise to the tea for absorbing. During the smoking process flame is avoided because there is no air flow to the hole. The pine material is heated instead of being burned.

Manufacture of Smoked Souchong. The smoked souchong is manufactured from congou black tea through the smoking process described above. Congou black tea is produced outside the Wuyi Mountain area under nonsmoking conditions.

Aroma of Tea Samples. Table 1 shows the major volatile constituents of the samples; 49 compounds were identified, including 17 alcohols, 12 phenols, 7 aldehydes, 5 alkenes, 2 ketones, 2 esters, 2 acids, 1 ether, and 1 epoxy compound. The main constituents of lapsang souchong were longifolene, geraniol, α-terpineol, (*E*)-2-hexenal, and phenylacetaldehyde, which accounted for 45.6% of the total aroma constituents. The most abundant constituents of smoked lapsang souchong were longifolene, α-terpineol, 4-methylguaiacol, geraniol, juniperol, and phenylacetaldehyde, which accounted for 44.2%. Those of smoked souchong were α-terpineol, longifolene, 4-methylguaiacol, 4-ethylguaiacol, and β-caryophyllene, which accounted for 44.4%. Longifolene and α-terpineol were the major components of volatile oils contributing to the odor of such teas. **Figure 1** shows the chromatogram of smoked lapsang souchong. The high content of longifolene has not been reported in teas. Longifolene, juniperol, and fenchol are found in tea for the first time, to our knowledge.

Kawakami et al. reported the aroma of Zheng Shan Xiao Zhong (lapsang souchong) (*6*). However, they did not identify longifolene. The difference in results is considered to be due to the difference of samples. Their sample may have been obtained through a commercial route and might have been blended and redried.

Longifolene is present in many oleoresins of pinaceous plants, especially in subsection Sylvestres (*7*). Most of the exotic pine species in China contain low concentrations of longifolene (*7*). The pines grown in Finland, Italy, Russia, Peru, Greece, and Turkey also contain low levels of longifolene (*8*−*13*). On the other hand, some local pine species in southern and eastern China, for example, *Pinus taiwanensis*, *Pinus massoniana*, and *Pinus massoniana* var. *hainanensis*, which belong to subsection Sylvestres, were proved to have high levels (9.5−12.4%) of longifolene in the oleoresins (*14*). *P. taiwanensis* is the species in the Wuyi Mountain and neighboring regions. It contains the highest concentration of longifolene in the *Pinus* genus, as we have known. The amounts of longifolene and α-terpineol were of main constituents in the volatiles of pine sample up to 30% (**Table 2**). The concentrations of longifolene and α-terpineol were also high in the volatile oils of the tea samples. Longifolene can be considered as the unique constituent in such tea.

Table 1. Contents of Volatile Constituents in Tea Samples (Micrograms per Gram of Tea)

	constituent	Kovats index	lapsang souchong	smoked lapsang souchong	smoked souchong	method of identification[a]
			From Tea Leaves			
1	hexanal	1124	3.64	2.19	0.96	St, MS
2	(*E*)-2-hexenal	1228	12.36	7.65	5.14	MS
3	(*Z*)-3-hexenyl acetate	1320	0.78	0.53	0.24	MS
4	(*Z*)-3-hexenol	1386	2.15	1.54	1.46	St, MS
5	(*E*,*E*)-2,4-hexadienal	1406	0.15	0.24	0.32	MS
6	linalool oxide I	1443	2.69	0.82	0.70	MS
7	acetic acid	1466	1.18	1.14	1.05	St, MS
8	linalool oxide II	1470	4.32	2.20	1.29	MS
9	benzaldehyde	1529	2.61	1.54	1.05	St, MS
10	linalool	1551	5.91	3.34	2.09	St, MS
11	3,7-dimethyl-1,5,7-octatrien-3-ol	1614	1.79	1.50	1.36	MS
12	phenylacetaldehyde	1651	14.84	10.02	7.84	St, MS
13	linalool oxide III	1751	2.16	1.84	1.06	MS
14	linalool oxide IV	1776	4.44	2.25	1.58	MS
15	methyl salicylate	1788	1.19	1.62	2.98	St, MS
16	geraniol	1863	16.50	11.20	10.38	St, MS
17	hexanoic acid	1868	4.82	1.87	1.32	St, MS
18	benzyl alcohol	1893	7.40	3.23	1.58	St, MS
19	2-phenylethanol	1926	4.91	2.21	1.27	St, MS
20	β-ionone	1949	1.95	1.50	1.52	St, MS
21	nerolidol	2047	3.18	2.29	1.59	St, MS
			Pine Terpenoids			
22	longicyclene	1497	3.13	4.48	3.83	MS
23	camphor	1517	0.82	1.62	0.63	MS
24	sativene	1525	0.97	1.71	1.28	MS
25	longifolene	1566	31.83	55.43	31.26	MS
26	fenchol	1585	0.73	2.57	0.93	MS
27	β-caryophyllene	1592	2.40	5.29	12.17	MS
28	1-terpinen-4-ol	1602	1.46	2.95	1.31	MS
29	α-caryophyllene	1672	1.08	3.33	1.33	MS
30	estragole	1680	1.03	0.48	1.85	MS
31	α-terpineol[b]	1706	15.57	49.43	44.08	St, MS
32	borneol[b]	1710	1.76	6.85	5.84	MS
33	caryophyllene oxide	1988	0.77	2.96	3.30	MS
34	caryophyllene alcohol	2054	0.61	0.91	1.03	MS
35	juniperol	2147	5.75	11.18	11.77	MS
			Pyrolysis Products			
36	furfural[b]	1472	3.76	4.50	2.37	St, MS
37	5-methylfurfural	1578	1.06	2.04	2.19	MS
38	guaiacol	1877	3.54	8.06	9.27	St, MS
39	4-methylguaiacol	1970	5.44	14.82	21.27	MS
40	2,3,5-trimethyl-1,4-benzenediol	1980	0.88	3.33	3.69	MS
41	phenol	2020	4.25	8.37	10.24	St, MS
42	4-ethylguaiacol	2039	3.72	8.98	15.96	MS
43	dimethylphenol	2086	1.31	2.71	2.72	MS
44	dimethylphenol	2090	1.57	8.18	12.16	MS
45	dimethylphenol	2097	3.25	6.15	7.80	MS
46	dihydroeugenol	2113	1.57	5.16	6.74	MS
47	eugenol[b]	2167	1.79	5.56	6.19	St, MS
48	2-ethyl-6-methylphenol	2170	1.08	5.95	7.34	MS
49	2-ethylphenol	2178	1.00	3.30	3.43	MS
	total		**201.10**	**297.02**	**278.76**	
	group 1, tea origin aroma		98.97	60.72	46.78	
	group 2, pine terpenoids		67.91	149.19	120.61	
	group 3, pyrolysis products		34.22	87.11	111.37	

[a] St, standard chemical; MS, mass spectrum. [b] Also from tea leaves.

α-Terpineol is also an aroma constituent from tea, but apparently most of it was from pine.

Longifolene is a product of the pine chemical industry. In a product of heavy turpentine from southern China the concentration of longifolene was as high as 58% followed by β-caryophyllene (11%) (*15*). The chemical use of longifolene is for the syntheses of, for example, β-caryophyllene (*16*), isolongifolene, and derivatives (*17*). Because of its high concentration in *P. taiwanensis*, the presence of longifolene in lapsang souchong as a main aroma constituent is reasonable.

Shen and Yang reported the burning products of volatile compounds in a smoked souchong, most of which were guaiacols, phenols, and furfurals (*18*). However, they did not report the identification of other volatile compounds.

Figure 2 shows the structures of some volatile constituents from pine.

The aroma constituents in **Table 1** can be divided into three groups according to the forming sources: (1) tea origin aroma, such as linalool and its oxides, geraniol, benzyl alcohol, 2-phenylethanol, and nerolidol; (2) pine terpenoids, which are directly from the pine wood, such as longifolene, α-terpineol, juniperol, β-caryophyllene, and longicyclene, although some are

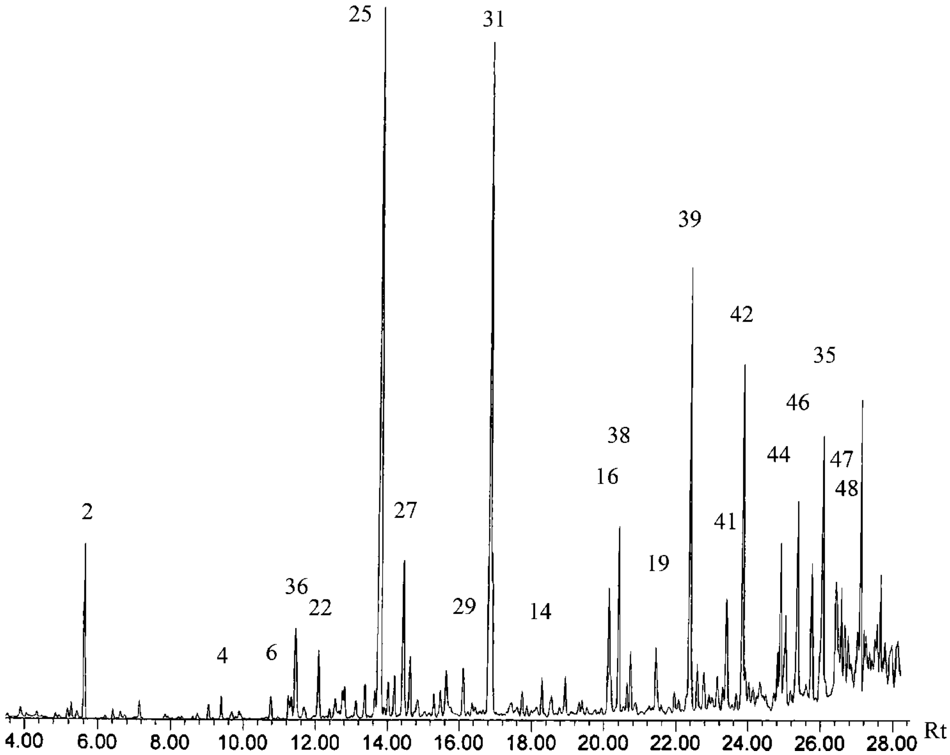

Figure 1. TIC chromatogram of the aroma of smoked lapsang souchong. Peak numbers refer to **Table 1**.

Table 2. Chemical Composition of Volatile Oil from Pine Material (*P. taiwanensis*)

constituent	RT[a]	%[b]	constituent	RT[a]	%[b]
α-pinene	3.41	42.91	α-terpineol	6.72	9.53
camphene	3.74	3.94	γ-terpineol	6.80	0.75
β-pinene	4.11	0.59	α-longipinene	7.44	0.76
limonene	4.64	1.06	longicyclene	7.67	1.20
terpinolene	5.44	0.75	sativene	7.79	0.64
fenchol	5.92	1.47	longifolene	8.05	21.22
β-terpineol	6.25	0.66	β-caryophyllene	8.11	4.66
1-terpinen-4-ol	6.54	3.02	α-caryophyllene	8.41	0.81
borneol	6.57	2.26	juniperol	9.72	0.87
camphor	6.63	0.52	**total**		**97.62**

[a] Retention time (minutes) on DB-17 column. [b] Peak area percent of the total area.

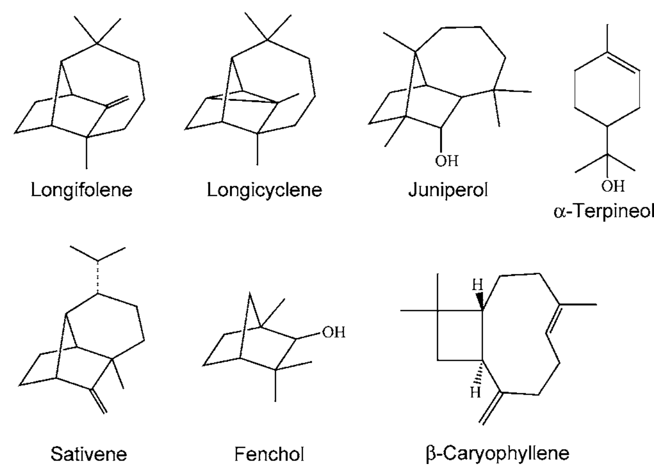

Figure 2. Some characteristic aroma constituents of the samples.

also present in tea aroma; and (3) pyrolysis products that formed from pine by burning or heating, such as phenols and guaiacols (*19*). **Table 1** shows the contents of each group, reflecting the effects of their processes. Groups 2 and 3 constituted quite large proportions in the aroma.

The quality of black tea aroma can be expressed with the volatile flavor compound (VFC) index, which is the ratio of the contents of some terpenoids, aromatics, and ionones to the contents of C5 and C6 aldehydes and alcohols (*20*). The VFC indices are, for example, 1.28−3.06 for Indian black tea and 2.51 for keemun black tea (*21*), whereas the VFC indices are 3.80, 3.67, and 4.41 for lapsang souchong, smoked lapsang souchong, and smoked souchong, respectively, calculated from **Table 1**. However, there is no evidence yet about the relationship of the VFC index to the quality of black tea from var. *sinensis*.

The sensory characteristic of lapsang souchong is the aroma of pine smoke like dried longan pulp plus the sweet note of black tea. The volatile constituents from the pine smoke contribute to the aroma of the samples. The smoked lapsang souchong can be brewed several times and still give satisfactory aroma and taste. Moreover, there is still apparent aroma remaining in the cup after the infusion is poured out. The specific quality of lapsang souchong and smoked lapsang souchong is called the "mountain flavor". Another specific property of lapsang souchong and smoked lapsang souchong is that they can be stored for years without change of their quality. Such black teas are usually drunk with sugar and milk or honey.

Comparison of the Volatile Constituents of Lapsang Souchong and Congou Black Teas. There is an obvious difference between lapsang souchong and keemun black tea, a grade of Chinese congou black tea. The main constituents of keemun black tea were geraniol, benzyl alcohol, 2-phenylethanol, linalool, linalool oxides, β-ionone, phenylacetaldehyde, (*E*)-2-hexenal, *trans*-geranic acid, methyl salicylate, (*Z*)-3-hexenol, and α-terpineol, etc. (*21*−*23*). Most of the constituents of pine terpenoids and thermal pyrolysis products of lapsang souchong listed in **Table 1** (groups 2 and 3) were not detected in keemun black tea.

A characteristic of keemun black tea (*C. sinensis* var. *sinensis*) is the relatively high ratio of geraniol to the sum of linalool and its oxides (*21*, *22*) compared to that of var. *assamica*, for example, Indian and Sri Lankan teas (*20*, *24*, *25*). The contents of geraniol and linalool showed lapsang souchong to have the feature of var. *sinensis*. However, the relatively high content of (*E*)-2-hexenal, which is usually the most abundant constituent in the aroma of var. *assamica* (*20*, *25*−*27*), showed that lapsang souchong (cv. Bohea) is not the typical var. *sinensis* like keemum black tea.

The tea leaves of lapsang souchong absorb the burnt pine aroma, whereas keemum black tea does not contact smoke. Many compounds of lapsang souchong (group 3) such as guaiacol, 4-methylguaiacol, 4-ethylguaiacol, dihydroeugenol, 2,3,5-trimethyl-1,4-benzenediol, eugenol, 2,3-dimethylphenol, 3,4-dimethylphenol, 3-methylphenol, 2-ethyl-6-methylphenol, and 2-ethylphenol are characteristic compounds of wood smoke and typical flavor constituents in smoked foods (*28*). The pine wood contained a great deal of lignin, cellulose, and hemicellulose, which are the source of the smoke. Furfural and its derivatives are produced by thermal pyrolysis of cellulose, and phenol and the derivatives are generated by thermal pyrolysis of lignin (*19*, *29*), which are also formed in tea leaves to a certain extent (*26*).

Comparison of Smoked Lapsang Souchong and Lapsang Souchong. The composition of the volatile oil from smoked lapsang souchong was similar to that of lapsang souchong, whereas their contents of the constituents were quite different. The amount of the constituents from tea leaves (group 1) in lapsang souchong accounted for 49.2% of the aroma, much higher than that in smoked lapsang souchong, which accounted for only 20.5%. On the other hand, the amount of the constituents from pine (groups 2 and 3) in lapsang souchong (33.8 and 17.0%, respectively) was less than that in smoked lapsang souchong (50.2 and 29.3%, respectively, see **Table 1**). This difference shows the effect of the smoking process.

It is clear that the constituents of the tea origin decreased with the heating of the smoking process, in which nearly all constituents of group 1 decreased, whereas the constituents from the pine smoke increased markedly, in which nearly all constituents of group 2 and all constituents of group 3 increased.

Comparison of Smoked Souchong with Smoked Lapsang Souchong. The composition of the volatile oil of smoked souchong was qualitatively similar to that of smoked lapsang souchong, and the total amount of the volatile oil of the former was a little less than the latter (**Table 1**). However, there were apparent differences between them. The contents of the volatile constituents of tea origin (group 1) in smoked souchong (16.8%) were lower than those in smoked lapsang souchong (20.5%), which showed the difference of the tea materials. The group of terpenoinds of pine origin (group 2) showed the same tendency (43.3 and 50.2%, respectively). On the other hand, the contents of the thermal pyrolysis products (group 3) were higher in smoked souchong (39.9%) than in smoked lapsang souchong (29.3%).

The quality of smoked lapsang souchong is superior to that of smoked souchong by sensory evaluation. The difference in the aroma constituents of tea origin (group 1) between these black teas may reflect the difference in quality of the tea leaves. The tea trees of lapsang souchong grow in the Wuyi mountain area, whereas those of souchong grow outside of that area.

During the smoking process the lapsang souchong absorbed more pine terpenoids than the souchong did. The contents of longifolene and α-terpineol in smoked lapsang souchong were 55.43 and 49.43 μg/g, respectively, whereas those in smoked souchong were 31.26 and 44.08 μg/g, respectively. The difference in selectivity of absorbing pine terpenoids and products of thermal pyrolysis seems to be decided by their properties. Nearly all other constituents of group 2 showed the same tendency, whereas nearly all constituents of group 3 showed the reverse tendency (**Table 1**). It seems that the high contents of the aroma of tea origin and pine terpenoids positively relate to the quality of smoked teas. The results showed that the tea grown in the Wuyi Mountain area has higher contents of aroma and higher absorbing property for the terpenoids of the pine material than the tea grown outside the mountain has.

Effect of Absorption of Volatile Constituents by Tea Leaves. The chemical composition of the volatile oil extracted according to the SDE method from the pine material is shown in **Table 2**. It contained 19 main constituents predominated by monoterpene and sesquiterpene alkenes and alcohols, which accounted for up to 97%. As the common feature of pine volatile, the most abundant constituent of the oleoresin was α-pinene, with the highest content exceeding 42%. The content of α-pinene is usually higher in oleoresin from Chinese pines. However, α-pinene was not detected in any tea sample.

There were no phenols detected in the volatile oil of the pine material. Accordingly, the phenols of pine origin in the volatile oils of the tea samples could be considered to be the thermal pyrolysis products of the pine wood (*29*, *30*).

Some volatile constituents of pine were not detected in any tea sample, such as α-pinene, camphene, limonene, β-pinene, and terpinolene. This may be attributed to their instability at high temperature or their low boiling points, causing them to volatilize readily in the process of smoking. It seems that the constituents of pine wood of relatively high boiling points or polarity are easily absorbed by the tea leaves. The differences between the smoked teas and the pine material can explain the aroma characteristic of smoked lapsang souchong, which is rich and lasting with a note like the dried longan pulp, instead of the simple combination of the flavors of black tea and pine wood.

In Chinese traditional smoke-cured meats with burning wood the most abundant aroma constituents are phenols, plus some acids, esters, aldehydes, and ketones (*31*). On the other hand, the solid and aqueous smoke flavorings are also composed of phenols as the main constituents and furans, pyrans, aliphatic aldehydes and ketones, acids, and esters, etc. (*32*−*34*). Terpenoids are very low in any example. Compared to other smoked foods, the black tea of lapsang souchong is quite specific, containing a high concentration of terpenoids from pine which is rich in longifolene. The strict requirement for species of tea plant, processing techniques, and the pine wood for smoking are the important factors conferring the unique quality of the product.

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