





The Effect of Temperature on the Aroma of Soft Stick Oolong Black Tea

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Abstract: Soft stick oolong black tea using the processing technology of Yunnan Gongfu black tea is made from Taiwan Qingxin oolong. In this study, in order to explore the effect of roasting temperature on the aroma of soft stick oolong black tea. Soft stick oolong black tea was roast at 100°C, 110°C, 120°C, 130°C temperatures, and then the aroma was determined by GC-MS. Our results showed that soft stick oolong black tea had the most volatile compounds and the highest sensory evaluation score at the baking temperature 120°C.


1 INTRODUCTION


Tea (*Camellia Sinensis* (L.) O. Ktze.) belongs to shrubs or small trees. China is the origin of tea (Lu 2013). Initially, it exerted its medicinal value. Legend recored that, Shennong tasted tea. Tea has been used to clear heat and quench thirst, diuresis and strengthen the heart. Oolong tea is mainly distributed in Fujian and Taiwan, which has a high aroma and low bitterness. It is often used to make black tea and green tea, and the produced tea has a unique aroma. An important factor influencing the quality of oolong tea is aroma, and processing techniques have different effects on the aroma components of oolong tea (Su 2019).


Gongfu black tea is named from its extremely labor-intensive production process. And Gongfu black tea is well-known in the world for its "high fragrance, bright color and strong taste" (Mi 2013). The production process is not complicated, but the control of the production process is very important. Withering is the process of losing some water, volatilizing grass gas, and affecting enzyme activity. The degree of withering can affect the quality of black tea. The process of rolling is the result of the


coordination of chemical and physical interactions, in which black tea has begun to ferment, and different chemical reactions have occurred. It is also the process of black tea shaping, rolling into a firmer curled shape, increasing the rate of cell damage and promoting oxidation reaction. The production process of Yunnan Gongfu black tea is wilting → rolling → fermentation → drying → fragrance, which is also a way to stimulate the aroma after the black tea has been dried (Fan, et al., 2020, Feng, et al., 2017). There are baking, hot wind, far infrared, microwave, light wave and other fragrant ways. But in Yunnan, baking is generally used to increase fragrance, because the boiling point of aromatic substances is different, the temperature of baking and fragrance will change accordingly. In recent years, consumers have much demanding on the quality of black tea. How to improve the aroma and taste of black tea has always been a hot research topic for black tea (Liu, et al., 2015).

The so-called "soft branch" refers to an oolong species introduced from Taiwan, also known as Qingxin Oolong. It was originally introduced to Taiwan by a century-old mother tree of dwarf oolong from Jian'ou City, Fujian Province. It belongs to a small-leaf species with a small tree shape. It is an open-type dwarf variety with small leaves and dense branches. The buds are initially purple and the leaves are oblong. The thick mesophyll makes it extremely rich in nutrition and

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aroma substances, which are suitable for making many different fermented teas. Soft branch black tea is made from the fresh leaves of soft branch oolong tea and the processing technology of Yunnan Kungfu black tea. Because the oolong tea has a higher aroma, making it into black tea can improve the aroma. Therefore, soft branch black tea is very popular nowadays.

Baking treatment is one of the important post-treatment processes of tea, which has an important effect on improving the quality of tea (Wen, et al., 2018). In recent years, Yunnan Gongfu black tea flavoring technology has been continuously developed, and the commonly used baking flavoring technology has also been continuously improved. The market has increasingly higher requirements for the aroma of black tea. Therefore, the related research and improvement on the standards for black tea fragrance enhancement are needed. Zheng Silin (Zheng 2016) studied the processing technology of the Qingxin Oolong variety Summer and Autumn black tea introduced in Sichuan, showed that the black tea made with Qingxin Oolong is of good quality. Although it is different from the processing technology of Yunnan Gongfu black tea, it also has a guiding significance for Yunnan Oolong Gongfu black tea. However, the current research results on the aroma of soft-branch black tea are lacking, the research on the fragrance standard needs to be perfected, and there is no research specifically aimed at the aroma of soft-branch oolong black tea. Shi Daliang (Shi et al. 2018) studied the drying methods of Qingxin Oolong Gongfu black tea. The results proved that Qingxin Oolong Gongfu black tea had the best quality under the conditions of 120 °C first firing and 90 °C full firing. This study explored aromatic substances in soft stick oolong black tea to find out aroma-stimulating temperatures. Then through the evaluation of the tea critics, we want to find optimal time and temperature for soft stick oolong black tea processing, which will have certain guiding significance for the production of soft stick oolong black tea.

2 MATERIALS AND METHODS

2.1 Materials and Instruments

Material: The soft stick oolong black tea purchased in the market has uniform material quality and has not been treated with fragrance after drying.

Instrument: American Agilent 6890-5973 Gas Chromatography Mass Spectrometer (GC-MS).

Manual solid phase microextraction instrument PC-420D, Supelco, USA; Extraction head 65 μ m, PDMS/DVB, Supelco, USA.

2.2 Sample Preparation

Divide the material into 4 groups, each group is 500g and set 3 repetitions for 60 minutes. The temperature of control group was set at 100°C; the temperature of other three groups were set at 110°C (I), 120°C (II) and 130°C (III). Spread the fragrance and cool it in the same room. The indoor temperature is 20-23°C, and the relative humidity of the air is 70%-80%. After spreading to room temperature, put it in a No. 10 ziplock bag for sealing, and the air is full of self-sealing. After the bag is sealed, the fragrance is prevented from being lost after the gas exchange with the outside (Luo et al. 2016).

2.3 GC-MS Conditions

The solid phase microextraction (SPME) method is used to collect volatile substances. Use a clean dissecting needle to poke a small hole about 5cm below the top of the ziplock bag, insert a 0.5mm length of 65 μ m PDMS/DVB solid phase microextraction head manual SPME sampler into the ziplock bag, headspace extraction and adsorption for 1 hour, sampling. After the end, it was transferred to the gas chromatograph mass spectrometer for injection (Zhang, et al., 2020).

An Agilent 7890A-5975C gas chromatography-mass spectrometer was used to analyze plant volatiles. Capillary column HP-5MS (30m \times 0.25mm, 0.25 μ m), the carrier gas is high-purity helium, and the flow rate: 1.0mL \cdot min⁻¹. Heating program: the initial temperature is 50°C, the temperature is raised to 280°C in 10 minutes, and after waiting 5 minutes, the temperature is lowered to 50°C, and the instrument runs for 52 minutes after sample injection. The solvent is delayed by 2 min. The ion source temperature is 230°C, and the quadrupole temperature is 150°C. The detector temperature is 280°C, and the inlet temperature is 220°C (Zhang, et al., 2020, Ma, et al., 2019, Chen, et al., 2021, Xie, et al., 2019).

2.4 Materials and Instruments

After completing the GC-MS test, take the sample to the tea review room for sensory review and scoring. The sensory review of black tea is carried out with reference to GB/T23776-2018 "Tea Sensory

Evaluation Method" (GB/T23776-2018), and the determination of sensory attributes refers to GB/T 14487-2017 "Tea Sensory Evaluation Terminology" (GB/T14487-2017), which was reviewed by 5 professionals. Sensory evaluation of tea leaves is conducted, and the scores are based on the average of the tea judges to find the group with the best taste.

2.5 Data Analysis

Delete all compounds with matching degree <80 and silicon-containing oxides. Query, identify and analyze the compound components in the samples by CAS number, and use Microsoft Excel 2019 software to carry out the data statistical analysis of aroma components (Zhang et al. 2020, Ma et al. 2019, Liu et al. 2021, Zhou et al. 2011).

3 RESULTS AND ANALYSIS

3.1 Analysis of Aroma Components of Soft-branch Oolong Black Tea

A total of 113 volatile compounds were identified in the 4 treatments by GC-MS. The control group identified 56 volatile substances, including 12 olefins, 10 alcohols, 8 alkanes, 7 aldehydes, and 6 aromatic hydrocarbons. The contents of various volatiles were 21.4%, 17.9%, 14.3%, 12.5%, and 10.7% respectively. Group A has identified 55 volatile substances, including 17 olefins, 11 alkanes, 8 aromatic hydrocarbons, and 4 alcohols. The contents of various volatiles were 30.9%, 20.0%, 14.6%, and 7.3%, respectively. Group B has identified 66 volatile substances, including 21 olefins, 18 alkanes, 10 aromatic hydrocarbons, and 4 alcohols. The contents of various volatiles were 31.8%, 27.3%, 15.2%, and 6.1%, respectively. Group C has identified 46 volatile substances, including 18 olefins, 11 alkanes, 9 aromatic hydrocarbons, and 2 alcohols. The contents of various volatiles were 39.1%, 23.9%, 19.6%, and 4.3% respectively. The 4 treatments mainly include 23 kinds of alkanes, 31 kinds of olefins, 13 kinds of aromatic hydrocarbons, 12 kinds of alcohols, 10 kinds of aldehydes, 5 kinds of ketones, 3 kinds of esters, 4 kinds of acids, and 2 kinds of oxygen heterocycles, 3 kinds of nitrogen heterocycles, 3 kinds of ethers, 2 kinds of sulfides and 2 kinds of other substances (Table 1,2,3).

With the increase of flavoring temperature, the proportion of olefins and aromatic hydrocarbons gradually increases, and the proportion of alcohols

gradually decreases. It shows that with the increase of the flavoring temperature, most of the volatile substances in soft stick oolong black tea are converted into olefins, and alcohols are decomposed into other volatile substances.

Table 1: The total fraction of aroma substance in soft stick oolong black tea.

Varieties	Total fraction			
	control	I	II	III
Alkanes	8	11	18	11
Olefins	12	17	21	18
Aromatic hydrocarbons	6	8	10	9
Alcohols	10	4	4	2
Aldehydes	7	4	6	3
Ketones	3	3	1	1
Esters	3	0	1	0
Acids	2	2	0	0
Oxygen heterocycles	1	1	1	0
Nitrogen heterocycles	2	0	1	1
Ethers	0	2	1	0
Sulfide	1	2	1	1
Other	1	1	1	0
Total	56	55	66	46

The relative content of alkanes with more than 10 carbon atoms (Dodecane, Tetradecane, Undecane, Tridecane, 3-methyl-) decreases with the increase of the flavoring temperature, while those with less than 10 carbon atoms The relative content of alkanes (Nonane, Decane) increases with the temperature, indicating that long-chain alkanes are decomposed into short-chain alkanes at high temperature. Myrcene is made from linalool as the raw material. The relative content of myrcene gradually increases with the fragrance temperature, while the relative content of linalool has a downward trend with the increase of the fragrance temperature. It shows that with the increase of fragrance temperature, a large amount of linalool is converted into myrcene. beta-Pinene and (1S)-L-β-Pinene are both intermediates in the manufacture of flavors and fragrances. As the flavoring temperature increases, their relative content tends to increase, indicating that as the flavoring temperature increases Some of the elevated substances are converted into beta-Pinene and (1S)-L-β-pinene (Table 2).

Table 2: Percentage of aroma substances in soft stick oolong black tea.

Varieties	Percentage (%)			
	control	I	II	III
Alkanes	14.3%	20.0%	27.3%	23.9%
Olefins	21.4%	30.9%	31.8%	39.1%
Aromatic hydrocarbons	10.7%	14.6%	15.2%	19.6%
Alcohols	17.9%	7.3%	6.1%	4.3%
Aldehydes	12.5%	7.3%	9.1%	6.5%
Ketones	5.4%	5.5%	1.5%	2.2%
Esters	5.4%	0	1.5%	0
Acids	3.5%	3.6%	0	0
Oxygen heterocycles	1.8%	1.8%	1.5%	0
Nitrogen heterocycles	3.5%	0	1.5%	2.2%
Ethers	0	3.6%	1.5%	0
Sulfide	1.8%	3.6%	1.5%	2.2%
Other	1.8%	1.8%	1.5%	0
Total	100%	100%	100%	100%

3.2 Sensory Review

Compared with the control group, different temperatures had no effect on the cord and clarity, but slightly affected the color, evenness, tea liquor color, tea taste and infused leaves of the dry tea, and had a greater impact on the flavor and height of the tea aroma (Table 3-4).

Table 3: Sensory evaluation of shape.

Titian treatment	Shape- Sensory evaluation (score)			
	Cord	Color	Evenness	clarity
control	82.8	85.4	86.2	86.0
I	82.8	85.4	86.2	86.0
II	82.8	85.4	86.2	86.0
III	82.8	84.8	83.6	86.0

Table 4: Sensory evaluation of quality.

Titian treatment	Quality - Sensory evaluation (score)				overall ratings
	Tea liquor color	Tea aroma	Tea taste	infused leaves	
control	85.2	83.0	83.6	85.0	84.65
I	85.8	84.8	83.0	85.0	84.88
II	85.6	89.4	82.6	85.0	85.38
III	84.0	84.0	80.8	82.6	83.58

Proper high-temperature fragrant treatment will make the black tea liquor color red slightly, and the tea liquor color will become turbid after over-

baking. The unflavored black tea tastes mellow, and the flavored black tea has different degrees of high fire flavor. Over-baked black tea will cause throat discomfort. In the sensory evaluation, the aroma gradually changed from rock and flower aromas to sweet and sugary aromas. After the caramel aroma, a burnt smell would appear.

In summary, with the increase of temperature, soft stick oolong black tea has the most volatile substances and compounds at 120°C, and the sensory evaluation score is the highest.

4 DISCUSSION AND CONCLUSIONS

The aromatic substances in tea are organic compounds. So far, more than seven hundred aroma substances have been separated from tea (Zhang, et al., 2008). To date, more than 400 aroma components have been detected in black tea (Zhang et al. 2019, Shi 2010, Wan 2003). Alcohols, esters, aldehydes, ketones, alkenes, alkanes, etc. are the main aroma substances in black tea, among which linalool, nerol, methyl salicylate, nonanal and phenylacetaldehyde are among the aroma components. The content of black tea is higher (Wang, et al., 2013, Li, et al., 2021).

According to the source of aroma, it can be divided into tea aroma and aroma produced during production; most aromas contain unsaturated double bonds, which are chemically active and volatile. Aroma is not only an important factor in determining the quality of tea, but also an important indicator for distinguishing the quality of different tea leaves (Ye, et al., 2018).

With the increase of fragrance temperature, alcohols such as linalool show a downward trend. Some previous studies have shown that alcohols are the key aroma substances for the aroma of black tea. For example, Li Jun (Li, et al., 2021) found that 2,6-Octadien-1-ol, 3,7-dimethyl-, (E)-, 1,6-Octadien-3-ol, 3,7-dimethyl-, Benzyl Alcohol, Benzaldehyde, and Acetic acid are the key aromas compound of Guizhou black tea. Liu Yang (Liu, et al., 2021) found that alcohols and aldehydes are the most important aroma components in tribute eyebrows. Peng Yun (Peng, et al., 2021) showed that 1,6-Octadien-3-ol, 3,7-dimethyl- and its oxides are characteristic aroma components in Yunnan black tea. In order to explore the reasons for the formation of the special aroma and flavor of Sichuan Qingxin Oolong black tea, Luo Xueping used SPME-GC-MS

combined technology and found that the main aroma-contributing components of Sichuan Qingxin Oolong black tea were alcohol compounds, with a content of 58.20% (Luo, et al., 2021). Lin Yanping (Lin, et al., 2021) found that the aroma components of Wuyi black tea "Jinjunmei" were mainly alcohols, hydrocarbons, esters, aldehydes and ketones. Zhou Senjie (Zhou, et al., 2021) research found that the aroma components among the tender, fresh and high-grade Longjing tea types are alcohols, aldehydes, terpenes, ketones, alkanes, alkenes, heterocycles and esters and other compounds. There are differences in species and relative content. 3-methylpentane, 3-ethylpentane, myrcene, linalool, nerol, trans-2-hexene and α -terpinene are the key aroma compounds of Dianhong (Shu, et al., 2022). Xu Yuanjun (Ge, et al., 2015) research shows that the content of alcohols, alkanes, lipids and ketones in floral black tea is higher

In summary, with the increase of the flavoring temperature, alcohols will be decomposed and converted into other substances. It is not that the higher the flavoring temperature, the better quality of tea. The soft stick oolong black tea has an extremely rich aroma and overall best quality after 60 minutes flavoring at 120°C. The aroma substances of tea have a great relationship with the production process. Consumers' demand for the aroma of tea is still increasing. Detecting the aroma of different production processes is conducive to the standardization and improvement of the tea production process. Soft branch oolong is made into black tea. It is an innovation. Different Titians will produce different aromas or different aroma ratios. There is still a lot of research space for the aroma of soft branch oolong black tea.

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