

# Roasted Sesame Flavor Baijiu

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The sesame flavor Baijiu was accidentally discovered after the founding of the People's Republic of China.

Sesame flavor Baijiu is known for its special aroma of roasted sesame, which attracts people's attention.

sesame flavor Baijiu

volatile compounds

odorants

## 1. Introduction

As the name implies, sesame flavor Baijiu is known for its special roasted sesame aroma. Flavor quality is one of the most important factors that people consider when choosing Baijiu <sup>[1]</sup>. As reported, the flavor quality of Baijiu is mainly dependent on the trace components in Baijiu. Although water and ethanol are the main components of Baijiu, the difference in the types and concentrations of trace components in different Baijiu flavors are the key factors that cause a difference in Baijiu flavor quality <sup>[2]</sup>.

## 2. Qualitative Analysis of Trace Components in Sesame Flavor Baijiu

The formation of sesame flavor Baijiu was late, thus, the research on the trace components of sesame flavor Baijiu was carried out later than strong, light, and sauce flavor Baijiu. In 1957, sesame flavor was occasionally discovered in Jingzhibai <sup>[3]</sup>. In 1965, paper chromatography was used to analyze the trace components of Jingzhibai, which was the earliest exploration into sesame flavor Baijiu <sup>[4]</sup>. With the progress of science and technology, an increasing number of techniques and methods formed, which greatly promoted research on sesame flavor Baijiu. In 1991, the triangle theory was put forward by Hu <sup>[5]</sup>, and it was established that the style and characteristics of sesame flavor Baijiu were different from strong, light, and sauce flavor Baijiu. In 1994, the research on the sulfur compound in sesame flavor Baijiu was carried out <sup>[6]</sup>. Consequently, 3-methylthiopropyl alcohol was identified by a GC-SPD combined with a standard comparison, and these were supposed to be the characteristic components of sesame flavor Baijiu. The research laid a foundation for the analysis of trace components in sesame flavor Baijiu, and the confirmation of sesame flavor Baijiu. Based on this research, in 1995, the former Light Industry Federation of China issued the industry standard of sesame flavor Baijiu (National Standard Number QB/T 2187-1995). Of note, 3-methylthiopropyl alcohol was identified as the characteristic component of sesame flavor Baijiu and the concentration of 3-methylthiopropyl alcohol in sesame flavor Baijiu was specified to be greater than or equal to 0.4 mg/L. With the establishment of industry standard, research on the analysis of trace components in sesame flavor Baijiu was gradually developed, which promoted the formation of the national standard for sesame flavor Baijiu. In 2006, the national standard of sesame flavor Baijiu (National Standard Number GB/T 20824-2007) <sup>[7]</sup> was officially

promulgated, stipulating that the concentration of 3-methylthiopropanol in sesame flavor Baijiu should be no less than 0.5 mg/L for a high alcoholicity sample and no less than 0.4 mg/L for a low alcoholicity sample. Thus, sesame flavor Baijiu was widely recognized by Chinese people, which greatly promoted the development of research on its trace components.

In the inception phase, researchers mainly focused on the qualitative analysis of trace components in sesame flavor Baijiu. In 2009, GC–FID was used to analyze the trace components of Shengliyuan (a kind of sesame flavor Baijiu) and 50 kinds of trace components were found by spectral library retrieval [8].

However, due to the limitations of techniques and methods, it was difficult to guarantee the accuracy of qualitative results and there were fewer trace components detected; thus, the trace components of sesame flavor Baijiu could not be fully explored. With the enrichment of techniques and the increase of personnel input, the number of trace components identified in sesame flavor Baijiu increased rapidly, and the accuracy was also greatly improved. In 2012, a total of 120 trace components were detected from two kinds of Meilanchun samples using HS–SPME combined with GC–MS. It includes 48 esters, 20 alcohols, 16 aldehydes and ketones, 15 organic acids, 3 alkanes, 2 phenols, 5 sulfur compounds, 8 nitrogen compounds, and 3 polyols [9]. In 2014, the trace components of Jingzhibaigan were analyzed using LLE combined with GC–MS [10]. The results showed that a total of 65 trace components were extracted with three kinds of solvents, including 9 alcohols, 19 esters, 11 acids, 14 hydrocarbons, 3 aromatic compounds, 3 furans, 3 aldehydes, 1 ketone, 1 kind of nitrogen compound and 1 sulfur compound. In 2017, 241 compounds were identified by DI and LLE in sesame flavor Baijiu, including 62 esters, 59 hydrocarbons, 24 acids, 32 alcohols, 10 furans, 8 aldehydes, 6 ketones, 25 aromatic compounds, 8 sulfur compounds, 6 nitrogen compounds, and 4 other compounds [11].

With the expansion of research, researchers found that during actual production, the quality of sesame flavor Baijiu fluctuated greatly. Although the sample of sesame flavor Baijiu reached the national standard, it did not have the typical roasted sesame aroma. It was speculated that 3-methylthiopropanol had no direct correlation with the typical roasted sesame aroma of sesame flavor Baijiu. Of note, the 3-methylthiopropanol had a typical meat and onion fragrance, which was not consistent with the characteristic aroma of sesame flavor Baijiu. Based on this, it was generally doubted whether the single compound of 3-methylthiopropanol could be identified as the characteristic component of sesame flavor Baijiu in the national standard of sesame flavor Baijiu (National Standard Number GB/T 20824-2007). This problem greatly restricted the development of sesame flavor Baijiu; therefore, the exploration of characteristic trace components in sesame flavor Baijiu has become the focus of research. Although the concentrations of sulfur compounds and nitrogen compounds in sesame flavor Baijiu is relatively low, both of them have a strong flavor releasing effect due to their extremely low odor threshold values [12], which are thought to play important roles in the formation of the characteristic flavor of Baijiu. Sesame flavor Baijiu has a special aroma of roasted sesame, similar to the flavor characteristics of coffee and sesame. Of note, sulfur compounds and nitrogen compounds exist in coffee and sesame seeds, and make an important contribution to the formation of coffee and roasted sesame aromas [13][14]; hence, sulfur and nitrogen compounds are presumed to be the characteristic components of sesame flavor Baijiu due to their similar aromas of roasted sesame. Thus, it

is necessary to study sulfur and nitrogen compounds in sesame flavor Baijiu to reveal what contribution of sulfur and nitrogen compounds make to the formation of the characteristic flavor of sesame flavor Baijiu.

In 2012, LLE and SPME coupled with GC–MS was conducted to extract sulfur compounds in Guojing sesame flavor Baijiu, and 4 sulfur compounds were identified [15], including 3-methylthiopropanol, ethyl 3-methylthiopropionate, dimethyl trisulfide, and ethyl methylthioacetate. In 2014, a total of 31 nitrogen compounds were confirmed in sesame flavor Baijiu by LLE combined with GC–MS and a GC–NPD [16], including 14 pyrazine compounds, 1 pyrrole, 4 pyridines, 1thiazole, 1 oxazole and 2 other compounds. In 2016, SPME and LLE combined with GC–MS was used to conduct qualitative analysis on sulfur compounds in sesame flavor Baijiu [17]. Consequently, a total of 14 sulfur compounds were detected.

### 3. Quantitative Analysis of Trace Components in Sesame Flavor Baijiu

On the basis of the qualitative analysis for the trace components in sesame flavor Baijiu, in order to determine the concentrations of important trace components, researchers carried out quantitative analysis on the trace components in sesame flavor Baijiu. Researchers mainly focused on 3-methylthiopropanol as stipulated by the national standard, establishing corresponding quantitative analysis methods in accordance with the national standard. Thus, the concentration of 3-methylthiopropanol in sesame flavor Baijiu from different regions was determined. In 2014, a GC–FPD was used to monitor the concentration of 3-methylthiopropanol in sesame flavor Baijiu [18]. With the development of research, the sensitivity and accuracy of the method for the determination of 3-methylthiopropanol in sesame flavor Baijiu has been greatly improved. In 2015, a method for the determination of 3-methylthiopropanol in sesame flavor Baijiu by gas chromatography-mass spectrometry/selective ion scanning (GC–MS/SIM) combined with the external standard method was established [19]. The limit of detection was 5 µg/L and the limit of quantitation was 10 µg/L. Based on the results, it was also proven that 3-methylthiopropanol has no relationship with the typical sesame flavor of Baijiu.

Of note, not all base samples of sesame flavor Baijiu contained 3-methylthiopropanol. In addition, the concentrations of 3-methylthiopropanol in base samples were significantly different. The concentrations of 3-methylthiopropanol in some base samples were much lower than the concentration specified in the national standard. In addition, 3-methylthiopropanol also exists widely in chi flavor Baijiu and fruit wine. Among them, the concentration of 3-methylthiopropanol in sesame flavor Baijiu was similar to that in chi flavor Baijiu, but lower than that in fruit wine. It is further indicated that 3-methylthiopropanol was not the characteristic component of sesame flavor Baijiu. Recent studies have shown that there are many trace components in sesame flavor Baijiu, thus, the characteristic flavor of sesame flavor Baijiu may be formed by the interaction of different trace components. Based on the above results, it is not reasonable for the national standard of sesame flavor Baijiu (National Standard Number GB/T 20824-2007) to regard the single compound of 3-methylthiopropanol as the characteristic component of sesame flavor Baijiu. This research promotes the enhancement and revision of the national standard for sesame flavor Baijiu. Other important trace components in sesame flavor Baijiu were also quantitatively analyzed. In 2012, Meilanchun was analyzed by HS–SPME and GC–MS [20], which found that the concentration of

tetramethyl pyrazine was 1.5~4.4 times higher than that of the same flavor Baijiu. In 2018, DI and GC–MS were used to analyze the other sulfur compounds in different brands of sesame flavor Baijiu [21]. Five sulfur compounds were detected and quantified by internal standard method.

## 4. Screening and Evaluation for the Aroma Compounds of Sesame Flavor Baijiu

In a further study, the researchers found that not all trace components were directly related to the flavor formation of sesame flavor Baijiu; hence, screening and evaluation of aroma compounds from thousands of trace components became a novel research focus.

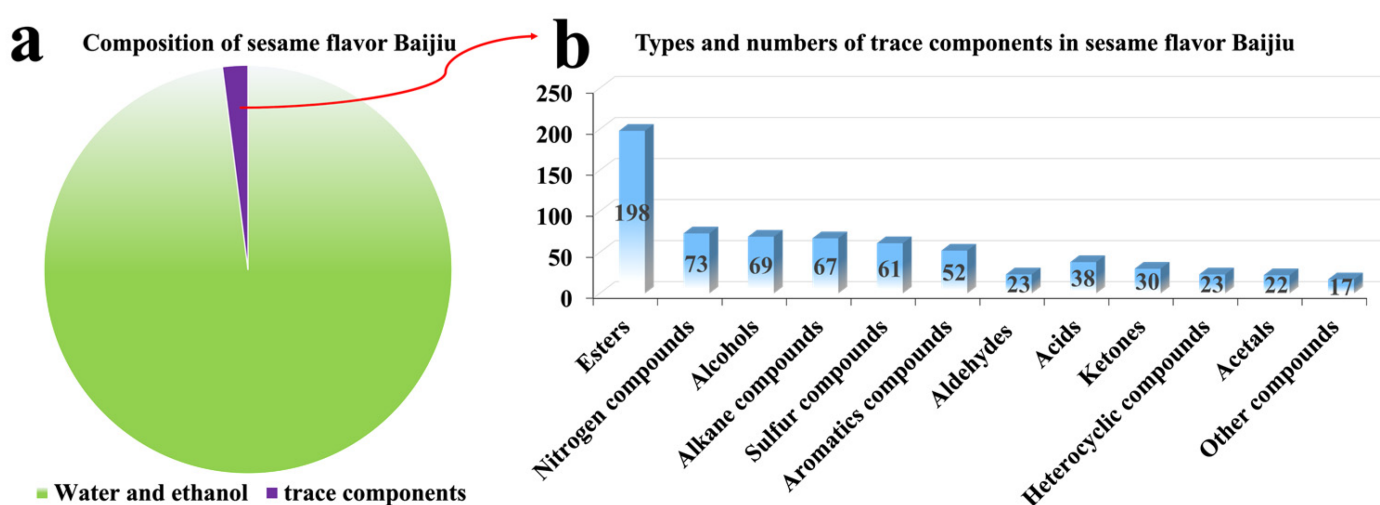
In 2011, Fan et al. measured the olfactory thresholds of 79 trace compounds in Baijiu under the same substrate conditions [22]. It has promoted research investigating the expression of the aroma compounds.

In 2015, LLME and HS–SPME coupled with a GC–FID were used to screen and evaluate the aroma compounds in sesame flavor baijiu [23]. As a consequence, 87 aroma compounds in 36 kinds of sesame flavor baijiu with different brands, grades, and production years were quantitatively analyzed. Among them, 15 kinds of aroma compounds with an OAV greater than 100 were screened out and identified as important aroma compounds in sesame flavor baijiu, including 9 esters, 1 aldehyde, 1 alcohol, 2 sulfur compounds, 1 acid and 1acetal. Among them, esters mainly showed the fruit aroma, and pyrazines primarily contributed to the roasted aroma. Whereas sulfur compounds expressed the odor of rotten vegetables, alcohols mainly contributed to the aroma of grass. With the introduction of molecular sensory omics, the research on the necessity of aroma compounds for the aroma profile of sesame flavor baijiu has been gradually developed. In 2016, aroma compounds in Jingzhi were screened out and evaluated [24]. The aroma expression of 56 aroma compounds were further determined by AEDA combined with an OAV value calculation. The importance of ethyl hexanoate and methional to the overall aroma of sesame flavor baijiu was further confirmed by an omission experiment. On this basis, the aroma compounds of Jingzhi were further investigated by HS–SPME–AEDA [25]. As a result, the research showed that ethyl hexanoate, 2-furfurylthiol, dimethyl trisulfide, 3-methylbutanal, ethyl butanoate, ethyl 2-methylbutanoate, ethyl pentanoate, and ethyl 4-methylpentanoate were confirmed with higher FD factors. In particular, 8 sulfur compounds were identified as being potentially important to sesame flavor Baijiu. As a result, the concentration of these aroma compounds was further quantified by the internal standard method, then 36 aroma compounds were found to be above the corresponding odor threshold. On the basis of the sensory evaluation, 2-furfurylthiol (OAV = 1182), dimethyl trisulfide (OAV = 220),  $\beta$ -damascenone (OAV = 116), and methional (OAV = 99) could be the main source of the unique aroma of sesame flavor Baijiu.

In addition, the screening and evaluation of aroma compounds in other brands of sesame flavor Baijiu has also been gradually carried out. In 2018, 92 kinds of aroma compounds were detected in Meilanchun [26], among which 35 kinds of aroma compounds had OAVs  $\geq 1$ , then an omission experiment showed that methional and dimethyl trisulfide played an important role to the flavor formation of sesame flavor Baijiu. After that, research was aimed to study the aroma compounds in Baotuquan (a kind of sesame flavor Baijiu) [27]. Consequently, 75 kinds of trace

components in Baotuquan were detected by GC–MS; and finally, ethyl hexanoate, ethyl butanoate and 3-methylbutanal were further confirmed as important odorants after an omission experiment. In 2019, Li et al. explored the key aroma compounds in Guojing, and identified benzyl mercaptan for the first time [28], which makes an important contribution to the unique aroma of sesame flavor Baijiu. It had a strong roasted sesame aroma, thus, it was presumed to be the key aroma compound of sesame flavor baijiu.

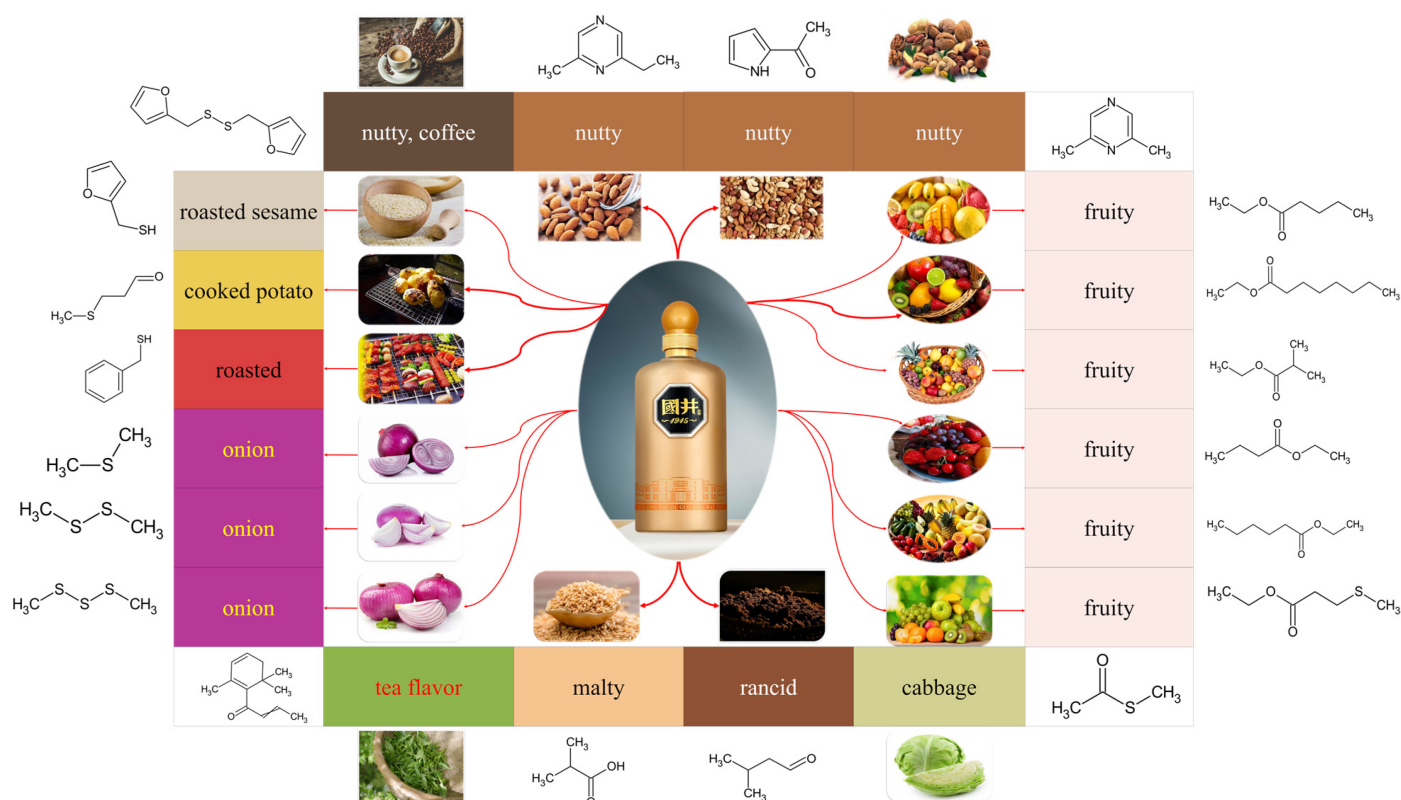
So far, a total of 673 trace components have been identified in sesame flavor Baijiu, including 198 esters, 69 alcohols, 38 acids, 23 aldehydes, 30 ketones, 22 acetals, 73 nitrogen compounds, 61 sulfur compounds, 23 heterocyclic compounds, 52 aromatic compounds, 67 alkane compounds and 17 kinds of other compounds (as shown in Figure 1).



**Figure 1.** Types and numbers of trace components in sesame flavor Baijiu.

Among them, esters contribute to the fruity, flowery, and sweet aroma. Although sesame flavor Baijiu is characterized by its roasted sesame flavor, which is significantly different from other flavors of Baijiu, its main aroma compounds are still esters. Esters play an important role in the aroma profile for sesame flavor Baijiu. Of note, ethyl hexanoate (FD factor = 4096, OAV = 2691), ethyl butanoate (FD factor = 2048, OAV = 447), ethyl pentanoate (FD factor = 2048, OAV = 1019), and ethyl octanoate (FD factor = 25, OAV = 782), are the main aroma compounds of esters in sesame flavor Baijiu. Alcohols are the precursors of esters, and they mainly contribute to a fruity and fatty aroma. The common alcohols in sesame flavor Baijiu are 1-propanol (FD factor = 100, OAV = 6) and 2-methyl-1-propanol (FD factor = 8, OAV = 4). Acids contribute to a sour taste in the flavor profile of sesame flavor Baijiu, thus affecting the taste and the aftertaste of sesame flavor Baijiu. The lack of acids in sesame flavor Baijiu is the main reason for the poor aftertaste. Butanoic acid (FD factor = 1024, OAV = 57), pentanoic acid (FD factor = 256, OAV = 46), and hexanoic acid (FD factor = 2048, OAV = 35) are deemed to be the main acids in sesame flavor Baijiu. Aldehydes and ketones are also important aroma compounds in sesame flavor Baijiu and they contribute to a herbal and buttery aroma. For instance, 3-methylbutanal (FD factor = 8, OAV = 2403) and  $\beta$ -damascenone (OAV = 116) are recognized as important aroma compounds in sesame flavor Baijiu. Acetals such as 2-furaldehyde diethyl acetal (FD factor = 4) primarily contribute to a grassy, fruity, and sweet aroma.

Heterocyclic compounds such as 2-acetyl furan (FD factor = 25) and 2-acetyl-5-methyl furan (FD factor = 5) mainly contribute to a nutty and sweet aroma in sesame flavor Baijiu. Phenolic compounds such as phenol (FD factor = 4), 4-methylphenol (FD factor = 1, OAV = 43), and 4-ethyl-2-methoxyphenol (FD factor = 4, OAV = 2) mainly contribute to a smoky aroma. Of note, the presumed reason why sesame flavor Baijiu differs from other flavor types of Baijiu is because of its sulfur compounds and nitrogen compounds (as shown in **Figure 2**). Sulfur compounds mainly expressed an aroma of roasted incense, scorched incense, coffee incense, whereas nitrogen compounds mainly presented nutty aromas, sweet aromas, and roasted aromas; therefore, sulfur compounds and nitrogen compounds are thought to be the characteristic aroma compounds of sesame flavor Baijiu.



**Figure 2.** The key aroma components for the characteristic aroma of sesame flavor Baijiu.

Subsequently, 76 kinds of aroma compound were deemed to be responsible for the aroma profile of sesame flavor Baijiu through use of the aroma expression and static sensory evaluation method. Of note, a total of 7 sulfur compounds and 11 nitrogen compounds made a significant contribution to the flavor characteristics of the roasted sesame aroma. More specifically, benzyl mercaptan exhibits a roasted taste. Dimethyl disulfide and dimethyl trisulfide exhibit an aroma of onion, cabbage, meat, and corn. Methional exhibits the aroma of roasted potato. Furfurylthiol exhibits the aroma of coffee, onion, roasted, sesame oil, and burnt incense. Difurfuryl ether also exhibits the aroma of coffee and roasted sesame. Ethyl 3-methylthio propionate exhibits a fruity aroma. Pyrazines (such as 2,6-dimethylpyrazine, 2-ethyl-6-methyl-pyrazine, 2-acetyl-pyrrole) primarily contribute to the nutty aroma.

At the same time, the multivariate analysis method has also been applied to the screening and evaluation of aroma compounds in sesame flavor Baijiu. In 2018, Sun et al. applied principal component analysis (PCA) to analyze the

aroma compounds in sesame flavor Baijiu. The result showed that 6 compounds have higher PC1 and PC2 weight scores [\[21\]](#). Indeed, the application of this method on the aroma compounds in Baijiu also has broad prospects for development.

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