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Analysis of Aroma Compound Characteristics During Spreading and Cooling of Nongxiang Baijiu

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Abstract

Spreading and cooling is a crucial process in strong-flavor (Nongxiang) Baijiu production, during which volatile aroma substances are emitted and closely related to Baijiu flavor formation and brewing resource utilization. In this study, dynamic sampling was used to collect gaseous substances at six time nodes (0-2 min to 10-12 min) during the spreading and cooling process in a semi-automatic workshop of a southwest Chinese strong-flavor Baijiu distillery. Gas chromatography-mass spectrometry (GC-MS) combined with mass spectrometry (MS) and retention index (RI) was applied for qualitative and semi-quantitative analysis of gas components, and the dynamic changes of key aroma substances were analyzed by referencing the Wuliangye flavor wheel. The results showed that a total of 83 aroma substances were identified, belonging to seven categories including aromatic compounds, alcohols, aldehydes and acids, with aromatic compounds and aldehydes being the dominant components; the gas was characterized by grain, baking and sour aromas. Ten key aroma substances of strong-flavor Baijiu were screened, and their contents exhibited obvious dynamic changes with spreading and cooling time, generally increasing first and then decreasing, peaking at about 6 min and rebounding slightly at 10-12 min (possibly due to hot water vapor sedimentation). Cluster analysis indicated that the initial and middle stages differed mainly in aroma substance content, while the late stage was distinguished by the comprehensive effect of various aroma substances. This study established a preliminary method for collecting and analyzing spreading and cooling gas, and revealed the composition and dynamic change rules of its aroma compounds, providing a theoretical basis for the resource recovery of brewing by-products and the construction of a green low-carbon production mode for strong-flavor Baijiu.

Keywords

Nongxiang Baijiu; Spreading and Cooling Process; Gas Characteristics.

1. Introduction

The Baijiu industry is a traditional industry in China with a development history of more than 3,000 years [1, 2], playing a pivotal role in the construction and development of China's national economy. Among the diverse categories of Baijiu, strong-flavor Baijiu occupies a dominant position, accounting for 51% of China's Baijiu market share in 2021 [3]. In accordance with GB/T 10781.1-2021 *Quality Requirements for Baijiu - Part 1: Strong-Flavor Baijiu*, the production process of strong-flavor Baijiu adopts the brewing technique of "combined steaming and firing, and continuous grain fermentation", in which the spreading and cooling process is an indispensable and extremely important link. This process cools the high-temperature fermenting grains to a temperature suitable for pit entry [1]. During this period, blowers force air through the distiller's grains to lower their temperature to a specific range within a controllable time, while a large amount of gas is emitted into the spreading and cooling pit house. In addition to water vapor, the emitted gas contains a number of aroma substances.

A large quantity of gaseous substances released during spreading and cooling belongs to volatile organic compounds (VOCs) [4, 5] that need to be controlled in the field of environmental protection. VOCs are the main carbon-containing organic substances involved in atmospheric photochemical reactions and are classified as gaseous pollutants. Relevant literatures have pointed out that the unorganized volatile gas emissions from distilleries cause a certain degree of malodorous pollution, affecting the ambient air quality, and their sensory odor concentration has exceeded the national standard values[6]. However, researchers have so far qualitatively identified 861 trace components including esters, acids, alcohols, aldehydes and ketones in strong-flavor Baijiu[7]. Relevant studies have also shown that the volatile substances emitted during the spreading and cooling process are mainly derived from fresh grains, residues after the last distillation, and flavor-forming components produced during fermentation[8, 9] . These volatile organic compounds participate in forming the unique style of Baijiu and cannot be simply classified as environmental volatile pollutants[10]; to a certain extent, they are by-products of Baijiu production. Establishing a method for collecting volatile gases during spreading and cooling is conducive to the efficient resource recycling of brewing by-products and the construction of a green, low-carbon and environmentally friendly production mode[11].

Based on a semi-automatic production workshop of a strong-flavor Baijiu distillery in Southwest China, this study adopted dynamic sampling to collect the gases emitted during the spreading and cooling process[12], and analyzed the differences in gas composition at different time periods of the process, aiming to provide a theoretical basis for the recovery and utilization of gaseous substances during the spreading and cooling of strong-flavor Baijiu brewing.

2. Materials and Methods

2.1. Consumables and Instruments

Constant flow gas sampler EM-500 (Shenzhen Guoji Instrument Co., Ltd.); 1 L aluminum foil gas sampling bag (Hunan BKM Bio-tech Co., Ltd.); Tenax-TA adsorption tube 6.35×89 mm (Anhui Chutong Experimental Equipment Co., Ltd.); Nitrogen blow concentrator MD200-1 (Hangzhou AoSheng Instrument Co., Ltd.); Gas chromatography-mass spectrometry (GC/MS) 7890N-5975MSD (Agilent Technologies, USA); Agilent 122-7162 capillary column (60.0 m×0.25 mm×0.25 μm).

2-Octanol (chromatographically pure), n-amyl acetate (chromatographically pure), 2-ethylbutyric acid (chromatographically pure) (Tianjin Guangfu Fine Chemical Research Institute); Methanol (purity ≥ 99.9%), ethanol (purity ≥ 99.5%), C7-C30 n-alkane mixture, n-pentane (purity ≥ 99.0%), ethyl laurate, 1-heptanol, acetic acid, propionic acid, butyric acid, valeric acid, caproic acid, heptanoic acid, caprylic acid, capric acid, 2-phenylethyl caproate, 2-acetylfuran, 2-acetyl-5-methylfuran (all chromatographically pure) (Sigma-Aldrich Life Science, USA); 4-Methylvaleric acid, benzaldehyde (both chromatographically pure) (Aladdin Biochemical Technology Co., Ltd., Shanghai); Ethyl acetate, ethyl propionate, ethyl butyrate, ethyl linoleate, 1-butanol, 2-pentanol, 1-hexanol, 1-pentanol, nonanoic acid, 2-methylpropionic acid, phenylacetic acid, ethyl benzoate, phenethyl alcohol, phenol, p-cresol, 2-methylpropanal, hexanal, 2-pentanone, acetoin, 2,6-dimethylpyrazine, furfural, furfuryl alcohol (all chromatographically pure) (TCI (Shanghai) Development Co., Ltd.).

2.2. Sample Collection Method

The air inlet of the constant flow gas sampler was connected to a 50 mL glass funnel, and the air outlet was connected to a 1 L aluminum foil gas sampling bag. Sampling was carried out at the grain distiller's grains inlet of the automatic spreading and cooling machine for 2 min until the sampling bag was full. After sampling, the 1 L sampling bag was connected to the air inlet of the constant flow sampler, and a Tenax-TA adsorption tube was connected to the air outlet. The gas in the sampling bag was released until the sampler stopped, completing the adsorption. The adsorption capacity of the Tenax-TA adsorption tube for the spreading and cooling gas was 1 L per time. After gas adsorption, 100 μL of a mixed triple internal standard was added dropwise to the Tenax-TA adsorption tube. Both ends of the tube were wrapped with food-grade plastic wrap and stored in a refrigerator at -4 °C, and GC-MS analysis was performed within 24 hours.

It is worth noting that there are significant differences between different production batches of Baijiu. Therefore, this study only took a single spreading and cooling batch as an example. Due to the particularity of the spreading and cooling gas, only one sample could be collected for a single time period by specific equipment with dynamic sampling, and no parallel samples were set in this study. Whether this method is suitable for the general research of spreading and cooling gas remains to be further verified, so the research method in this study is only a preliminary attempt.

Table 1. Gas Substance Collection Scheme at Different Time Nodes during Spreading and Cooling Process

Sample name	Sampling start time	Sampling duration/min	Notes
W1	Start of the operation of spreading and cooling equipment	2	Gaseous substances at 0-2 min of spreading and cooling
W2	2 min of spreading and cooling		Gaseous substances at 2-4 min of spreading and cooling
W3	4 min of spreading and cooling		Gaseous substances at 4-6 min of spreading and cooling
W4	6 min of spreading and cooling		Gaseous substances at 6-8 min of spreading and cooling
W5	8 min of spreading and cooling		Gaseous substances at 8-10 min of spreading and cooling
W6	10 min of spreading and cooling (process completed)		Gaseous substances at 10-12 min of spreading and cooling

2.3. Instrument Analysis Conditions

GC conditions: Injector temperature was set at 250 °C; high-purity helium was used as the carrier gas with a flow rate of 1.2 mL/min; splitless injection mode was adopted. The column temperature program was as follows: initial temperature 40 °C held for 2 min, then increased to 250 °C at a rate of 4 °C/min and held for 5 min.

MS conditions: Electron ionization (EI) source with an electron energy of 70 eV; ion source temperature 230 °C; quadrupole temperature 150 °C; solvent delay 1 min; mass scan range 35~350 m/z.

2.4. Instrument Analysis Conditions

The identification of volatile components was carried out through the following strategies: first, preliminary qualitative analysis of unknown substances was conducted based on the NIST database, with 244 compounds initially identified in this study. Then, the mass spectra of the compounds were compared with the standard spectra in the NIST20 standard database, and a matching degree > 700 was regarded as the preliminary qualitative result (MS). The retention index (RI) of each compound was calculated and compared with the reported RI values in literatures. Data analysis was performed using SPSS 23.0, Python 3.5 and scikit-learn, and plotting was conducted with Origin and matplotlib.

Semi-quantitative analysis was performed using the triple internal standard method (the mass concentrations of 2-octanol, n-amyl acetate and 2-ethylbutyric acid were 37.218 mg/L, 21.238 mg/L and 41.890 mg/L, respectively) [13]. The semi-quantitative concentration was calculated according to the ratio of the peak area of the unknown substance to that of the three adjacent standard substances.

3. Results and Analysis

3.1. Analysis of Gas Components during Spreading and Cooling

The production of strong-flavor Baijiu adopts the combined steaming and firing technique, in which grain raw materials are mixed with mature fermenting grains from the previous batch and steamed bran, followed by steaming. High-temperature distillation is carried out during steaming to collect the distillate [14]. During spreading and cooling, the volatile flavor substances originally enclosed in the high-temperature steamed grain distiller's grains are gradually released with the decrease in temperature and molecular motion.

The overall aroma characteristics during spreading and cooling are dominated by grain aroma, baking aroma and sourness. A total of 83 compounds were detected in the volatile gases of the process, mainly including aromatic compounds, alcohols, acids, ketones and esters.

For toluene, ethylbenzene and other aromatic compounds in the spreading and cooling gas, the pungency of the odor decreases with the increase in substituents, and the aroma becomes softer and richer, adding a unique aromatic foundation to the spreading and cooling aroma. The detected benzyl alcohol has almond and nut aromas, and acetophenone has a certain fruit aroma. Benzyl alcohol also has a faint almond and floral aroma [15], which is soft and long-lasting without obvious pungency. These aromatic gases endow the spreading and cooling gas with a certain sense of hierarchy. However, the overall floral aroma of the spreading and cooling gas is not obvious in the actual production workshop[8], while the baking aroma similar to cooked rice is more prominent. Toluene, benzaldehyde and other substances have all been reported in the analysis of steamed five-grain powder components, indicating that these aroma substances present in the steaming process of five-grain powder are potential sources of grain aroma in Wuliangye Baijiu and belong to recoverable and recyclable aroma components[8, 11] .

Among the alcoholic compounds detected in the spreading and cooling gas, 1-octanol, 1-hexanol and 1-pentanol have all been detected in Baijiu by various methods in previous literatures[16], but their specific contents in this study are different from those reported. The synergistic aroma effect of alcohols constitutes the core foundation of the baking and grain oil aroma characteristics of the spreading and cooling gas[8]. Aldehydes are the most diverse category of aroma components. Unsaturated fatty aldehydes, furans, lactones and other substances enter the Baijiu body during distillation, thus affecting the Baijiu body. Furfural is formed by the cyclization of pentoses such as D-xylose and oligosaccharides after losing one water molecule under high temperature conditions[17]. The furfural in this study may be converted from pentosans in the husks of sorghum, corn and other grains under the action of high-temperature water vapor during the combined steaming process, with a strong caramel and nut aroma and a slight burnt bitter taste.

Ethyl caproate, detected in the spreading and cooling gas, is the iconic ester and the main aroma component of strong-flavor Baijiu, with a rich pineapple, banana and pit mud aroma[15], which is mellow and long-lasting. In the spread and cooled grain distiller's grains, its aroma can mask some off-flavors and endow the grain distiller's grains with a "typical pit mud aroma", which is one of the key indicators for judging the gas quality during the spreading and cooling stage.

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Table 2. Approximately 83 different compounds were detected in the volatile gases during the cooling process of the pile-up

Retention time/min	name	CAS	Concentration (mg/L)
	Aromatic compounds(20)		
10.716	Toluene	108-88-3	1.615±0.272
13.515	Ethylbenzene	100-41-4	0.022±0.006
13.731	o-Xylene	95-47-6	0.025±0.016
14.015	Benzene, 1,3-dimethyl-	108-38-3	0.018±0.005
14.528	Benzene, 1,2,3-trimethyl-	526-73-8	0.003*
17.315	Styrene	100-42-5	0.179±0.018
18.527	Benzene, 1,4-diethyl-	105-05-5	0.022±0.006
19.366	alpha.-Methylstyrene	98-83-9	0.062±0.018
26.386	Benzaldehyde	100-52-7	0.319±0.030
29.079	Benzaldehyde, 4-methyl-	104-87-0	0.020±0.002
29.203	Benzoic acid, methyl ester	93-58-3	0.058±0.006
29.727	Benzaldehyde, 2-methyl-	529-20-8	0.024±0.002
30.403	Acetophenone	98-86-2	0.603±0.071
33.374	Ethanone, 1-(4-methylphenyl)-	122-00-9	0.027±0.003
33.748	1-Propanone, 1-phenyl-	93-55-0	0.011±0.004
35.631	Ethanone, 1-(4-ethylphenyl)-	937-30-4	0.289±0.081
37.233	Benzyl alcohol	100-51-6	0.553±0.057
40.539	Phenol	108-95-2	0.086±0.021
42.404	p-Cresol	106-44-5	0.009±0.003
44.637	Phenol, 4-ethyl-	123-07-9	0.123±0.033
	Alcohols(10)		
17.001	1-Pentanol	71-41-0	0.207±0.106
21.397	1-Hexanol	111-27-3	0.010*
24.675	1-Hexanol, 4-methyl-	2364-51-4	0.045*
25.069	1-Hexanol, 2-ethyl-	104-76-7	0.289±0.055
27.249	1-Octanol	111-87-5	0.073*
29.438	1-Undecanol	112-42-5	0.216±0.155
29.702	1-Nonanol	143-08-8	0.065±0.011
34.688	(S)-(+)-1,2-Propanediol	4254-14-6	0.225
36.240	Quinoline	91-22-5	0.043±0.004
37.986	2-Propanol, 2-methyl-	75-65-0	0.051*
	Aldehydes (15)		
5.631	Propanal	123-38-6	0.013±0.001*
5.850	Propanal, 2-methyl-	78-84-2	0.033±0.186
6.671	2-Propenal	107-02-8	0.007*
6.849	Butanal	123-72-8	0.022±0.003
7.603	Butanal, 3-methyl-	590-86-3	0.118±0.2185
8.992	Pentanal	110-62-3	0.042
15.171	Heptanal	111-71-7	0.025*
18.714	Octanal	124-13-0	0.102
21.686	Nonanal	124-19-6	0.255±0.141
22.298	Methylal	109-87-5	0.223
24.181	Decanal	112-31-2	0.122±0.019
25.073	1,2,4-Methenoazulene,decahydro-1,5,5,8a-tetramethyl-,[1S(1.alpha.,2.alpha.,3a.beta.,4.alpha.,8a.beta.,9R*)]-		0.331±0.164
25.144	3-Furaldehyde	498-60-2	0.243±0.048
26.617	Naphthalene, 1,2-dihydro-	447-53-0	0.270± 0.022
31.659	Naphthalene	91-20-3	0.057±0.006
	Acids(9)		
26.452	Acetic acid	64-19-7	0.807±0.171
29.405	Propanoic acid, 2,2-dimethyl-	75-98-9	0.105±0.023
29.502	Propanoic acid, 2-methyl-	79-31-2	0.075±0.011
30.993	Butanoic acid	107-92-6	0.206±0.046
31.912	Butanoic acid, 3-methyl-	503-74-2	0.153±0.025

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Continue Table 2			
34.658	Pentanoic acid	109-52-4	0.074±0.014
35.372	Butanoic acid, 2-methyl-	116-53-0	0.058*
36.532	Hexanoic acid	142-62-1	0.384±0.101
42.064	Octanoic acid	124-07-2	0.120±0.037
	Esters(8)		
7.053	Ethyl Acetate	141-78-6	0.657±0.119
8.955	n-Propyl acetate	109-60-4	0.034*
9.174	Acetic acid ethenyl ester	108-05-4	0.022*
11.251	Carbonic acid, ethyl-, methyl ester	623-53-0	0.218±0.041
16.595	Hexanoic acid, ethyl ester	123-66-0	0.071±0.022
21.599	Propanoic acid,2-hydroxy-,ethyl ester	97-64-3	0.172±0.112
21.649	Formic acid, hexyl ester	629-33-4	0.024*
25.070	Acetic acid, methyl ester	79-20-9	0.014±0.001
	Ketones (10)		
4.366	2-Pentanone	107-87-9	0.053*
6.820	Acetone	67-64-1	0.582±0.079
7.624	2-Butanone	78-93-3	0.063±0.009
8.304	2-Butanone, 3,3-dimethyl-	75-97-8	0.146±0.013
9.856	Methyl Isobutyl Ketone	108-10-1	0.897±0.125
14.205	3-Heptanone	106-35-4	0.003*
25.309	2-Propanone, 1-hydroxy-	116-09-6	0.031±0.017
28.467	Cyclobutanone,2-(1,1-dimethylethyl)-	3053-79-0	0.014*
28.972	Isophorone	78-59-1	0.117±0.032
21.531	Acetoin	431-03-8	0.041*
	Others(11)		
3.302	(2-Aziridinylethyl)amine	1943-83-5	0.013±0.004
4.154	2-Butene	107-01-7	0.221±0.023
4.387	Cyclopentene	142-29-0	0.004*
4.548	Ethyl ether	60-29-7	0.009±0.002
6.772	Ethene, ethoxy-	109-92-2	0.020±0.004
9.084	Cyclohexene, 1-ethyl-	1452-73-9	0.010*
9.152	Propene	115-07-1	0.235±0.185
9.924	Trimethylene oxide	503-30-0	0.198±0.108
14.292	Furan, 3-methyl-	1708-61-8	0.011±0.001
23.080	Furan, 2-methoxy-	25414-22-6	0.003±0.001
47.673	Glycerin	56-81-5	0.165±0.074

Note: The concentration of gaseous substances during spreading and cooling is the mean and standard deviation of 6 collected samples from the same batch; the concentration of some gaseous substances in samples was < 0.001 mg/L, so they were not included in the statistical description.

The detection of ethyl caproate in the workshop spreading and cooling link further indicates that the free ethyl caproate in raw materials directly enters the Baijiu body during grain steaming[18]. The flavor formation of Baijiu is a complex multi-factor and multi-level process. The fatty and sour aroma of caproic acid blends with the pit mud aroma of ethyl caproate, forming a synergistic effect of sour and pit mud aromas.

Aldehydes and ketones in the spreading and cooling gas are the most diverse and abundant category of bound aroma substances. Meanwhile, relevant literatures[19] have reported that 2,3-dimethyl-2-cyclohexen-1-one and 3,4-dimethyl-2-cyclohexen-1-one exhibit grain astringency and an odor similar to steamed rice during sensory evaluation, which may be related to the grain aroma in Baijiu, but these substances were not detected by the sampling method in this study. Butanone and heptanone were also detected in the flavor of steamed raw materials, further indicating that they are derived from the steaming aroma of raw materials[9].

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There are some differences between the substances detected in this study and those reported in relevant literatures, which is due to the differences in the sampling method of spreading and cooling gas, research objects and experimental environment in this study compared with the above literatures. In actual production, the spreading and cooling link is carried out after grain steaming and Baijiu distillation. Due to the need for rapid cooling, it is carried out in a semi-open and spacious environment, with more uncertain interference factors than laboratory conditions. Therefore, the results of this study can reflect the changes in aroma substances during the spreading and cooling process in the actual production link to a certain extent.

3.2. Analysis of the Changes in Key Aroma Substances

A wide variety of gases are released during the spreading and cooling process. To quickly identify the differences in key gases during the process, 10 aromatic organic compounds that positively contribute to the aroma of Wuliangye Baijiu [20, 21] were screened by comparing the components of the gas emitted during spreading and cooling with the Wuliangye flavor wheel, including ethyl caproate, ethyl acetate, 1-hexanol, isovaleraldehyde, benzaldehyde, furfural, caproic acid, butyric acid, acetic acid and acetoin. According to the flavor descriptors of the Wuliangye flavor wheel for these aromatic organic compounds, the overall flavor characteristics of the spreading and cooling aroma are summarized as grain aroma, baking aroma and acid-ester aroma, which are somewhat similar to the actual sensory experience of the spreading and cooling process.

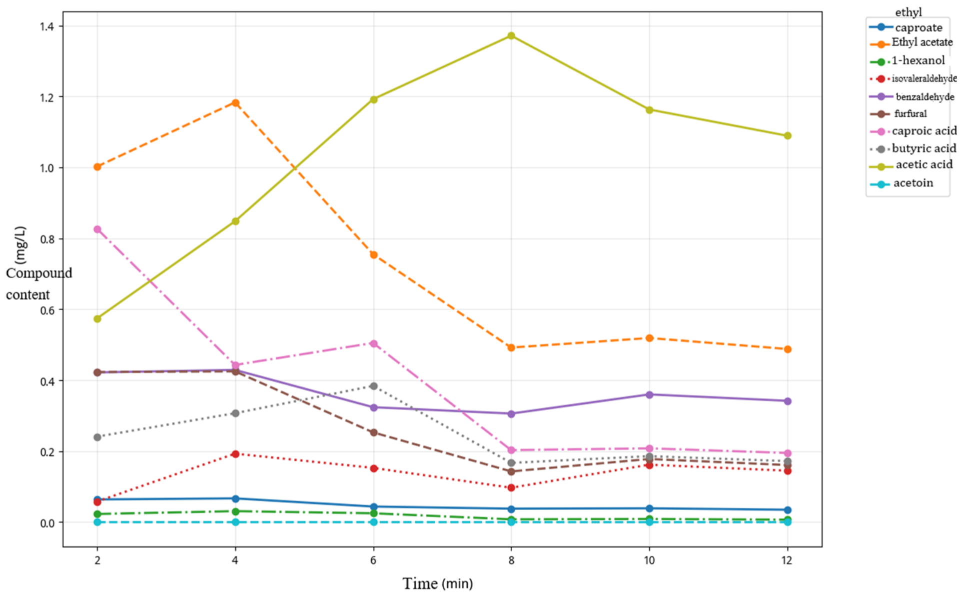

Figure 1. Changes in key compound content at different time points

As shown in Fig. 1, the content of ethyl caproate showed an obvious decreasing trend with time, dropping continuously from 0.206 mg/L at the initial stage of spreading and cooling to subsequent time points, with the contents at every 2-minute interval being 0.064 mg/L, 0.067 mg/L, 0.044 mg/L, 0.038 mg/L and 0.039 mg/L, respectively, and the content remained at a low level in the later stage of spreading and cooling. The content of ethyl acetate increased first and then decreased, reaching a peak of 1.183 mg/L in the middle stage of spreading and cooling, then gradually declined, with the contents in the middle and later stages being 0.755 mg/L, 0.492 mg/L and 0.519 mg/L in turn, and the content at the final stage was slightly higher than that at the initial time point. The content of 1-hexanol fluctuated with an increasing-then-decreasing trend over time.

There were certain differences in the change trends of isovaleraldehyde, benzaldehyde and furfural contents. The content of isovaleraldehyde increased significantly and then fluctuated and decreased, rising from 0.024 mg/L at the initial stage of spreading and cooling to a peak of 0.193 mg/L in the middle stage, then dropping to 0.153 mg/L and further to 0.097 mg/L, with a slight rebound to 0.162 mg/L at the final stage of spreading and cooling. The content of benzaldehyde increased first and then stabilized overall, with the initial content of 0.268 mg/L rising to a peak of 0.429 mg/L in the middle stage, followed by a slight drop, and remaining between 0.324~0.360 mg/L in the later stage of spreading and cooling. The change trend of furfural content was similar to that of benzaldehyde, with the initial content of 0.180 mg/L rising rapidly to 0.425 mg/L, then dropping to 0.253 mg/L and continuing to fall to 0.143 mg/L at the final stage, which was slightly lower than the initial value.

Caproic acid, butyric acid and acetic acid were detected as organic acids, all showing dynamic changes with time to varying degrees. The content of caproic acid reached a peak of 0.827 mg/L at the 2-4 min stage of spreading and cooling, significantly higher than that at other time points, dropped to 0.443 mg/L after 2 min, then rebounded to 0.505 mg/L, and then fell again to 0.203 mg/L and 0.208 mg/L, with the content at the final stage close to the initial value at the start of spreading and cooling. The content of butyric acid increased first and then decreased with time, reaching a peak of 0.384 mg/L after the start of spreading and cooling, then dropping to 0.167 mg/L and 0.186 mg/L at 8-12 min. The content of acetic acid continued to rise and then slightly declined, increasing gradually from 0.211 mg/L at the initial stage, reaching a peak of 1.371 mg/L at 8 min of spreading and cooling, then slightly falling to 1.163 mg/L, showing a significant overall increasing trend.

The contents of these eight substances all rebounded slightly in the later stage of spreading and cooling, which may be due to the decrease in the temperature of grain distiller's grains and the reduction in the quality of grain distiller's grains on the conveyor belt in the later stage of spreading and cooling by the automatic spreading and cooling equipment, and the falling back of high-heat water vapor blown into the air, resulting in a slight increase in the content of collected organic substances.

Acetoin, which contributes to the aroma of base Baijiu with a fresh and soft flavor, was only detected at the initial stage of spreading and cooling and not in the subsequent stages[22]. This may be due to the low content of this substance during the spreading and cooling stage and the high wind power of heat dissipation equipment in the semi-open spreading and cooling environment, leading to the failure to collect this substance in the subsequent spreading and cooling stages.

4. Conclusion

In this study, a constant flow gas sampler was used to collect and adsorb the gas emitted during the actual workshop spreading and cooling link of strong-flavor Baijiu, and GC-MS was used to analyze the composition of spreading and cooling gas at different time periods. Combined with MS+RI analysis, the characteristics of spreading and cooling gas at different time periods and their effects on Baijiu aroma were determined.

(1) After analyzing the composition of spreading and cooling gas at different time periods, 83 aroma substances were identified in all samples combined with literatures, including 20 aromatic compounds, 10 alcohols, 15 aldehydes, 9 acids, 8 esters, 10 ketones and 11 other heterocyclic substances. The analysis of gas composition at different time periods of spreading and cooling showed that aromatic compounds and aldehydes accounted for a relatively high proportion in the spreading and cooling gas.

(2) 10 aroma substances including ethyl caproate, ethyl acetate, 1-hexanol, isovaleraldehyde, benzaldehyde, furfural, caproic acid, butyric acid, acetic acid and acetoin were screened by

comparing with the Wuliangye flavor wheel. The key aroma substances were supplemented by combining with literatures related to the combined steaming of grain distiller's grains, and the changes of key aroma substances were analyzed. The results showed that with the increase in spreading and cooling time, the content of aroma substances increased first and then decreased. The key aroma components at the 2-6 min stage of spreading and cooling were similar, and the overall content of key aroma substances was relatively abundant. The content of aroma substances reached the highest at about 6 min after the start of spreading and cooling, and slightly rebounded at the late stage of 10-12 min, which may be related to the sedimentation of hot air. The cluster analysis results of key aroma substances showed that the main difference between the initial and middle stages of spreading and cooling was the content of aroma substances, and the difference between the late stage and the previous stages was caused by the comprehensive effect of various aroma substances.

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