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Original Research Article

Comparative Analysis of Different Sampling Methods for Gases Generated During Spreading and Cooling of Nongxiang Baijiu

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Abstract: Open spreading and cooling of solid-state fermented grains for strong-aroma Baijiu releases uncollected volatile aromas, resulting in flavor loss and air pollution. Four sampling methods (a constant-flow sampler and 300 W, 260 W, 220 W fans) were used to collect waste gas from a Yibin distillery; samples were enriched by Tenax-TA and analyzed via GC-MS. The constant-flow sampler afforded better peak resolution but lower total peak area (2.3×10^9). The 300 W fan captured the most volatiles (4.6×10^9 peak area). Moreover, the grain feeding inlet showed the highest collection efficiency. Constant-flow sampling fits laboratory qualitative analysis, while 300 W fan parameters guide industrial exhaust recovery, with the feed inlet recommended as the optimal sampling site.

Keyword: Nongxiang Baijiu, Spreading and Cooling Procedure, Volatile Flavor Compounds, Gas Sampling, GC-MS.

1. INTRODUCTION

As one of the six world-famous distilled spirits, Baijiu is a unique traditional Chinese distilled liquor carrying thousands of years of brewing craftsmanship and traditional Chinese food culture, integrating traditional manufacturing techniques and dietary civilization closely. Conventional solid-state Baijiu brewing follows the core philosophy of *co-fermentation by nature and manual operation*, consisting of sequential processes including raw material pretreatment, Daqu starter making, grain steaming, spreading and cooling, pit fermentation, distillation, blending and formulation, forming multiple typical flavor types such as Nongxiang, Qingxiang, Jiangxiang and Mixiang [1]. Nevertheless, traditional Baijiu production heavily relies on experienced brewers' sensory evaluation and manual manipulation. A large quantity of water vapor and volatile organic compounds are fugitively emitted during the open spreading and cooling stage of fermented grains, leading to waste of energy and valuable flavor substances alongside environmental burdens. Such drawbacks restrict the green production, standardization and high-quality development of modern Baijiu manufacturing industry.

The predominant flavor substances of brewed Nongxiang Baijiu originate from brewing raw/auxiliary materials, Daqu starters and pit mud [4]. Brewing raw materials serve as nutritional substrates for microbial metabolism during fermentation, and abundant aroma precursors bound in raw materials transform into characteristic flavor components via enzymatic reactions or distillation procedures. Previous research identified 35 kinds of bound aroma substances covering alcohols, aldehydes & ketones, organic acids, aromatics, terpenoids and heterocyclics from six common brewing cereals including sorghum, corn, barley, wheat, glutinous rice and ordinary rice; these bound compounds convert into sensory-active ingredients through microbial catalysis or thermal distillation [5]. Furthermore, aroma extract dilution analysis verified that ethyl esters, lactones, sulfur-containing compounds and most aromatic components detected in five-grain steamed condensate consistently exist in finished Nongxiang Baijiu, confirming tight correlation between steaming-derived volatiles and liquor flavor profile [6]. Relevant monitoring technologies have been developed for fermentation exhaust: a customized testing system combining condensation dehumidification and infrared sensors realized continuous dynamic monitoring of CO_2 and CH_4 throughout solid-state fermentation cycles, supporting greenhouse gas control and waste gas resource utilization in Baijiu factories [7]. Portable GC-based online detection methods were established to quantify key flavor volatiles from fermented grain exhaust, enabling quantitative evaluation of fermentation

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maturity according to gaseous metabolites [8]. Additionally, mixed gaseous compositions generated during Xiaoqu Qingxiang Baijiu fermentation were proven feasible as novel physical indicators to assess solid fermentation quality by monitoring component varieties and concentration variations [9].

Currently, integrated equipment for collection and recycling of spreading/cooling aroma volatiles remains unavailable in industrial production, making adsorptive capture feasible by retrofitting adsorption modules on closed spreading-cooling devices. Adsorption technology is characterized by high efficiency, cost-effectiveness and environmental friendliness for volatile recovery. Three mainstream ambient gas sampling methodologies are widely applied: direct sampling, active pump-driven dynamic sampling and passive sampling [10]. Direct sampling employs gas-tight syringes, inert sampling bags or SUMMA canisters for high-concentration gaseous analytes. Dynamic sampling uses vacuum pumps to pass target gas through solid adsorbents such as activated carbon, silica gel and Tenax, with target organics retained on adsorbent media for subsequent analysis.

The core purpose of spreading and cooling is to cool high-temperature steamed fermented grains down to the optimal temperature for pit entry [11]. Forced air blowing by industrial fans accelerates heat dissipation, accompanied by massive exhaust discharge loaded with water vapor and diversified flavor compounds into open workshop space. Quantitative collection of target volatiles via direct in-situ adsorption is challenging under ambient open conditions due to uncontrollable ambient airflow disturbance. Aluminum foil gas bags feature fixed volume and chemical inertness, enabling quantitative collection and short-term storage of spreading exhaust. In this study, exhaust gas was collected into calibrated aluminum foil bags using different sampling devices followed by Tenax-TA adsorption; GC-MS analysis was performed on adsorbed extracts to compare component differences among sampling schemes and select optimal protocols for laboratory testing and industrial recovery. This work aims to establish preferable sampling strategies targeting both academic laboratory analysis and scaled-up industrial recovery of volatiles from Baijiu spreading and cooling waste gas.

2. MATERIALS AND METHODS

2.1 Sample Source and Experimental Instruments

All gaseous samples were collected from an experimental five-grain Nongxiang Baijiu production workshop of a liquor enterprise in Yibin, Sichuan Province, strictly complying with on-site actual spreading-cooling technological parameters to guarantee sample representativeness.

Table 1: List of Main Instruments and Equipment

Equipment Name	Manufacturer
MD200-1 Nitrogen Blowing Instrument	Aosheng Instrument Co., Ltd., Hangzhou, China
7890B-5977B GC-MS System	Agilent Technologies, USA
Capillary Chromatographic Column (60.0 m×0.25 mm×0.25 μm)	Agilent Technologies, USA
EM-500 Constant-Flow Air Sampler	Guoyi Instrument Co., Ltd., Shenzhen, China
1 L Aluminum Foil Gas Sampling Bag	Bikman Biotechnology Co., Ltd., Hunan, China
Tenax-TA Adsorption Tube (6.35 mm×89 mm)	Chutong Experimental Equipment Co., Ltd., Anhui, China
4-inch 220 W Ventilation Fan	Wugen Qiyue Electrical Appliance Firm, China
6-inch 260 W Ventilation Fan	Wugen Qiyue Electrical Appliance Firm, China
6-inch 300 W Ventilation Fan	Wugen Qiyue Electrical Appliance Firm, China

2.2 Gas Sampling Protocols for Spreading and Cooling Exhaust

Four distinct sampling strategies were designed as listed in Table 2. Gaseous samples were firstly stored in 1 L aluminum foil bags, then adsorbed onto Tenax-TA tubes with controlled gas flow; pretreated adsorbent tubes were preserved under low temperature prior to GC-MS measurement.

Table 2: Detailed Sampling Schemes

Code	Sampling Power Equipment
a	Portable constant-flow gas sampler
b	300 W small-sized ventilation fan
c	260 W medium-sized ventilation fan
d	220 W medium-sized ventilation fan

Scheme a: Portable Constant-Flow Sampler Sampling:The inlet of constant-flow sampler was connected with a 50 mL glass funnel, and the sampler outlet was linked to a 1 L aluminum foil bag. Sampling was implemented for 2 min at the fermented grain feeding inlet of automatic spreading-cooling machine

until full inflation of the sampling bag. After collection completion, the filled gas bag was connected to the sampler inlet with Tenax-TA tube attached to the outlet; residual gas inside the bag was fully purged through the adsorption tube until automatic shutdown of sampling equipment to complete adsorptive enrichment.

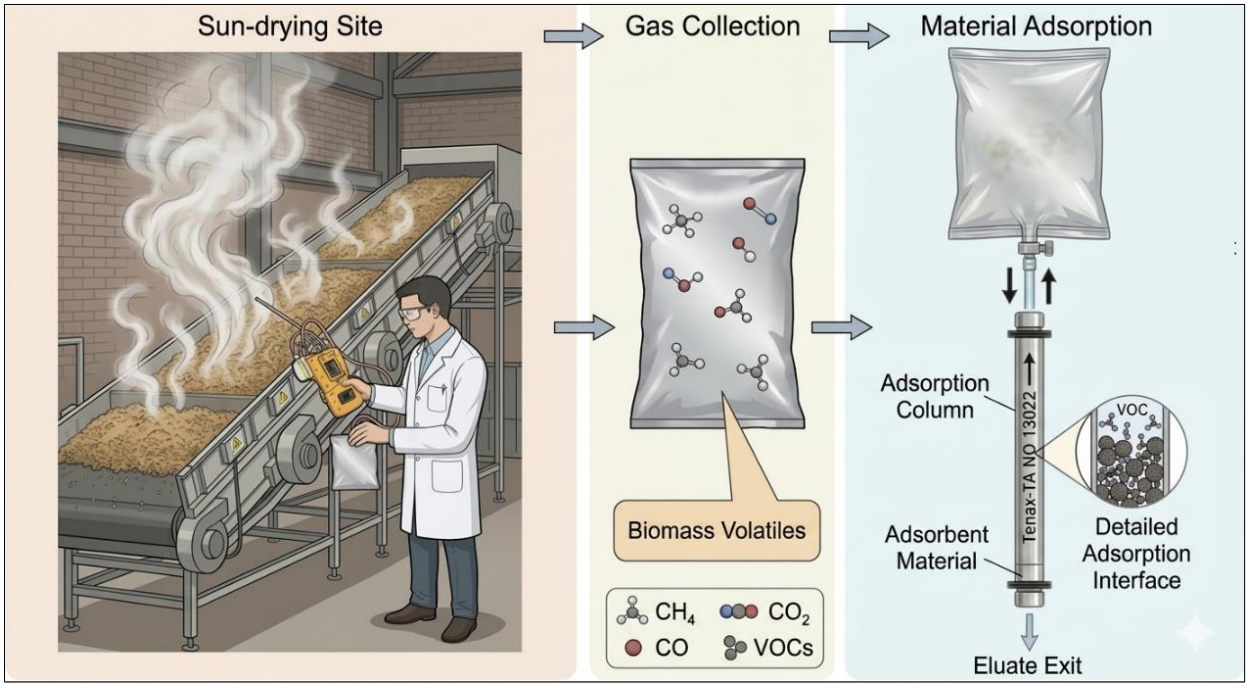

Figure 1: Schematic Diagram of Gas Collection During Spreading and Cooling Process

Schemes b/c/d: Variable-Power Fan Sampling: Fan inlet and outlet were sequentially connected to rubber hoses via PVC adapters, with a 500 mL plastic funnel fixed at the gas intake end and a 1 L aluminum foil bag assembled on the exhaust side. Fans were switched on synchronously at the initiation of spreading operation, with formal sampling started after 30 s pre-aeration until complete filling of sampling bags.

Afterwards, one end of filled gas bags was coupled with Tenax-TA tubes, and high-purity nitrogen was continuously purged through the system for 5 min at constant velocity to achieve full adsorption of contained volatile analytes onto solid sorbent.

2.3 Gas Sampling Protocols for Spreading and Cooling Exhaust

Volatile compositions trapped on Tenax-TA tubes were characterized by Agilent 7890B-5977B GC-MS equipment with optimized chromatographic and mass spectrometric parameters as below:

GC Conditions:

Injector temperature: 250 ℃; carrier gas: high-purity helium (≥99.9%); constant flow rate: 1.2 mL/min; splitless injection mode. Oven temperature program: initial hold at 40 ℃ for 2 min, ramp up to 250 ℃ at 4 ℃/min and maintain final temperature for 5 min.

MS Conditions:

Electron ionization (EI) ion source, electron impact energy 70 eV; ion source temperature 230 ℃, quadrupole temperature 150 ℃; solvent delay: 1 min to eliminate solvent interference; full-scan mass range: m/z 35–350 covering molecular weights of predominant spreading-cooling volatile organics.

3. RESULTS AND DISCUSSION

3.1 Chromatographic Peak Resolution of Samples from Different Sampling Approaches

Thermal processing of brewing grains generates abundant flavor substances closely associated with final Baijiu sensory quality, which evaporate along with water vapor during subsequent spreading and cooling procedures [6]. Differences in pumping power and air intake caliber among sampling devices alter actual gas collection efficiency and introduce exogenous impurities, deteriorating chromatographic peak separation accuracy for qualitative and quantitative analysis.

Three pairs of characteristic compounds distributed across early, middle and late chromatographic elution intervals (ethyl acetate-acetone, ethyl acetate-amyl alcohol, ethyl caproate-2-ethyl-1-hexanol) with divergent polarity and separation difficulty were selected to calculate chromatographic resolution (R_s) for systematic evaluation of sampling performance.

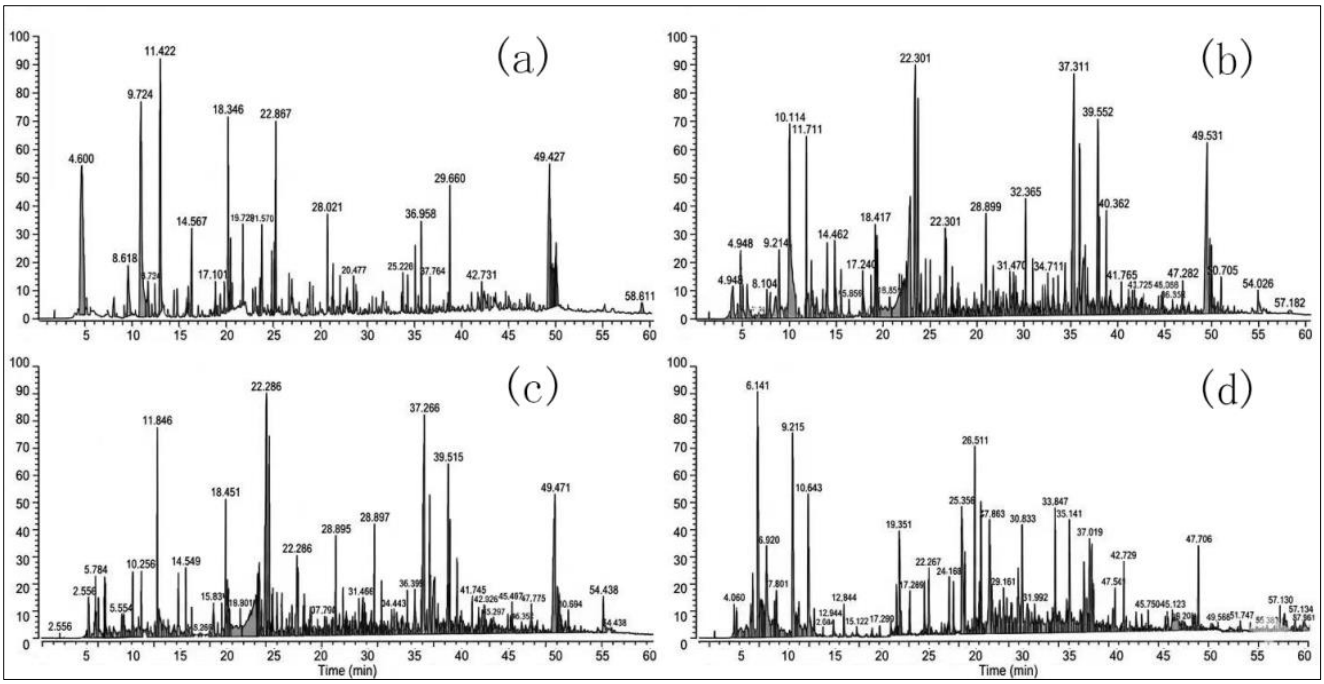

Figure 2: Comparison of Different Acquisition Equipment

Note: (a) Portable constant-flow sampler; (b) 300 W fan; (c) 260 W fan; (d) 220 W fan sampling TIC.

Resolution calculation formula:

$$R_s=\frac{2(t_{R2}-t_{R1})}{W_1+W_2}$$

t_{R1}、t_{R2}: retention time of adjacent chromatographic peaks; W1、W2: baseline peak width of corresponding peaks. Baseline complete separation is defined as R_s≥1.5, while partial peak overlap occurs when R_s<1.

Table 3: Resolution Values of Three Characteristic Peak Pairs from Four Sampling Groups

Sampling Code	R1(ethyl acetate-acetone)	R2(ethyl acetate-amyl alcohol)	R3(ethyl caproate-2-ethyl-1-hexanol)
a	1.37	1.25	0.84
b	0.92	0.82	0.73
c	0.96	0.71	0.76
d	0.81	0.73	0.61

As summarized in Table 3, group a (constant-flow sampler) obtained the highest resolution for all three compound pairs despite no full baseline separation (Rs<1.5), basically satisfying laboratory qualitative analysis requirements; resolution values decreased sequentially from group b, c to d, with group d showing severe peak overlapping and the poorest separation efficiency (R1=0.81,R2=0.73,R3=0.61).

High moisture content introduced by high-flow fan sampling is the primary contributor to peak broadening and co-elution phenomena, lowering chromatographic separation compared with low-flow constant-flow sampling. Total ion current chromatograms visually confirmed superior peak shape and separation of group a samples, though dense minor peaks appeared within retention time ranges of 5–10 min and 25–40 min limiting trace-component discrimination; chromatograms from fan-based sampling (b, c, d) exhibited drastically increased dense miscellaneous peaks, hindering identification of low-abundance volatiles, consistent with quantitative resolution statistics.

3.2 Total Chromatographic Peak Area for Overall Volatile Capture Capacity

Variations in sampling flow velocity and entrained water content among different collection modes cause discrepancy in adsorption efficiency and total captured volatile quantity reflected by overall chromatographic peak area, which was adopted to quantify total trapping performance of each sampling scheme.

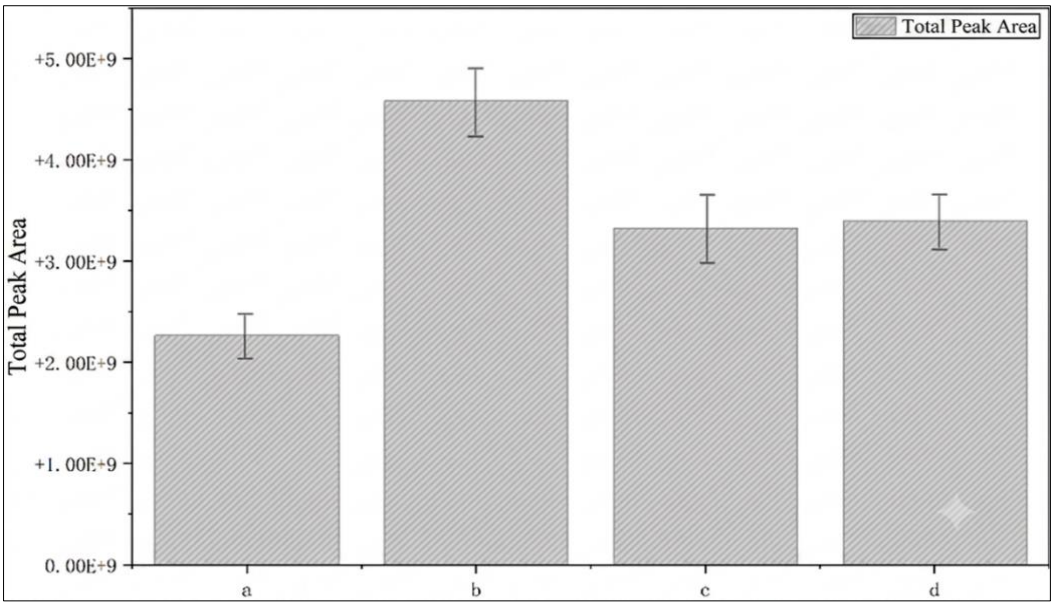

Figure 3: Comparison of Peak Areas of Different Acquisition Method

Statistically significant differences existed in cumulative peak area across four experimental groups: group b (300 W fan) achieved the maximum total peak area of 4.6×109 ($P<0.05$ vs other groups), indicating the strongest comprehensive trapping capacity for spreading-cooling volatile organics and complete retention of full flavor spectrum; group c and d showed comparable intermediate total peak area (~3.4×109, $P>0.05$ between c and d), competent for conventional routine analysis yet unsuitable for full-spectrum flavor recovery research; group a yielded the minimum total peak area (2.3×109) with extremely significant difference against group b ($P<0.01$), prone to omission of trace-level flavor components and incomplete characterization of actual gaseous flavor composition.

The outstanding total capture capability of 300 W fan sampling provides fundamental data support for industrial-scale waste gas recovery equipment development, whereas low cumulative peak area of constant-flow sampling restricts its application in quantitative total volatile recovery testing.

3.3 Detected Compound Categories Collected by Distinct Sampling Devices

Detected peak numbers of six typical flavor categories (aromatics, organic acids, esters, alcohols, ketones, aldehydes) were counted to assess selective trapping performance toward different chemical families of aroma substances.

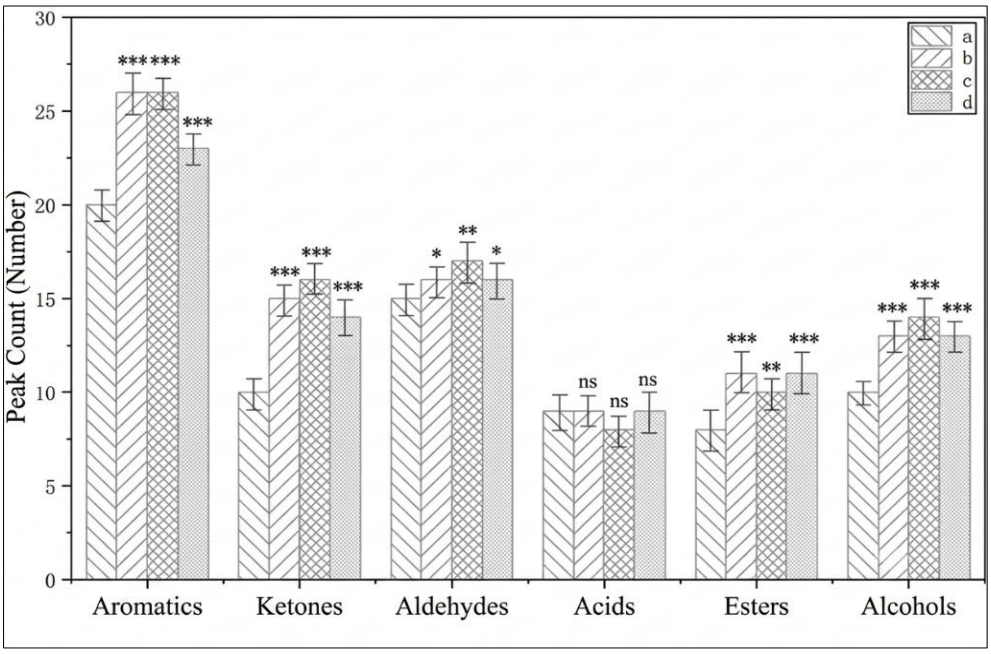

Figure 4: Comparison of Peak Numbers of Different Acquisition Method

Group b presented remarkably higher detectable peak counts for core Nongxiang characteristic constituents including aromatics, aldehydes, esters and alcohols, enabling optimal reconstruction of real spreading-cooling volatile profile. Although group a exhibited numerically fewer detected peaks for all compound classes, one-way ANOVA and LSD post-hoc test revealed no statistically significant difference versus group b and d (all P>0.05, P range: 0.068~0.093 for paired comparison between a and b, 0.075~0.090 between a and d).

Hence, portable constant-flow sampling can effectively capture core characteristic flavor components and reflect primary gaseous flavor composition despite slightly inferior total compound recovery. Benefiting from convenient operation and low energy consumption, the constant-flow sampler is applicable for rapid preliminary screening and rough qualitative characterization in low-precision detection scenarios.

3.4 Variation of Collection Efficiency Along Different Sampling Positions on Spreading Conveyor

Uneven spatial distribution of temperature and ambient airflow along automatic spreading-cooling equipment leads to heterogeneous volatile release from fermented grains, resulting in variable sampling outcomes at different conveyor locations.

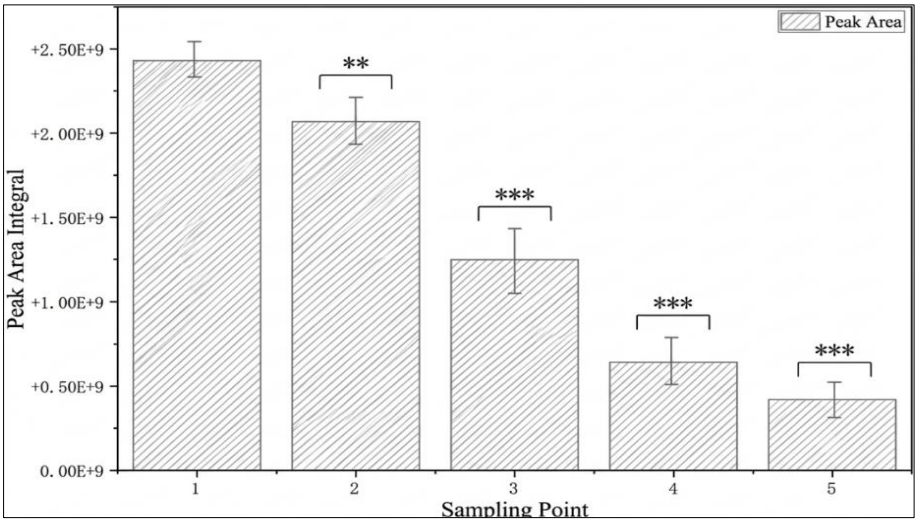

Figure 5: Comparison of Peak Areas at Different Sampling Points

Five equidistant sampling points were set sequentially from fermented grain feed inlet to material outlet to investigate positional effects via total peak area and detectable compound variety. Total volatile abundance gradually declined from feed inlet toward discharge outlet: sampling point 1 (feeding inlet) owned the highest total peak area (2.4×10^9, P<0.05 vs downstream sites), followed by point 2 (2.1×10^9); total peak values of point 3, 4 and 5 decreased progressively to 1.3×10^9, 0.6×10^9 and 0.4×10^9, respectively.

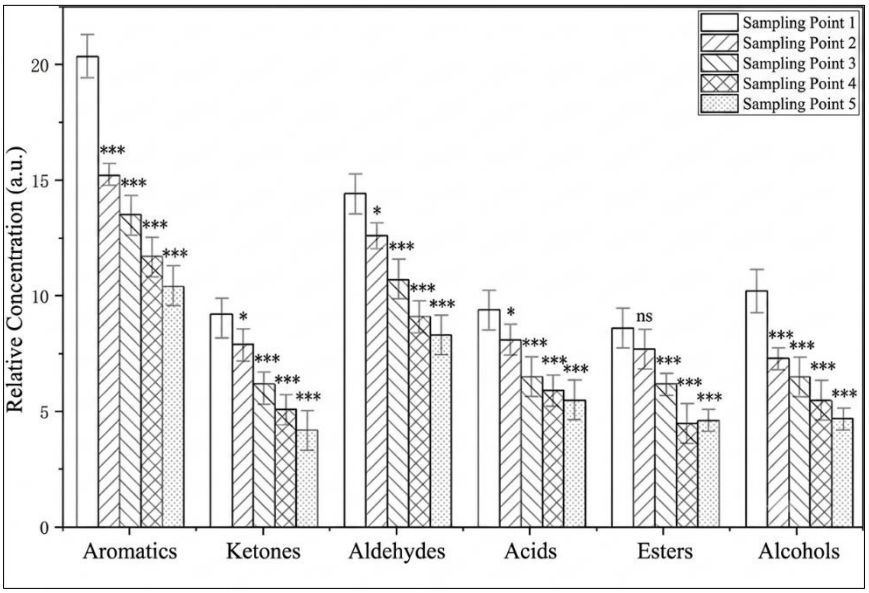

Figure 6: Comparison of Compound Types at Different Sampling Points

For individual chemical classes, sampling point 1 produced maximum detectable peaks for aromatics (~20), ketones (~9), aldehydes (~14), organic acids (~9), esters and alcohols; peak counts continuously dropped at subsequent downstream positions with the minimum value observed at point 5. Gradual volatilization loss and airflow-driven diffusion of flavor substances during grain transportation along spreading equipment account for continuous reduction of volatile concentration from inlet to outlet. Consequently, sampling at fermented grain feed inlet (point 1) is recommended as the preferred sampling location for comprehensive full-component analysis of spreading-cooling exhaust gas in follow-up related experiments.

4. CONCLUSION

In this study, four gas sampling strategies combining portable constant-flow sampler and three-power ventilation fans were compared for collecting volatile waste gas from Nongxiang Baijiu spreading and cooling procedure, and sampling position effects on recovery efficiency were characterized systematically:

1) Marked discrepancies existed in sampling performance between different collection modes. The portable constant-flow sampler obtained superior chromatographic separation with resolution of 1.37, 1.25 and 0.84 for three paired marker compounds but limited total volatile capture (2.3×10^9 total peak area); the 300 W fan group realized optimal overall flavor trapping capacity with total peak area of 4.6×10^9 yet poor chromatographic separation (Rs: 0.92, 0.82, 0.73).
2) Portable constant-flow sampling is qualified for core flavor identification and rapid preliminary screening with easy operation and low power consumption, serving as a standard qualitative benchmark for laboratory research; sampling parameters of 300 W fan provide critical reference for structural design of future industrialized spreading exhaust recycling installations.
3) Sampling location significantly influences gaseous collection efficiency; fermented grain feed inlet achieves optimal recovery of both total volatile quantity and diversified flavor compounds, which is prioritized as standard sampling spot for subsequent experimental sample collection.
4) Total abundance of spreading-cooling volatiles decreases progressively from grain inlet to outlet, corresponding to continuous volatile dissipation and airflow migration throughout mechanical spreading processing.

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