

Identification and validation of key organic acids that modulate the temporal dynamics of oral irritation in baijiu

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ABSTRACT

Oral stimulation is an important component of the sensory profile of Baijiu, yet how specific organic acids modulate the temporal progression of ethanol-induced irritation remains unclear. In this study, the roles of organic acids were investigated by integrating time-intensity sensory analysis with quantitative profiling of 23 organic acids across 11 representative Baijiu samples. Multivariate analyses highlighted lactic, acetic, hexanoic, and octanoic acids as major contributors to differences in dynamic perceptual trajectories. Validation experiments in both model solutions and authentic Baijiu matrices showed that lactic and acetic acids significantly delayed the onset and buildup of oral irritation. Hexanoic acid was identified as a potential contributor to stronger sensory responses, although this effect was not statistically confirmed in the validation test. These findings show that specific organic acids can selectively modulate the temporal features of ethanol-induced irritation in Baijiu and provide a framework for linking chemical composition with dynamic sensory perception.

1. Introduction

Baijiu is a traditional Chinese distilled spirit renowned for its intricate flavor complexity and distinctive regional styles. While the aroma chemistry of Baijiu has been extensively studied, the chemical basis underlying its oral stimulation, which manifests as a combination of sensations including tingling, warmth, burning, and pungency experienced during consumption, remains comparatively underexplored (Y. He et al., 2021, 2022). In Baijiu, taste perception results from the combined effects of ethanol, which is the predominant component following water, and a wide range of dissolved flavor-active compounds, including organic acids, esters, and polyols, among others (Wang, Wang, Cheng, et al., 2025; Wang, Wang, Zhang, et al., 2025). Ethanol itself is strongly associated with the characteristic burning and warming sensations of Baijiu (Ickes & Cadwallader, 2017). Meanwhile, other taste-active compounds impart intrinsic flavors and can modulate the intensity or smoothness of ethanol's oral impact via complex interactions (Ickes & Cadwallader, 2017; Y. Luo et al., 2020). For instance, the presence of certain acids at low concentrations has been shown to

suppress ethanol's irritative pungency (Y. He et al., 2022; Yan et al., 2024). This interactive synergy among ethanol and minor constituents ultimately shapes Baijiu's distinctive mouthfeel.

Previous studies have attempted to link Baijiu's chemical composition with its oral irritation or pungency. However, most adopted a broad analytical scope that surveyed dozens of volatiles simultaneously, which can obscure the role of specific compound classes. For example, one recent study quantified 57 different volatile components in Baijiu and identified several esters, aldehydes, and organic acids as key contributors to its trigeminal "burn" sensation (Y. He et al., 2022). Similarly, comprehensive flavor analyses across various spirits have highlighted that organic acids, alcohols, and other components jointly influence the perception of mouthfeel (Dong et al., 2024; Ickes & Cadwallader, 2017; Yan et al., 2024). While these studies provided valuable insights into Baijiu's complexity, their all-encompassing approach makes it difficult to pinpoint clear mechanistic links between any given class of compounds and specific sensory effects. This limitation suggests the need for a more targeted inquiry focusing on a single functional group of flavor compounds. In other words, to fully elucidate the dynamics of Baijiu's

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taste, one should isolate and examine the contributions of a coherent class of taste-active substances rather than treating the flavor matrix as a black box. Such a focused approach would help to uncover definitive relationships between particular compounds and the sensations they evoke (de-la-Fuente-Blanco et al., 2017; He et al., 2025).

Organic acids stand out as strong candidates for targeted investigation because they are among the most important taste-active fermentation by-products in Baijiu and contribute substantially to sourness, astringency, and lingering mouthfeel (Junge et al., 2020; X. Luo et al., 2025; Yan et al., 2024). Their sensory activity arises from dissociable carboxyl groups, which release protons and also participate in intermolecular interactions within the complex Baijiu matrix. Depending on their structure and physicochemical properties, different organic acids may contribute differently to oral perception: short-chain acids are often associated with sharper sourness and stimulation, whereas medium- and long-chain acids may exert weaker direct taste effects but still modulate mouthfeel through interactions with other flavor compounds (Bouchez & De Vuyst, 2022; Dong et al., 2024; Ferrero-del-Teso et al., 2024; Green et al., 2010). Previous studies have also shown that certain acids can influence sweetness, sourness, or burning-related sensations in mixed systems, suggesting that their role extends beyond isolated taste attributes (Junge et al., 2020). In Baijiu, such interaction effects are particularly relevant because oral stimulation is not determined by ethanol alone, but may also be shaped by coexisting non-volatile compounds, including organic acids. However, despite their sensory importance, organic acids in Baijiu have not been comprehensively studied, partly because many are non-volatile, present at low levels, and analytically challenging to characterize. As a result, previous studies have focused more heavily on volatile aroma compounds while giving less attention to organic acids (Wang, Jing, Wang, et al., 2022; Wang, Song, et al., 2022). This gap has limited a more complete understanding of Baijiu taste, particularly regarding how ethanol-induced oral stimulation may be modulated by the presence and interaction of organic acids.

Sensory analysis methods are crucial for linking chemical composition to flavor perception (Valentin et al., 2012). These methods can be divided into static approaches and dynamic approaches. Static sensory evaluation methods, including quantitative descriptive analysis, check-all-that-apply, and conventional descriptive panels, are typically designed to assess and rate the overall intensity of specific sensory attributes present in a given sample (Li et al., 2023; Malfeito-Ferreira, 2023; Nougarede et al., 2023). While straightforward and effective for general flavor profiling, static methods have notable limitations. They rely heavily on panelists' cumulative impressions, limiting reproducibility and failing to capture how sensory perception evolves during consumption. This limitation becomes especially pronounced when evaluating complex beverages such as Baijiu, where oral sensations such as burning, numbing, and tactile sharpness fluctuate rapidly after ingestion. Dynamic sensory techniques are designed to monitor such changes in real time, thus offering richer insights into temporal sensory phenomena. Approaches such as the temporal dominance of sensations (TDS) and time-intensity (TI) analysis enable researchers to monitor the progression of one or more sensory attributes continuously throughout the tasting experience (Chaya et al., 2019; Gotow et al., 2018; Sokolowsky & Fischer, 2012). Among the various dynamic sensory techniques, TI analysis is particularly well-suited for quantifying the temporal evolution of a single sensory attribute, such as the intensity of oral stimulation. In a typical TI assessment, trained panelists are instructed to continuously rate the perceived intensity of the target sensation from the moment of intake, generating a time-intensity curve that describes how the perception develops and dissipates over time. This curve provides several key quantitative parameters, including the maximum intensity reached, the time required to reach that maximum, and the duration of the sensation before it fully dissipates, collectively defining the temporal sensory profile of the stimulus (Chaya et al., 2019; Pineau et al., 2009; Yan et al., 2024). The method is especially advantageous for high-ethanol beverages like Baijiu, whose sensory attributes

are shaped by rapid and complex interactions between ethanol and minor compounds in the oral environment. TI analysis potentially enables the second-by-second mapping of such trigeminal responses, offering critical insight into how compounds like organic acids modulate the timing and persistence of oral irritation. When combined with chemical quantification, TI profiling thus serves as a powerful analytical framework for elucidating dynamic interactions between flavor-active constituents and perceived sensory responses in complex alcoholic matrices.

Therefore, the present study aimed to elucidate the role of organic acids in modulating the oral stimulation of Baijiu by integrating targeted chemical characterization with dynamic sensory evaluation. To achieve this goal, a four-pronged methodological framework was established (Fig. 1). First, organic acids in Baijiu were comprehensively profiled using derivatization-assisted gas chromatography–mass spectrometry, enabling the quantification of both volatile and non-volatile acids across a representative set of samples. Second, time-intensity sensory analysis was used to capture the temporal progression of ethanol-induced oral stimulation. Third, chemometric techniques were applied to link chemical composition with dynamic sensory parameters and to identify specific acids associated with variations in the stimulation profile. Finally, validation experiments were conducted in both model solutions and authentic Baijiu matrices to verify the sensory relevance of selected compounds. This integrated approach establishes a scalable framework for pinpointing molecular drivers of temporal sensory modulation in complex beverages and offers actionable insights for optimizing flavor dynamics in high-ethanol systems such as Baijiu.

2. Materials and methods

2.1. Reagents and standards

A total of 23 organic acid standards used for compound identification and quantification were obtained from Merck (Shanghai, China), and all authentic standards were of $\geq 95\%$ purity. A homologous series of C₅–C₃₀ *n*-alkanes and *N,O*-bis(trimethylsilyl)trifluoroacetamide containing 1% trimethylchlorosilane were obtained from Merck (Shanghai, China). 1,2-Dichlorobenzene (internal standard 1), salicin (internal standard 2), and pyridine were supplied by J&K Chemical Corp. (Shanghai, China). For the validation experiments, food-grade ethanol (95%, HuaLin, Yunnan, China) was used as the base alcohol. To minimize sensory interference, pure ethanol without added volatile compounds or flavoring agents was diluted to the target concentrations with ultrapure water produced by an NW10VF purification system (Heal Force Development Ltd., Hong Kong, China). Food-grade lactic acid, acetic acid, and hexanoic acid were purchased from Sigma-Aldrich Co. Ltd. (Shanghai, China) and stored at $-18\text{ }^{\circ}\text{C}$ before use to maintain their stability.

2.2. Baijiu samples

A total of 11 commercial Baijiu samples were selected for this study. To ensure consistency in flavor style and ethanol content, all samples belonged to the *Strong* flavor Baijiu and had an alcohol content of 52% vol. These products were produced using comparable traditional solid-state fermentation processes, with sorghum as the primary raw material, medium-temperature *Daqu* as the fermentation starter, and mud pits as the fermentation vessels. All samples were commercial products sourced from major Chinese distilleries in 2023 and were coded S1 through S11 for subsequent analyses. This selection strategy was intended to minimize variation arising from flavor type and alcohol strength while preserving natural compositional differences among commercial *Strong* flavor Baijiu samples, thereby allowing sensory differences to be examined in relation to other coexisting compounds, including organic acids. Samples were stored at $4\text{ }^{\circ}\text{C}$ prior to chemical and sensory evaluation.

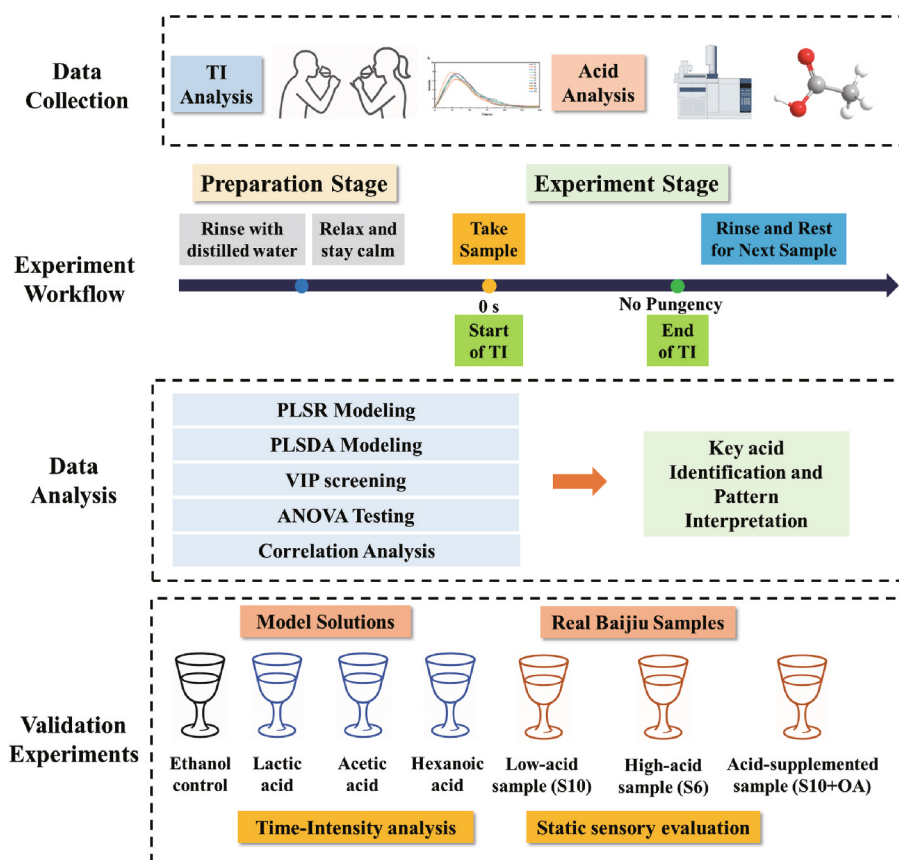


Fig. 1. Schematic of the experimental workflow.

2.3. Analysis of volatile organic acids

Volatile organic acids were extracted from Baijiu samples by liquid-liquid extraction (LLE) following a previously reported protocol for Baijiu analysis (Wang, Jing, Wang, et al., 2022). Each 25 mL sample was diluted to 15% (v/v) ethanol with Milli-Q water and spiked with 1,2-dichlorobenzene (internal standard 1, 200 mg/L). After saturation with 50 g of sodium chloride, the sample was extracted three times with 50 mL of dichloromethane. The combined organic phases were dried over anhydrous sodium sulfate and concentrated to 500 μ L using a Vigreux column under gentle distillation. GC-MS analysis was performed using a PerkinElmer Clarus 680 gas chromatograph coupled to an SQ8C mass spectrometer. A DB-Wax capillary column (60 m \times 0.25 mm i.d., 0.25 μ m film thickness) was used for compound separation. The injection port was maintained at 250 $^{\circ}$ C in splitless mode. Helium was used as the carrier gas at a constant flow rate of 1.5 mL/min. The oven temperature program was as follows: held at 40 $^{\circ}$ C for 2.5 min, ramped to 50 $^{\circ}$ C at 10 $^{\circ}$ C/min (held for 5 min), then to 80 $^{\circ}$ C at 4 $^{\circ}$ C/min (held for 5 min), followed by 200 $^{\circ}$ C at 8 $^{\circ}$ C/min, and finally to 240 $^{\circ}$ C at 10 $^{\circ}$ C/min (held for 5 min). The ion source and transfer line were both maintained at 250 $^{\circ}$ C. *Electron ionization* (EI) was performed at 70 eV, scanning over an m/z range of 30–550. Compound identification was based on mass spectral data, retention indices (RIs), and comparison with authentic standards. RIs were calculated using a homologous series of n -alkanes (C₅–C₃₀) analyzed under identical GC conditions. Quantification was performed using external standard calibration. Standard solutions at varying concentrations were prepared in dichloromethane, and calibration curves were constructed by plotting the analyte-to-internal standard peak area ratios against known concentrations. The same extraction and GC-MS conditions were applied to all standards and Baijiu samples. Calibration linearity was assessed based on the coefficient of determination (R^2).

2.4. Analysis of non-volatile organic acids

Non-volatile organic acids were analyzed using a BSTFA derivatization protocol based on a previously reported method for Baijiu analysis (Wang, Li, et al., 2022; Wang, Liu, et al., 2024). Each 10 mL Baijiu sample, containing salicin (internal standard 2, 1 mg/L) was initially concentrated to approximately 1 mL using a rotary evaporator. The concentrate was then transferred to a 2 mL GC vial and dried completely under a gentle nitrogen stream. Due to its high abundance in Baijiu, lactic acid was quantified separately by evaporating a 100 μ L aliquot of Baijiu in a 2 mL vial without pre-concentration. For derivatization, 100 μ L of BSTFA (containing 1% TMCS) and 50 μ L of pyridine were added to the dried residue. The vial was sealed and heated at 80 $^{\circ}$ C for 3 h. After cooling, 1 μ L of the derivatized solution was injected into the GC-MS system. GC-MS analysis was conducted using a PerkinElmer Clarus 680 gas chromatograph coupled to an SQ8C mass spectrometer. Separation was achieved on a DB-5 capillary column (30 m \times 0.25 mm i.d., 0.25 μ m film thickness, Agilent Technologies, CA, USA). The injector was set at 250 $^{\circ}$ C in splitless mode. Helium served as the carrier gas at a constant flow rate of 1.0 mL/min. The oven temperature program started at 65 $^{\circ}$ C (held for 2 min), increased to 280 $^{\circ}$ C at 6 $^{\circ}$ C/min, and held for 5 min. The ion source and transfer line temperatures were set at 300 $^{\circ}$ C and 280 $^{\circ}$ C, respectively. EI was performed at 70 eV, with a scan range of m/z 45–300. Tentative compound identification was carried out using the NIST 2014 mass spectral library, and further confirmed by comparing both the RIs and mass spectra with those of authentic standards under identical conditions. Quantification was based on external calibration. Mixed standard solutions were prepared in a 53% ethanol model matrix and serially diluted to obtain a range of working concentrations. Calibration curves were constructed by plotting the peak area ratios (analyte/internal standard) against the analyte concentrations. Linearity was evaluated based on the R^2 .

2.5. Analytical method validation

The limit of detection (LOD) and limit of quantitation (LOQ) were defined as the lowest concentration points on the calibration curves corresponding to signal-to-noise ratios of 3 and 10, respectively. All measurements were conducted in triplicate. Intra-day precision was evaluated by analyzing standard solutions (1 mg/L) three times within a single day, while inter-day precision was assessed by repeating the procedure on three non-consecutive days. Precision was expressed as relative standard deviation (RSD, %). Method accuracy was assessed through spike recovery tests. Target analytes were added to Baijiu samples at concentrations close to their native levels. Recoveries were calculated by comparing the difference in concentrations between spiked and unspiked samples relative to the amount added. All samples were treated under identical conditions, and the results were expressed as percentages.

2.6. TI sensory evaluation

The study received ethical approval from the Ethics Review Committee of Zhejiang University (Approval No. 2024–2) and was conducted in accordance with the ethical principles outlined in the Declaration of Helsinki. Sixteen right-handed, healthy Chinese participants (8 females and 8 males, all aged 23–27 years) were recruited. All participants were screened to ensure they had no history of alcohol sensitivity, dependency, or allergic reactions. Written informed consent was obtained prior to participation, and panelists received monetary compensation for their involvement in the study. The panel underwent a structured three-month training program aimed at developing their ability to recognize and rate oral stimulation. The training included both theoretical instruction and practical exercises using TI software for real-time data acquisition. Simulated evaluations were performed using aqueous ethanol solutions of varying concentrations to familiarize panelists with the target sensation. Panel performance was assessed based on the consistency of replicated TI curves for the same panelist using the SensoMaker interface. Training was considered sufficient when the replicate curves were aligned at least 40% of the time, following a previously reported time-intensity training protocol (Chaya et al., 2019; Y. He et al., 2022). All 16 participants completed the final sensory evaluation, and all were included in the subsequent data analysis. All training and formal evaluation sessions were conducted in a dedicated sensory evaluation room maintained at a controlled temperature of 21 ± 1 °C.

During the evaluation, Baijiu samples (5 mL) were served in coded disposable cups and presented in randomized order to minimize potential bias. To reduce sensory fatigue and desensitization, each panelist participated in only one session per day, with a maximum of three samples evaluated per session. A minimum interval of 10 min was maintained between samples, during which skim milk was provided as a palate cleanser. Before each evaluation, panelists rinsed their mouths with water. Each test was conducted using the SensoMaker interface. Panelists were instructed to pour the entire sample into their mouth, distribute it evenly across the tongue, and initiate the evaluation by pressing the “Start” button. The perceived intensity of oral stimulation was continuously rated using keyboard inputs on a 10-point scale, ranging from 0 (not perceptible) to 10 (extremely intense). Intensity data were recorded at one-second intervals throughout the evaluation period.

2.7. Statistical analysis

Data for organic acids were acquired and processed using TurboMass 6.0 software. TI data were processed using SensoMaker to extract seven temporal sensory parameters describing the dynamic trajectory of oral stimulation. These sensory descriptors were standardized before hierarchical clustering to evaluate sensory similarities among Baijiu samples. To explore the relationship between organic acid concentrations

and sensory dynamics, partial least squares regression (PLSR) was performed using Microsoft Excel, and partial least squares discriminant analysis (PLS-DA) was conducted using SIMCA 14.1 (Umetrics, Sweden). Before PLSR and PLS-DA, the data were standardized to reduce the influence of differences in variable scale. For each PLS-DA model, including those based on composite scores and individual descriptors, variable importance in projection (VIP) scores were calculated, and compounds with $VIP > 1.0$ were considered key contributors to sensory variation. Model significance and robustness were assessed by cross-validation and permutation testing in SIMCA. Further validation was conducted using Pearson correlation analysis and one-way ANOVA to assess statistical significance across grouped samples (Fig. 1). All statistical analyses and data visualization were performed using Origin 2018 (OriginLab, USA) and Python 3.13.3.

3. Results and discussion

3.1. Temporal dynamics of stimulation perception in baijiu samples based on TI analysis

The temporal evolution of oral stimulation induced by Baijiu consumption was characterized using TI analysis across eleven representative samples. As illustrated in Fig. 2A, all samples exhibited a typical sensory trajectory consisting of three sequential phases: an initial ascending slope, a brief plateau phase, and a gradual decline. Peak stimulation was generally observed between 20 and 30 s after ingestion. Although the overall shape of the sensory curve appeared consistent, likely due to the similar ethanol content and flavor characteristic of the Baijiu samples, considerable differences were observed among the samples in terms of onset speed, maximum intensity, and decay rate. To quantify these temporal variations, seven parameters were extracted from the TI curves, which provided a detailed characterization of the dynamic trajectory of oral stimulation for each sample (see Table S1). Specifically, TI_5% and TI_90% represent the times required to reach 5% and 90% of the maximum intensity during the rising phase, reflecting the onset and buildup speed of stimulation. TD_90% and TD_5% represent the times required for the perceived intensity to decline from the maximum to 90% and 5% during the descending phase, describing the decay characteristics of the sensation. I_max represents the peak perceived intensity, Plateau_90% indicates the duration for which the sensation remains above 90% of the maximum intensity, and Area represents the area under the TI curve, reflecting the overall cumulative oral stimulation over time.

I_max values ranged from 6.28 to 7.85, reflecting inter-sample variability in peak stimulation. Area values spanned from 332.02 to 400.96, indicating differences in cumulative mouthfeel impact. The early onset of irritation was supported by TI_5% values between 0.9 and 1.7 s, while TI_90% ranged from 18.0 to 24.9 s, showing that most samples reached near-maximum intensity within 25 s. In contrast, TD_90% exhibited a broader distribution (31.9–43.6 s), suggesting sample-specific differences in stimulation persistence. Plateau_90% varied substantially, with S3 exhibiting a prolonged sensation (20.2 s) and S5 showing a more transient profile (13.9 s). Overall, these results indicated that samples such as S4 and S8 produced milder and shorter-lived stimulation, whereas samples such as S2, S5, and S6 elicited stronger and more persistent sensory effects. Given that all samples had the same concentration of ethanol, these differences are likely attributed to variations in other taste-active compounds. Previous research has demonstrated that non-ethanol constituents, particularly organic acids and esters, can significantly modulate the perception of oral stimulation in alcoholic beverages, further supporting this interpretation (Y. He et al., 2022).

To explore sensory variability among the samples, HCA was performed using the seven TI parameters. This analysis resulted in three distinct clusters, as shown in Fig. 2B. Group T1 was characterized by delayed onset, relatively high peak intensity, and extended decline

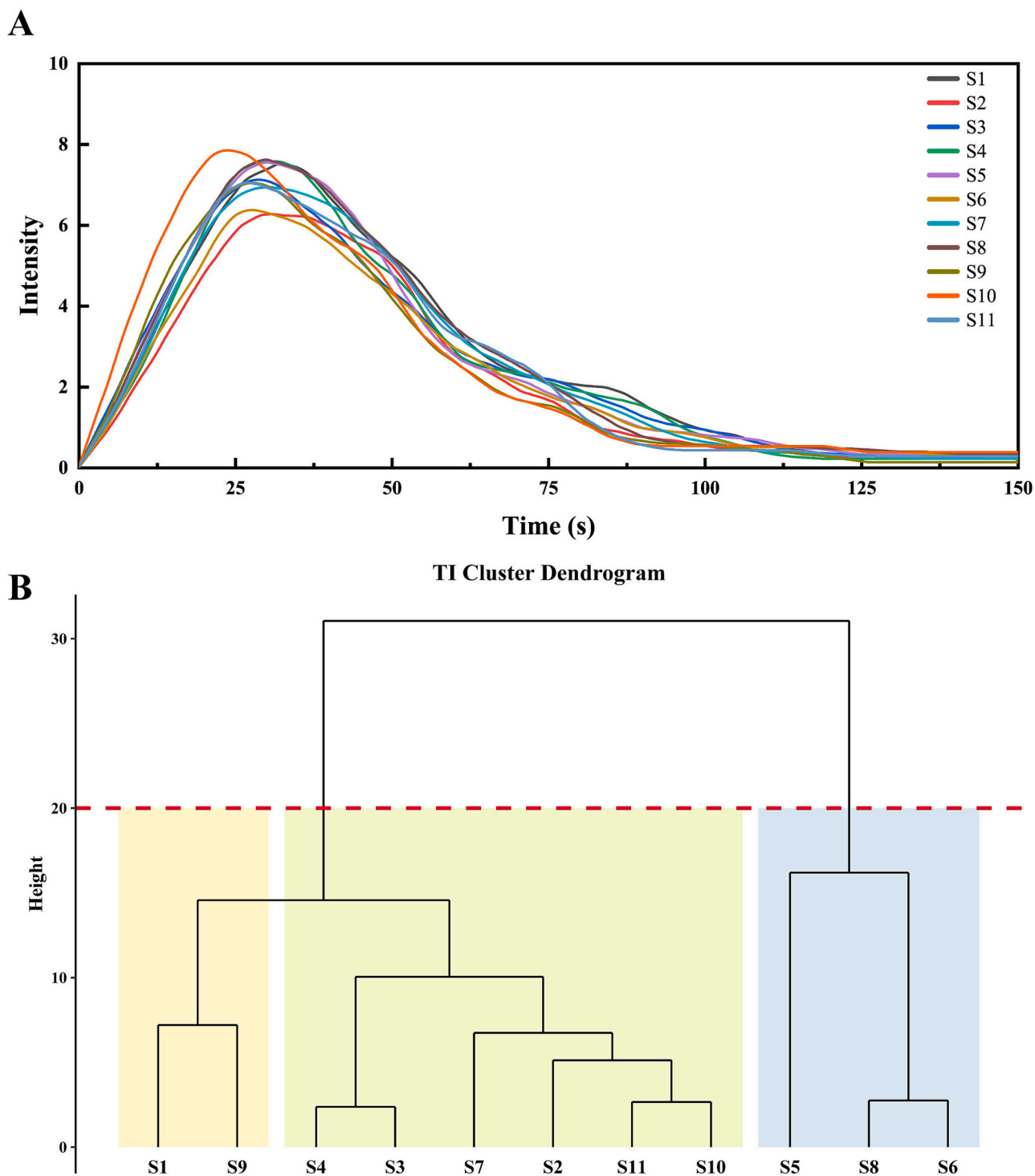


Fig. 2. (A) Time-intensity curves of oral stimulation for eleven strong-flavor Baijiu samples. (B) Hierarchical clustering of samples based on time-intensity parameters.

durations, suggesting persistent stimulation. Group T2 exhibited the shortest onset times, highest I_{max} , and largest cumulative intensity, reflecting rapid and intense stimulation profiles. Group T3 showed intermediate or variable values across most TI parameters, with I_{max} ranging from 6.28 to 7.56 and Area values from 332.02 to 400.96. The Plateau_{90%} durations were relatively shorter in some samples, indicating more transient stimulation. Despite intergroup variability, a rapid-onset pattern was consistently observed across most samples, indicating a common temporal trajectory among Baijiu products. These

clustering outcomes provide a structured foundation for subsequent chemical-sensory correlation modeling.

3.2. Quantification of volatile organic acids

Volatile organic acids in Baijiu samples were identified and quantified using GC-MS. Identification was based on the combination of retention index matching, mass spectral library comparisons, and confirmation with authentic standards. A total of twelve volatile acids

was successfully detected across all samples. Method validation demonstrated excellent analytical performance, with R^2 exceeding 0.99 for all compounds across their respective calibration ranges. The LOD and LOQ ranged from 0.79 to 3.75 $\mu\text{g/L}$ and from 2.63 to 12.50 $\mu\text{g/L}$, respectively, confirming the high sensitivity of the method. Precision was verified by calculating intra-day and inter-day, which ranged from 0.58% to 9.10% and from 1.00% to 7.48%, respectively. Recovery rates ranged from 93.57% to 102.31%, further validating the accuracy and reproducibility of the analytical procedure (see Table 1). These validation results, together with the previously reported application of this procedure in Baijiu analysis, support the reliability and suitability of the method for profiling volatile organic acids in the present study.

Among the volatile acids detected, hexanoic acid was the most abundant, with concentrations ranging from 505 to 886 mg/L across the samples. Acetic acid was the second most abundant, with concentrations ranging from 178 to 533 mg/L (see Table 2). Both compounds are well-known fermentation by-products. Acetic acid is typically formed through microbial oxidation and fermentative metabolism, whereas hexanoic acid is generally associated with microbial fatty acid metabolism during fermentation. As major metabolites generated during Baijiu fermentation, both acids can accumulate at relatively high concentrations and are therefore likely to contribute substantially to Baijiu acidity and oral sensory perception. Previous studies have also linked these acids to sensory attributes such as sourness, irritation, and oral tactile intensity in alcoholic systems and Baijiu. Their high concentrations in the present study further suggest that they may play important roles in shaping the acidity of Baijiu and the resulting oral stimulation

(Dong et al., 2024; Yan et al., 2024). In addition to these dominant acids, a second group of mid-level acids, including propanoic, butanoic, valeric, heptanoic, and octanoic acids, was detected at concentrations typical of strong-flavor Baijiu. These compounds contribute diverse flavor characteristics and, due to their moderate volatility and amphiphilic properties, may interact with ethanol and other volatile components to influence mouthfeel attributes such as the smoothness of entry or the perceived delay in the onset of oral stimulation. By contrast, only trace amounts of higher fatty acids, such as nonanoic acid, decanoic acid, and 2-methylhexanoic acid, were sporadically detected. These trace-level compounds are unlikely to exert significant direct sensory effects. However, potential matrix interactions, such as the modulation of the release of other flavor-active compounds, cannot be entirely ruled out even at these low concentrations.

3.3. Quantification of non-volatile organic acids

Compared to volatile organic acids, NVOAs exhibit lower volatility and higher polarity, characteristics that complicate their analytical detection but also suggest a potentially significant role in contributing to oral stimulation and mouthfeel persistence. In the present study, NVOAs in Baijiu samples were identified and quantified using GC-MS following derivatization with BSTFA. This derivatization procedure improves the volatility and thermal stability of polar organic acids, thereby enabling their reliable quantification in complex, ethanol-rich matrices (Wang, Cheng, et al., 2024). A total of eleven NVOAs were successfully quantified across all samples. Method validation confirmed excellent

Table 1

Linear Range, Coefficients of Determination, LOD, LOQ, Precision, and Recovery Rate for the Analytical Identification of Organic Acids in Baijiu by GC-MS.

Compound	CAS	Identification	Linear Range	Linearity		R^2	LOD ($\mu\text{g/L}$)	LOQ ($\mu\text{g/L}$)	Intraday Precision (%)	Interday Precision (%)	Recovery Rate (%)
				slope	intercept						
3-Methylvaleric acid	105-43-1	RI, MS, S	0.1-50	0.4549	-0.6602	0.9918	1.46	4.87	3.39	2.58	97.82
Butanoic acid	107-92-6	RI, MS, S	1-500	0.0752	-1.5168	0.9969	0.79	2.63	2.56	1.22	99.02
Valeric acid	109-52-4	RI, MS, S	1-500	0.0882	-0.7142	0.9919	3.75	12.50	4.25	1.86	100.37
Succinic acid	110-15-6	RI, MS, S	0.1-50	0.9605	-0.2928	0.9911	1.77	5.90	3.24	5.86	100.21
Maleic acid	110-16-7	RI, MS, S	0.1-50	0.0191	0.0192	0.9986	0.95	3.17	5.80	1.84	96.82
Heptanoic acid	111-14-8	RI, MS, S	1-500	0.1464	-1.7971	0.9909	3.41	11.37	3.34	2.04	100.38
Nonanoic acid	112-05-0	RI, MS, S	0.1-50	0.0433	-0.0108	0.9927	2.08	6.93	9.10	3.34	93.57
Octanoic acid	124-07-2	RI, MS, S	1-500	0.0687	-0.1873	0.9916	1.52	5.07	1.37	1.00	96.48
Hexanoic acid	142-62-1	RI, MS, S	10-5000	0.0291	-0.1508	0.9993	2.92	9.73	1.49	2.83	102.31
Decanoic acid	334-48-5	RI, MS, S	0.1-50	0.0509	0.0741	0.9944	2.31	7.70	7.48	7.48	98.74
2-Methylhexanoic acid	4536-23-6	RI, MS, S	0.1-50	0.2807	-0.4004	0.9925	3.22	10.73	4.76	4.26	96.73
2-Hydroxy-4-methylpentanoic acid	498-36-2	RI, MS, S	0.1-50	1.133	0.0182	0.9956	1.03	3.43	9.20	4.17	101.42
Lactic acid	50-21-5	RI, MS, S	10-5000	0.0361	0.6422	0.9947	15.21	50.70	3.26	4.76	99.72
3-Hydroxypropionic acid	503-66-2	RI, MS, S	0.1-50	0.0625	0.0082	0.9992	0.56	1.87	3.95	2.33	96.44
Palmitic acid	57-10-3	RI, MS, S	0.1-50	0.2788	0.0491	0.9933	3.22	10.73	3.37	0.94	95.28
Hydroxyisovaleric acid	625-08-1	RI, MS, S	0.1-50	0.4455	0.0432	0.9931	0.92	3.07	3.12	2.91	99.21
3-Hydroxybutyric acid	625-71-8	RI, MS, S	0.1-50	0.8781	0.1241	0.9914	0.84	2.80	6.36	8.05	95.66
Acetic acid	64-19-7	RI, MS, S	10-5000	0.0471	-3.5093	0.998	2.51	8.37	5.95	5.66	95.33
4-Methylvaleric acid	646-07-1	RI, MS, S	0.1-50	0.1216	-0.5302	0.9948	1.02	3.40	0.58	3.31	99.24
Benzoic acid	65-85-0	RI, MS, S	0.1-50	0.893	-0.0131	0.9972	0.76	2.53	7.44	1.01	95.73
Propanoic acid	79-09-4	RI, MS, S	1-500	0.0481	-0.6969	0.9995	0.92	3.07	1.14	2.37	96.54
Glycolic acid	79-14-1	RI, MS, S	1-500	0.9321	0.257	0.9955	0.41	1.37	7.06	4.71	90.71
2-Furoic acid	88-14-2	RI, MS, S	0.1-50	0.7581	-0.0917	0.9974	1.32	4.40	4.61	5.39	93.42

Table 2
Concentrations of Organic Acids in Eleven Strong-flavor Baijiu Samples Determined by GC-MS (mg/L).

Compound	S1	S2	S3	S4	S5	S6	S7	S8	S9	S10	S11
3-Methylvaleric acid	6.43 ± 0.17	6.72 ± 0.23	8.07 ± 0.10	13.42 ± 0.47	15.56 ± 0.27	9.15 ± 0.35	9.70 ± 0.41	5.94 ± 0.08	16.81 ± 0.38	13.24 ± 0.39	6.30 ± 0.12
	242.17 ± 2.95	199.23 ± 5.10	196.71 ± 5.62	165.59 ± 4.65	357.65 ± 7.66	236.15 ± 3.34	200.81 ± 11.91	179.26 ± 7.15	225.42 ± 2.24	238.1 ± 3.59	202.27 ± 2.23
Butanoic acid	49.71 ± 0.93	44.75 ± 1.90	49.28 ± 1.13	49.78 ± 0.92	79.14 ± 0.87	83.91 ± 1.32	61.74 ± 1.37	51.44 ± 1.09	69.98 ± 2.35	66.46 ± 1.18	40.73 ± 0.56
	1.22 ± 0.04	0.99 ± 0.12	0.68 ± 0.04	0.89 ± 0.03	0.57 ± 0.01	1.34 ± 0.09	1.61 ± 0.02	4.62 ± 0.51	2.02 ± 0.12	1.27 ± 0.11	0.83 ± 0.04
Valeric acid	16.36 ± 0.30	8.85 ± 0.51	4.50 ± 0.10	45.06 ± 1.96	11.82 ± 0.09	26.87 ± 0.61	6.31 ± 0.08	–	9.12 ± 0.61	22.78 ± 0.35	10.38 ± 0.24
	28.65 ± 0.58	24.58 ± 0.82	28.30 ± 0.40	14.11 ± 0.13	53.59 ± 0.62	34.20 ± 0.21	29.33 ± 0.38	36.14 ± 0.52	37.69 ± 0.59	49.21 ± 0.50	27.53 ± 0.50
Heptanoic acid	–	4.70 ± 0.43	–	–	–	–	–	–	3.64 ± 0.08	5.37 ± 0.18	–
	35.87 ± 0.36	27.01 ± 0.37	58.90 ± 0.73	48.14 ± 0.72	153.33 ± 4.05	61.67 ± 1.58	51.11 ± 1.02	64.52 ± 0.52	60.26 ± 2.10	89.11 ± 2.15	38.55 ± 1.01
Octanoic acid	885.94 ± 25.10	629.34 ± 9.38	678.09 ± 11.32	504.76 ± 12.31	764.85 ± 9.97	748.13 ± 3.93	564.99 ± 12.50	644.72 ± 17.69	556.55 ± 18.64	655.01 ± 5.63	608.43 ± 23.75
	–	–	–	–	–	–	–	–	2.44 ± 0.38	3.56 ± 0.27	–
Decanoic acid	–	–	–	–	–	1.85 ± 0.02	1.97 ± 0.09	–	–	–	–
	–	–	–	–	–	–	–	–	–	–	–
2-Methylhexanoic acid	1.93 ± 0.08	3.49 ± 0.32	15.41 ± 0.48	7.63 ± 0.35	10.48 ± 0.45	9.96 ± 0.97	12.60 ± 0.31	5.81 ± 0.19	22.78 ± 0.04	7.00 ± 0.83	3.65 ± 0.25
	1318.09 ± 62.73	1225.53 ± 40.00	1793.85 ± 69.05	1196.03 ± 8.92	1294.06 ± 9.22	1645.67 ± 27.89	1532.58 ± 12.72	2079.02 ± 12.25	1786.46 ± 9.43	958.59 ± 8.48	1322.9 ± 24.93
3-Hydroxypropionic acid	14.27 ± 0.33	12.52 ± 0.49	9.45 ± 0.51	9.44 ± 0.34	6.70 ± 0.27	19.66 ± 0.92	20.81 ± 0.57	15.24 ± 1.48	17.61 ± 1.11	12.55 ± 1.11	3.82 ± 0.10
	2.74 ± 0.03	4.00 ± 0.14	3.60 ± 0.13	6.53 ± 0.43	7.73 ± 0.54	4.56 ± 0.36	12.07 ± 0.69	3.50 ± 0.14	6.34 ± 0.29	9.37 ± 0.46	8.61 ± 0.22
Palmitic acid	0.49 ± 0.07	0.16 ± 0.005	3.04 ± 0.15	0.22 ± 0.02	0.93 ± 0.06	0.02 ± 0.01	0.35 ± 0.03	–	–	0.31 ± 0.01	0.55 ± 0.03
	0.28 ± 0.05	0.36 ± 0.02	1.53 ± 0.05	1.22 ± 0.13	1.84 ± 0.03	0.07 ± 0.01	7.34 ± 0.06	0.42 ± 0.02	0.86 ± 0.07	0.58 ± 0.02	0.15 ± 0.01
3-Hydroxybutyric acid	532.69 ± 30.17	324.21 ± 19.29	242.70 ± 10.20	284.34 ± 9.48	177.63 ± 1.06	289.22 ± 2.26	361.15 ± 7.96	243.25 ± 1.79	237.39 ± 8.24	236.94 ± 8.17	248.77 ± 2.62
	–	5.18 ± 0.03	17.60 ± 0.28	4.36 ± 0.02	7.78 ± 0.25	7.92 ± 0.20	6.04 ± 0.05	5.65 ± 0.04	8.5 ± 0.28	6.81 ± 0.06	5.77 ± 0.34
4-Methylvaleric acid	1.28 ± 0.01	1.16 ± 0.09	1.80 ± 0.09	1.90 ± 0.31	1.55 ± 0.04	2.42 ± 0.35	2.86 ± 0.24	2.48 ± 0.26	3.33 ± 0.01	1.48 ± 0.11	0.7 ± 0.06
	30.12 ± 0.71	35.76 ± 0.41	37.77 ± 1.17	41.41 ± 2.04	36.04 ± 0.42	35.00 ± 0.87	53.73 ± 1.00	27.44 ± 1.00	77.34 ± 0.61	44.67 ± 1.16	25.9 ± 0.78
Propanoic acid	18.12 ± 1.17	131.74 ± 9.30	48.15 ± 2.15	10.99 ± 0.68	215.31 ± 2.27	17.16 ± 0.47	89.89 ± 1.18	10.81 ± 0.75	17.53 ± 0.83	12.83 ± 1.73	45.84 ± 1.32
	0.88 ± 0.01	0.6 ± 0.03	0.39 ± 0.02	0.58 ± 0.02	1.60 ± 0.07	0.59 ± 0.06	2.26 ± 0.26	0.51 ± 0.01	1.39 ± 0.07	2.35 ± 0.21	0.65 ± 0.01
2-Furoic acid	–	–	–	–	–	–	–	–	–	–	–

analytical performance, with calibration curves showing R^2 greater than 0.99. LOD and LOQ ranged from 0.85 to 3.90 $\mu\text{g/L}$ and from 2.95 to 13.60 $\mu\text{g/L}$, respectively, indicating high sensitivity. Intra-day and inter-day precision ranged from 0.71% to 8.62% and from 1.03% to 7.54%, respectively. Recovery rates ranged between 92.34% and 104.12%, supporting the robustness and reproducibility of the analytical method for profiling NVOAs in distilled beverages (see Table 1).

Among the quantified compounds, lactic acid was the most abundant, with concentrations ranging from 959 to 2079 mg/L. This level exceeded that of any other NVOA by orders of magnitude. Glycolic acid was the second most prevalent, with concentrations ranging from 10.8 to 215.3 mg/L, while other acids were detected only at trace levels or in a subset of samples (see Table 2). Although NVOAs are not volatile and therefore contribute minimally to aroma through direct olfactory pathways, they are increasingly recognized as crucial modulators of Baijiu's overall flavor profile. Recent studies have demonstrated that the addition of NVOAs can attenuate the alcoholic and sweet notes while enhancing the perception of fruit, acidity, floral nuances, jujube, and grain aromas (Wang, Jing, Song, et al., 2022). Furthermore, lactic acid has been shown to influence not only taste directly through mild sourness, but also aroma release through molecular interactions with volatile compounds (Wang, Zhou, et al., 2024). For example, Wang et al.

reported a significant reduction in the threshold concentrations of ethyl lactate and ethyl acetate in the presence of lactic acid, thereby intensifying their fruity and milky characteristics (Wang, Jing, Wang, et al., 2022). These findings suggest additive or synergistic effects mediated by physicochemical interactions that enhance volatile release and perception. Given its exceptionally high concentration and functional relevance, lactic acid likely acts as a critical sensory modulator in Baijiu, helping to buffer oral harshness and promoting the integration of complex flavor elements. In fact, lactic acid and other NVOAs have been used as markers to differentiate Baijiu varieties and are thought to contribute to the smoother mouthfeel typically associated with well-aged or harmoniously blended products (Li et al., 2019; Liu & Sun, 2018; Wang, Song, et al., 2022).

3.4. PLSR-based analysis of relationships between TI features and organic acids

To better elucidate the contributions of specific organic acids to the oral stimulation characteristics of Baijiu, partial least squares regression (PLSR) was used to explore the multivariate relationships between chemical composition and sensory parameters. This technique enables the simultaneous handling of collinearity among predictors and

highlights compounds that most effectively account for variance in the temporal sensory profile (Regueiro et al., 2017).

As shown in the PLSR loading plot (Fig. 3), I_{\max} and Area, which reflect peak irritation intensity and overall sensory load, are located near the outer elliptical boundary along the positive t_1 and t_2 axes. Variables in this region are typically regarded as having stronger explanatory contributions. Several organic acids, including hexanoic acid, butanoic acid, and glycolic acid, appear in similar directions, suggesting that they may contribute to elevated or prolonged trigeminal responses. Among these, hexanoic acid stands out due to both its high abundance in strong-flavor Baijiu and its known role as a characteristic flavor compound. Its dual alignment with both chemical and sensory intensity metrics reinforces its status as a pivotal component influencing the multisensory profile of Baijiu.

In contrast, dynamic sensory features such as $TI_{5\%}$, $TI_{90\%}$, and $TD_{90\%}$ are located in the opposite quadrant of the loading plot. Notably, acetic acid is the only compound closely aligned with this early-phase cluster. This spatial relationship implies that acetic acid may play a distinctive role in modulating the temporal characteristics of oral stimulation, particularly influencing the onset and build-up of the sensation. Meanwhile, lactic acid appears on the opposite side of the plot from intensity-related parameters such as I_{\max} and Area. This positioning suggests a potential buffering effect, wherein lactic acid might serve to soften the perceived intensity of oral stimulation rather than amplify it. Notably, the three acids identified as the most influential contributors in the PLSR model, specifically hexanoic acid, acetic acid, and lactic acid, also ranked among the most abundant compounds detected in the analyzed Baijiu samples. Their prominence in both quantitative abundance and multivariate importance suggests that they are primary contributors to sensory variation. In summary, the PLSR analysis provided initial evidence that hexanoic acid may be associated with stronger irritation-related responses, that acetic acid may alter the temporal development of irritation, and that lactic acid may attenuate harshness.

3.5. Classification of Baijiu based on multiple stimulation features

To evaluate whether organic acid composition can discriminate between oral stimulation profiles in Baijiu, partial least squares discriminant analysis (PLS-DA) models were constructed. One model was constructed based on the clustering of TI parameters, representing global stimulation profiles. Four additional models were constructed using individual TI parameters, $TI_{5\%}$, $TI_{90\%}$, I_{\max} , and Area, as classification criteria, corresponding to different stages of sensory progression (Table S2). This multi-level modeling strategy allowed a comprehensive evaluation of how organic acid composition relates to stimulation dynamics.

All five models exhibited satisfactory classification performance (Fig. S1). The global model achieved a Q^2 value of 0.735, confirming that organic acid profiles provided sufficient discriminatory power to separate samples based on integrated oral stimulation characteristics. The four feature-specific models also demonstrated high predictive capacity, with Q^2 values of 0.744 ($TI_{5\%}$), 0.965 ($TI_{90\%}$), 0.953 (I_{\max}), and 0.911 (Area), indicating that differences in sensory perception at specific time points could also be explained by variations in organic acid composition.

VIP analysis identified several organic acids with high contributions across models (Table S3). Among these, hexanoic acid, acetic acid, and lactic acid appeared most consistently, suggesting their potential relevance to multiple phases of oral stimulation. These findings indicate that organic acids, as a group, carry sufficient explanatory power to differentiate between Baijiu samples with distinct temporal sensory characteristics. This observation is consistent with findings in other complex beverages, such as wine, where multi-component interactions have been shown to define attributes including astringency and prickling sensations (Cretin et al., 2018; Green et al., 2010). It may be inferred that in Baijiu, the perceived sensory balance results from the concerted effects of multiple acids rather than from a single dominant compound.

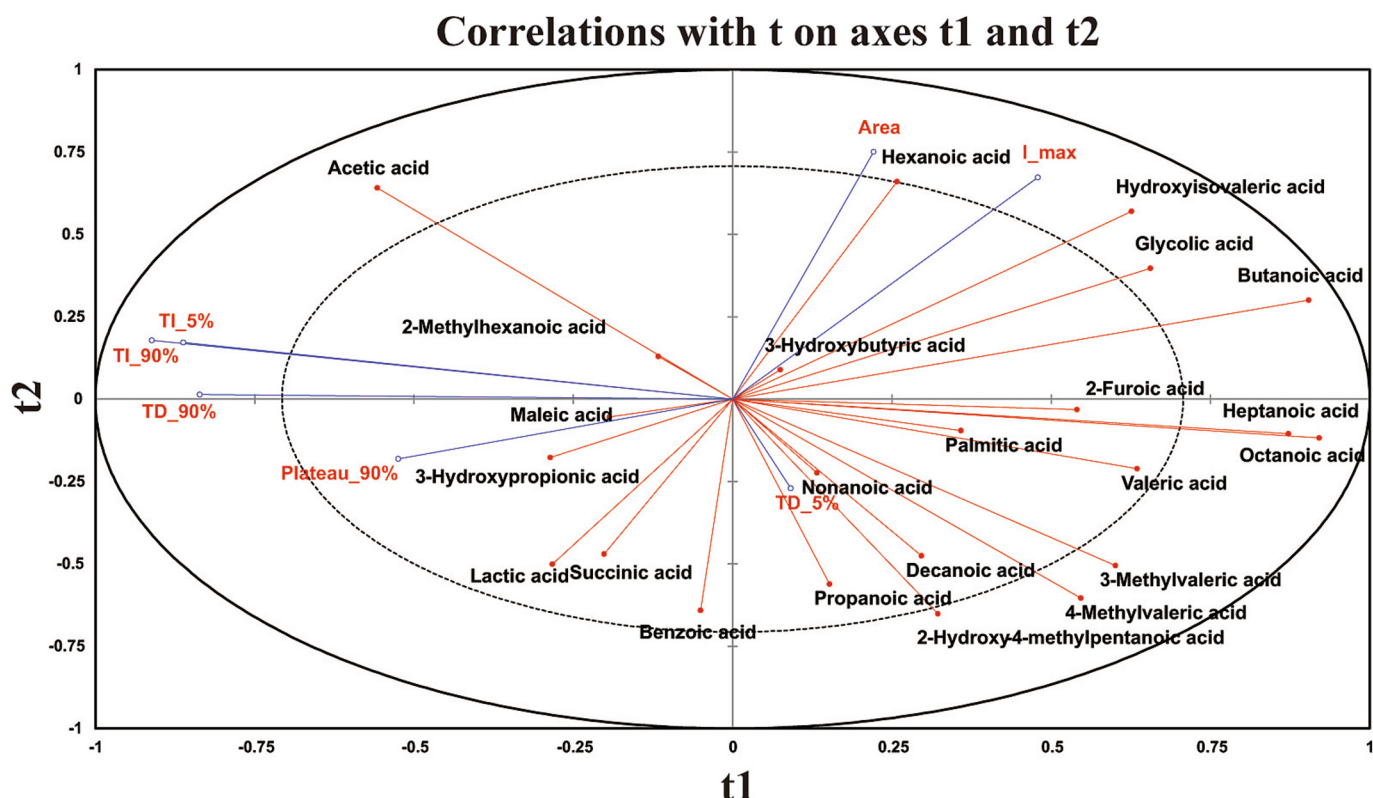


Fig. 3. PLSR loading plot of time-intensity parameters and organic acids.

3.6. Significance analysis of key organic acids in different stimulation features

To further validate the sensory relevance of organic acids identified through multivariate modeling, statistical significance testing was conducted on compounds selected by VIP analysis (Fig. 4). Ten organic acids from the global PLS-DA model were first evaluated for their concentration differences across stimulation clusters. Among them, hexanoic acid, acetic acid, and lactic acid showed statistically significant variation, confirming their consistent contribution to sensory differentiation. Additional tests were performed for each of the four feature-specific models. In the I_{\max} and Area models, lactic acid was significantly lower in samples with stronger or more persistent stimulation, supporting its hypothesized role in attenuating oral irritation. Hexanoic acid exhibited higher concentrations in samples with elevated Area values, consistent with its association with sustained sensory input. In the $TI_{5\%}$ and $TI_{90\%}$ models, lactic acid, acetic acid and octanoic acid demonstrated significant differences across onset speed categories, suggesting potential involvement in modulating early-phase perception. These results complement the outcomes of the PLS-DA and PLSR analyses and establish a subset of organic acids with both statistical and sensory relevance. The consistent differentiation of samples according to their organic acid profiles reinforces the role of these compounds in shaping the temporal dynamics of Baijiu's oral stimulation characteristics.

3.7. Correlation analysis between key organic acids and important stimulation features

Although earlier multivariate analyses identified organic acids associated with sensory variation, they did not elucidate the direction or nature of these relationships. To further clarify how specific organic acids modulate sensory perception, correlation analysis was performed between four representative compounds and four key TI parameters. These compounds were selected based on their repeated identification in earlier models and their mechanistic plausibility as modulators of sensory dynamics.

The correlation coefficients and corresponding variable distributions are shown in Fig. 5. Acetic acid exhibited strong positive correlations with both $TI_{5\%}$ and $TI_{90\%}$ ($r = 0.84$), indicating that higher concentrations of this acid were associated with longer times to initiate and build the sensory response. This supports the interpretation that acetic acid may delay the onset and progression of oral stimulation, potentially through competitive interactions with ethanol at sensory receptors or by modulating trigeminal activation thresholds (Izu et al., 2017). These findings are consistent with previous reports in alcoholic beverages where acetic acid was shown to suppress or slow the perception of ethanol-induced irritation (Lugaz et al., 2005). In contrast, octanoic acid showed significant negative correlations with both $TI_{5\%}$ and $TI_{90\%}$, suggesting a potential role in accelerating the temporal buildup of oral stimulation.

Hexanoic acid exhibited moderate positive correlations with I_{\max} ($r = 0.60$) and Area ($r = 0.40$), suggesting that it may contribute to both peak intensity and the overall sensory load of stimulation. This is aligned with prior modeling results in this study, where hexanoic acid was repeatedly associated with stronger and more persistent sensory profiles. As a major volatile acid in strong-flavor Baijiu, it may play a dual role by contributing to both olfactory and trigeminal dimensions of perception (Dong et al., 2024; Izu et al., 2017; Junge et al., 2020; Yan et al., 2024). Lactic acid did not show strong linear correlations with individual TI parameters but demonstrated a consistent pattern of lower concentrations in samples with stronger stimulation. Given its relatively high and narrow concentration range across samples, the absence of strong linear correlations may be attributed to limited variance. Nevertheless, its significant differences across stimulation groups and frequent appearance in VIP rankings suggest that lactic acid may exert a

modulatory effect, possibly attenuating the perceived sharpness or duration of ethanol-induced sensations.

Together, these results support a multi-mechanistic sensory modulation model, in which distinct organic acids contribute differentially to the initiation, intensity, and persistence of oral stimulation in Baijiu. Acetic acid and lactic acid appeared to delay or buffer the stimulation process, whereas hexanoic acid was more consistently associated with the intensity-related features I_{\max} and Area in the chemometric analyses. These mechanisms are not mutually exclusive but likely operate simultaneously within the Baijiu matrix. The overall sensory impression thus arises from the dynamic interplay of acids with contrasting functional properties, reinforcing the importance of matrix complexity in shaping mouthfeel in high-ethanol distilled beverages.

3.8. Validation of the modulatory effects of organic acids on ethanol-induced oral irritation

To confirm whether specific organic acids modulate ethanol-induced oral irritation, verification experiments were conducted using both simplified model systems and real Baijiu matrices (Fig. 6). Lactic acid, acetic acid, and hexanoic acid were selected based on their prominence in prior chemometric analyses and their differential distribution among representative samples, which suggested their potential contribution to sensory modulation. Model solutions were prepared in a 52% ethanol-water matrix containing one of the three selected acids. No co-solvent or auxiliary solvent was used during model preparation. Lactic acid (L), acetic acid (A), and hexanoic acid (H) were added at concentrations of 1645.67, 289.22, and 748.13 mg/L, respectively, based on their levels in a representative high-acid Baijiu sample. Ethanol alone served as the control (E). In parallel, three Baijiu samples were evaluated: a low-acid sample (S10), a high-acid sample (S6), and a modified version of S10 supplemented with the three selected acids using S6 as the reference basis for the selected acid levels, referred to as S10 + OA. This supplemented sample was included as a directional sensory-validation sample to examine whether increasing the selected organic acids in an authentic Baijiu matrix would shift the oral-irritation profile of S10 toward that of S6.

As shown in Fig. 6A, all acid-containing model solutions exhibited increased $TI_{5\%}$ and $TI_{90\%}$ values compared to the control. Acetic acid significantly increased both parameters ($p < 0.01$), while lactic acid selectively prolonged $TI_{90\%}$ ($p < 0.05$). Only lactic acid significantly reduced I_{\max} . Although both lactic and acetic acid groups showed a trend toward lower Area values, the differences were not statistically significant. Interestingly, although hexanoic acid showed positive associations with I_{\max} and Area in the correlation analysis, it did not significantly affect I_{\max} relative to the ethanol control in the validation experiment. This discrepancy may reflect the difference between authentic Baijiu samples, in which multiple acids and other matrix components vary simultaneously, and the model system, which assessed the effect of a single acid at one fixed concentration. This further highlights the complexity of oral irritation in Baijiu and the importance of validation experiments. Overall, these results suggest that organic acids primarily affect the early temporal profile of ethanol-induced irritation rather than its peak intensity or cumulative magnitude. Consistent patterns were observed in the Baijiu samples (Fig. 6B). The high-acid sample (S6) showed consistently higher $TI_{5\%}$ and $TI_{90\%}$ values than the low-acid sample (S10). The acid-supplemented version of the low-acid sample (S10 + OA) also displayed increased values in both parameters. These findings support the conclusion that acid composition modulates the temporal onset of oral irritation. The delayed perception of pungency may contribute to a reduced initial impact and a smoother entry profile. Mechanistically, these effects may reflect acid-related changes at the oral interface rather than simple changes in stimulus strength alone. Acetic acid may alter local acidity conditions and thereby delay the early development of ethanol-induced irritation, whereas lactic acid may attenuate the sharpness of the response through

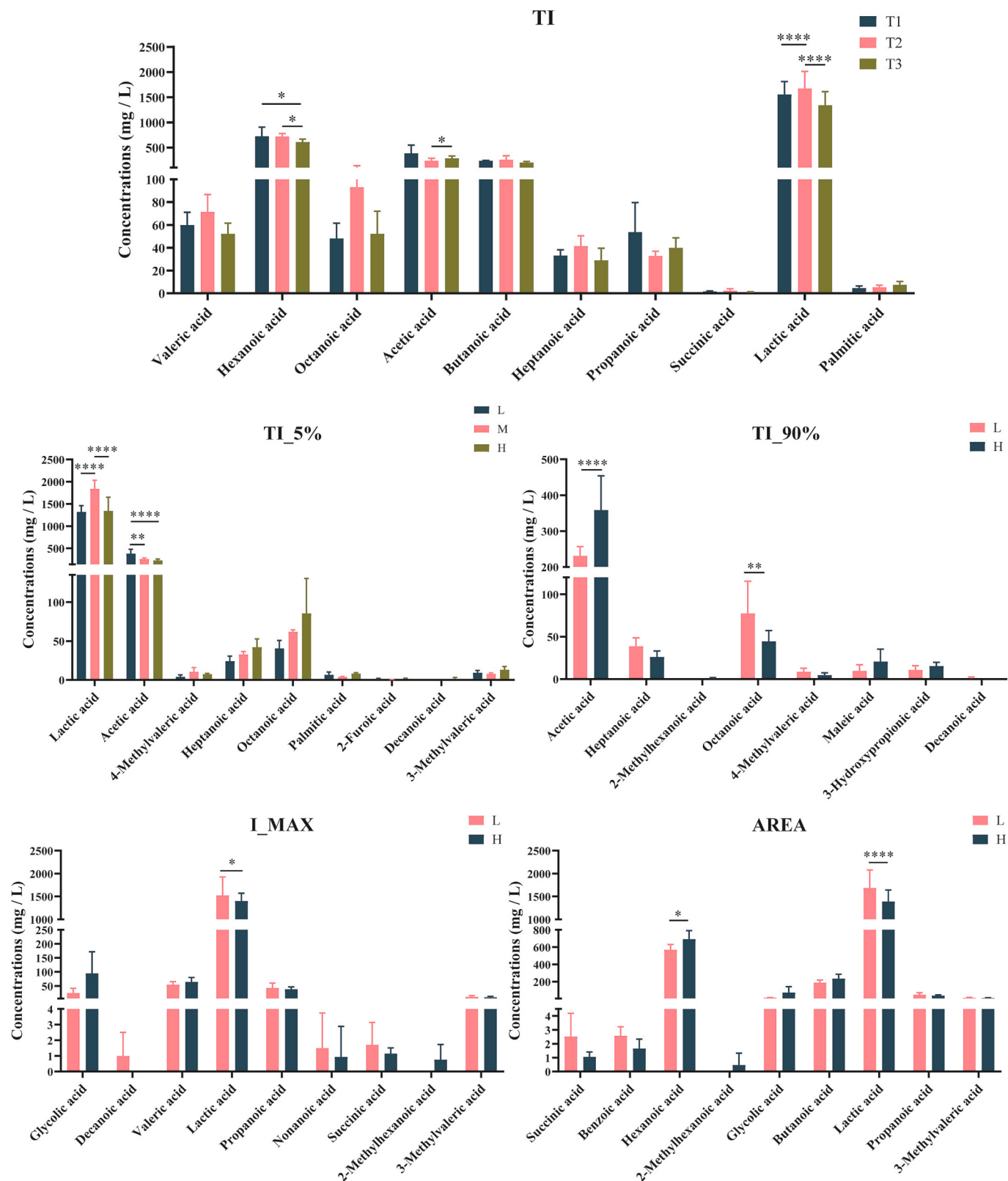


Fig. 4. Significance analysis of VIP organic acid concentrations across different TI-based sample groups. T1-T3 indicate sample clusters based on overall TI profiles. L, M, and H indicate low-, medium-, and high-value groups for the corresponding TI parameter, respectively. Asterisks indicate significant differences between groups: *, $p < 0.05$, **, $p < 0.01$, and ***, $p < 0.0001$.



Fig. 5. Pairwise correlation matrix of TI parameters and key organic acids in *Strong*-flavor Baijiu samples. I_{max} represents the peak perceived intensity, $TI_{5\%}$ and $TI_{90\%}$ represent the times required to reach 5% and 90% of the maximum intensity during the rising phase of the TI curve, and Area represents the area under the TI curve, reflecting the overall cumulative oral stimulation over time. Count refers to the frequency distribution shown in the diagonal histograms. The upper panels show Pearson correlation coefficients, the lower panels show scatter plots, and the diagonal panels show the distribution of each variable.

its high concentration, mild sourness, and possible interactions with salivary and chemesthetic processes. More generally, the sensory effects of organic acids are likely influenced not only by acidity itself, but also by differences in acid composition and their interactions with the oral environment (Izu et al., 2017; Lugaz et al., 2005; Yan et al., 2024). These factors may help explain why acetic acid more clearly affected $TI_{5\%}$ and $TI_{90\%}$, whereas lactic acid was more closely associated with reduced I_{max} . Nevertheless, these interpretations remain speculative and should be further tested using receptor-level and cellular approaches.

Static sensory ratings further validated the dynamic findings. As shown in Fig. 6C, acid-treated model solutions received significantly lower pungency scores than the ethanol control ($p < 0.001$). Similarly, S6 and S10 + OA were rated lower in pungency than S10. Since the static evaluation was performed within the early stimulation window, this reduction likely reflects the delayed onset of irritation revealed in dynamic analysis. These results further support the role of organic acids in modulating the initial perception of ethanol-induced irritation. This is consistent with previous findings that basic taste components, particularly sourness, can influence chemesthetic sensations by altering both their perceived intensity and temporal progression. Sensory interactions between taste and trigeminal inputs have been shown to delay irritation onset and reduce perceptual sharpness in various contexts. A concentration-dependent inhibitory effect of organic acids on oral

pungency has also been observed in related studies (Y. He et al., 2025; Smutzer et al., 2018). Building upon these findings, the present study suggests that organic acids contribute to shaping the temporal profile of oral irritation and may partly underlie sensory characteristics such as a softer entry, smoother mouthfeel, and more persistent aftertaste in Baijiu. Although several organic acids were identified as being associated with differences in temporal irritation features, the underlying molecular mechanisms were not directly investigated. In particular, how these acids interact with ethanol at the receptor level or influence downstream cellular signaling pathways remains unclear. In addition, the present study was limited to 52% vol *Strong*-flavor Baijiu samples from a single production year, and the validation experiments were conducted using representative acid concentrations derived from the selected samples rather than a gradient concentration design. Accordingly, broader Baijiu sample sets spanning different flavor styles and production years, together with dose-dependent validation experiments at varying ethanol concentrations, will help to further assess the generality and concentration-dependent effects of these findings. Further studies using receptor-level assays and cellular models will also be valuable for clarifying the biological basis of acid-mediated modulation of ethanol-induced oral irritation.

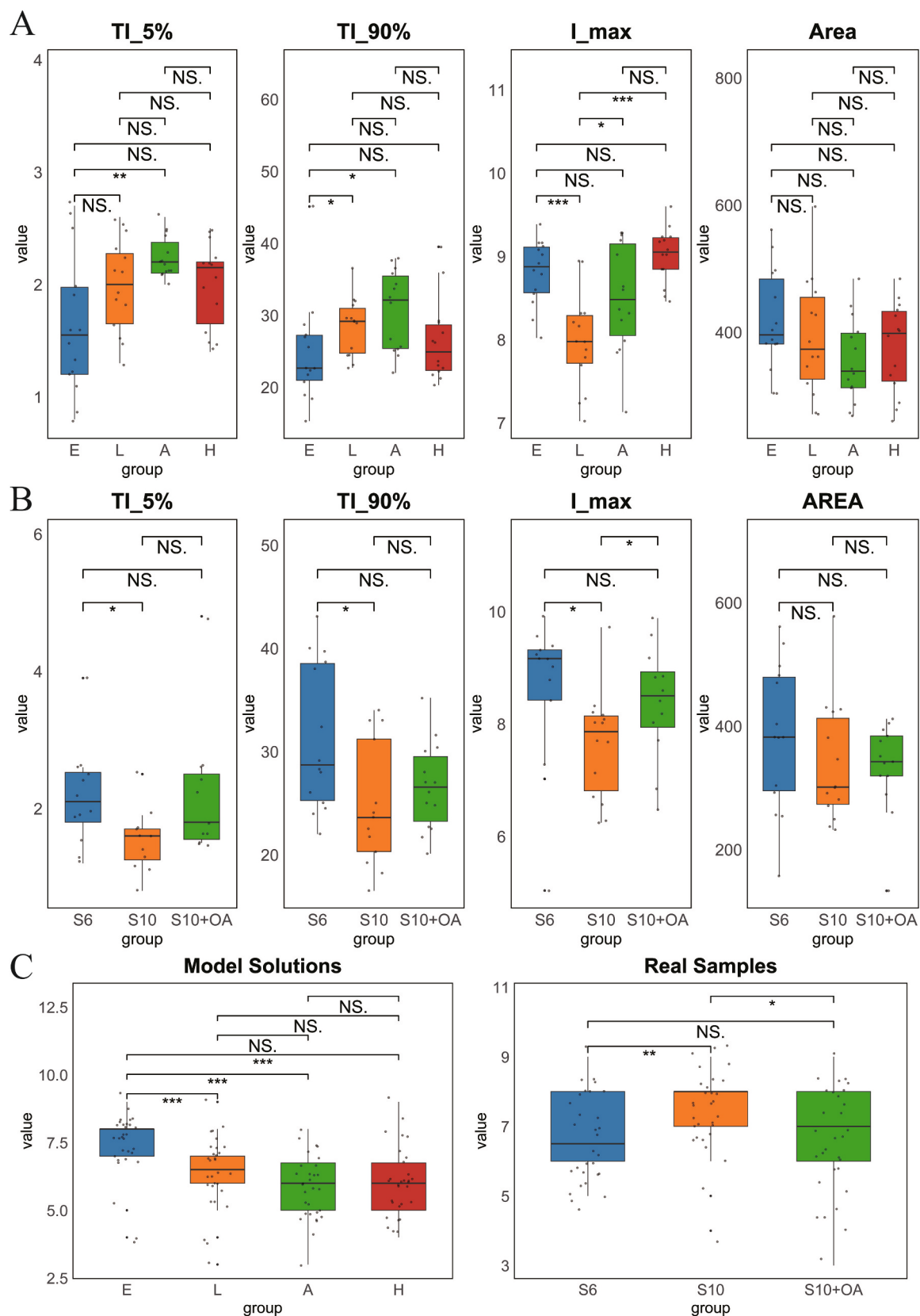


Fig. 6. Validation of the effects of organic acids on oral irritation using TI analysis (model solutions (A) and real Baijiu samples (B)) and static sensory evaluation (C). E, ethanol-only control; L, lactic acid model; A, acetic acid model; H, hexanoic acid model; S10, low-acid Baijiu sample; S10 + OA, low-acid sample supplemented with key organic acids; S6, high-acid Baijiu sample.

4. Conclusion

In conclusion, this work reveals that organic acids dynamically reshape the temporal progression of ethanol-induced oral irritation in Baijiu, offering new insight into sensory modulation in high-strength alcoholic beverages. Through integration of time-intensity sensory analysis and quantitative profiling of 23 organic acids across 11 representative samples, a consistent pattern of stimulation was established, encompassing variations in onset, intensity, and persistence. Lactic acid, acetic acid, and hexanoic acid were identified as principal contributors to these differences. Validation experiments in both model solutions and authentic Baijiu matrices confirmed that lactic and acetic acids significantly delayed the initiation and build-up of irritation, whereas hexanoic acid emerged as a relevant compound in the chemometric analyses, although its isolated effect was not statistically confirmed in the single-acid validation test. These findings suggest that organic acids influence not only the magnitude but also the temporal trajectory of ethanol-induced oral irritation, providing a novel dynamic perspective on how specific organic acids shape irritation perception in Baijiu. The integrative approach developed here, combining chemical profiling, temporal sensory analysis, and functional validation, provides a scalable framework for identifying dynamic sensory modulators in complex fermented systems. Although this study focused on the effects of individual organic acids, potential synergistic or antagonistic interactions among coexisting acids, as well as matrix interactions with other flavor compounds, may also influence oral stimulation and warrant further investigation in future studies.

CRedit authorship contribution statement

Guangnan Wang: Writing – original draft, Conceptualization. **Charles Spence:** Writing – review & editing, Supervision. **Wei Lu:** Methodology, Investigation. **Kui Sun:** Methodology, Investigation. **Fuping Zheng:** Writing – review & editing, Conceptualization. **Xing-qian Ye:** Writing – review & editing, Conceptualization. **Baoguo Sun:** Supervision, Resources. **Huan Cheng:** Supervision, Resources.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.fochx.2026.103976>.

Data availability

Data will be made available on request.

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