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Influence of the ageing time on the quality of three kinds of cold-climate mountain grape brandy

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Abstract: Northeast China boasts abundant resources of cold mountain grapes. This study focused on three varieties of cold mountain grape brandy, Beibing Hong (BBH), Shuanghong (SH), and Zuoshanyi (ZSY), with varying ageing periods to investigate changes in their physicochemical properties, nutrients, and aroma compounds. Results showed that alcohol content and pH gradually decreased with ageing, while total acidity (including inorganic acids) increased. Specific organic acids declined over time, whereas polyphenolic compounds increased with longer ageing. Furthermore, the total quantity of aroma substances in all three brandies was positively correlated with ageing duration, with 18-month-aged BBH brandy exhibiting the highest content and the richest aromatic variety. Clustering analysis via heat maps revealed that brandies aged 12 and 18 months grouped together, showing that ageing time correlated positively with most esters and alcohols and negatively with acids. No significant variations were observed in the contents of terpenes, aldehydes and ketones among the three brandy varieties.

Keywords: brandy; barrel ageing; physicochemical properties; grape distillates

Brandy, a distilled spirit derived from the Dutch word *Brandewijn* (literally meaning 'burnt wine') (Tsakiris et al. 2014), is considered a French speciality. It is primarily produced from grapes through fermentation, distillation, oak barrel ageing, and blending processes (Yang et al. 2011). Brandy embodies a rich cultural heritage and technical tradition. As a carefully distilled spirit, its main components include alcohol, organic acids, polyphenols, and volatile compounds (Schwarz et al. 2011). The type and concentration of organic acids significantly influence the biological stability, flavour quality, and ageing potential of brandy

(Chidi et al. 2018; Zheng et al. 2019). Although organic acids are present in lower concentrations than sugars in grape juice, they play a critical role in shaping the overall taste profile (Madrera et al. 2003b). Among organic acids, tartaric acid is generally the most abundant and influential, followed by malic, citric, and succinic acids (Lima et al. 2015; Yinshan et al. 2017). Polyphenols, important secondary metabolites in grapes, are gradually released during barrel ageing and maturation, contributing decisively to the unique flavour and quality of the spirit (Canas et al. 2008). Schreier et al. (1979) highlighted that the contents of organic acids and polyphenols

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serve as key indicators for determining the age of brandy. Volatile compounds, such as aldehydes, ketones, and ethers, also play an essential role in shaping the aroma profile of brandy (Yan et al. 2024).

In the brandy industry, the ageing process in oak barrels is a critical step that decisively influences the quality of the final product (Madrera et al. 2013). During ageing, polyphenols are continuously extracted from the oak, while lignin in the barrels contributes new aromatic compounds, enriching the flavour profile of the brandy. The ageing process involves a series of complex chemical reactions (Muñoz-Redondo et al. 2023), such as the oxidation of alcohols to aldehydes, further oxidation of aldehydes to acids, esterification of acids and alcohols to form esters, and reactions between alcohols and aldehydes producing acetal compounds. Additionally, compounds such as carbohydrate degradation products, volatile phenols, oak lactones, terpenes, and tannins dissolve into the spirit (Guerrero-Chanivet et al. 2020). These reactions contribute to a significant increase in the polyphenol content of brandy during ageing (Virirot et al. 1993; Madrera et al. 2003a, b). The aroma components become more complex and abundant, and the brandy mellows over time, transforming from a pungent and harsh spirit just after distillation into a rounder and smoother beverage. The colour also deepens to a golden yellow as a result of prolonged ageing and the continuous extraction of compounds from the oak barrels (Nie et al. 2023). Consequently, natural ageing yields brandy with desirable colour, taste, and aroma, resulting in a high-quality product (Tian et al. 2022).

The aroma of brandy is closely influenced by factors such as ageing time and grape variety (Xiang et al. 2020; Guerrero-Chanivet et al. 2024). In recent years, research on brandy quality has primarily focused on its nutritional components, aroma substances, and the effects of different grape varieties and production regions. However, most studies concentrate on brandies from traditional producing areas, with limited research available on brandies made from cold-climate mountain grapes. Additionally, there are a few reports detailing the quality changes of cold-climate mountain grape brandy throughout the production process.

In Northeast China, mountain grapes have adapted to the cold climate, resulting in grapes with low sugar content, high acidity, moderate aroma, high yield, and strong disease resistance (Ma et al. 2022). These characteristics make them particularly well-suited for brandy production. Niangqinggu Winery, located in the Niangqinggu Grape Industrial Park in Shuangyashan City, Heilongjiang Province, operates a mountain grape

plantation spanning approximately 266.67 hectares. Leveraging unique natural conditions and strict organic cultivation practices, the winery has successfully developed cold mountain grape varieties ideal for brandy brewing, providing rich and high-quality raw materials for brandy production.

In this study, three varieties of cold mountain grapes, Zuoshanyi (ZSY), Beibing Hong (BBH), and Shuanghong (SH), cultivated at Niangqinggu Winery, were used as raw materials. The changes in physico-chemical properties, nutrient content, aroma substances, and sensory attributes of these three grape varieties were analysed across different ageing periods. Analytical techniques such as liquid chromatography-mass spectrometry (LC-MS) and gas chromatography-mass spectrometry (GC-MS) were employed to investigate these changes, providing theoretical and practical insights for the industrial development of cold mountain grape brandy.

MATERIAL AND METHODS

Chemicals and reagents. Three cold mountain grape brandy varieties (ZSY, SH, BBH), provided by Niangqinggu Winery (Shuangyashan, Heilongjiang Province, China), were aged in oak barrels for 6, 12, and 18 months, respectively. Analytical standards including 3-octanol, tartaric acid, malic acid, lactic acid, citric acid, succinic acid, fumaric acid, and adipic acid were sourced from Shanghai Yuanye Biotechnology Co., Ltd. (China). In addition, vanillic acid, *p*-Coumaric acid, *o*-Coumaric acid, protocatechuic acid, caffeic acid, *p*-hydroxybenzoic acid, and epicatechin were obtained from Shanghai Anpu Experimental Technology Co., Ltd. (China).

Determination of ethanol, total acid, pH and total sugars and dry extract contents. Ethanol content was measured using an alcohol meter (Yaohua Instrument Factory, China) following the standard method outlined in GB 5009.225-2023. Total acidity was determined via automatic potentiometric titration in accordance with GB/T 12456-2021. The pH was determined directly with a pH meter (Shanghai Scientific Instrument Co., Ltd., China). Total sugar and dry extract contents were determined following the general analytical methods for wine and fruit wine in accordance with GB/T 15038-2006.

Determination of nutrient contents. Organic acid contents were determined using high-performance liquid chromatography (HPLC) (Agilent Technologies Inc., USA) following the method specified in standard GB 5009.157-2016. A 5 mL homogeneous sample was weighed into a 25 mL volumetric flask, diluted to the

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calibrated volume with ultrapure water, and then filtered through a 0.45 μm hydrophilic filter membrane prior to HPLC injection. The analysis was performed using a C18 column (250 \times 4.6 mm, 5 μm particle size) with a detector set at 210 nm. The mobile phase consisted of solvent A (0.1% phosphoric acid solution) and solvent B (methanol) at a volume ratio of 97.5 : 2.5 (v/v). Gradient elution was carried out over 13 min, with the column temperature maintained at 40 $^{\circ}\text{C}$.

Polyphenolic compounds were measured using the method described by Jaitz et al. (2010). A 5 mL wine sample was diluted to 50 mL with 10% methanol solution. Then, 1 mL of the diluted sample was filtered through a 0.45 μm hydrophilic filter membrane. Phenolic compounds were determined using an LC-MS (TQXS, Waters Technology Co., Ltd., USA). The LC column was a Luna Omega 1.6 μm Polar C18 (100 \times 2.1 mm). The mobile phases consisted of solvent A (5 mmol·L⁻¹ ammonium acetate) and solvent B (acetonitrile). The gradient elution program was as follows: 0–0.5 min, 50% B; 0.5–0.8 min, 50–10% B; 0.8–4.0 min, 10% B; 4.0–4.1 min, 10–50% B; 4.1–5.0 min, 50% B. The flow rate was maintained at 0.25 mL·min⁻¹, the column temperature was set at 40 $^{\circ}\text{C}$, and the injection volume was 10 μL . Mass spectrometry conditions were: electrospray ionisation in negative mode (ESI-), capillary voltage 2.5 kV, taper voltage 65 V, desolvation gas temperature was set at 150 $^{\circ}\text{C}$, and source temperature was maintained at 150 $^{\circ}\text{C}$.

Determination of aroma compounds. The detection of aroma substances was performed using headspace solid-phase micro-extraction (Supelco Corporation, USA) combined with GC-MS (7890A, Shimadzu Corporation, Japan), following the method of Li et al. (2020). A total of 5 mL of sample was placed into a 20 mL headspace vial, to which 50 μL of the internal standard 3-octanol and 1.5 g of NaCl, along with a stirring bar, were added. The vial was equilibrated in a 55 $^{\circ}\text{C}$ water bath for 10 min. Then, the extraction fibre (50/30 μm DVB/CAR/PDMS) (Supelco Corporation, USA) was inserted into the headspace vial and exposed for 10 min. GC-MS analysis was conducted using a Shimadzu gas chromatography system equipped with a DB-Wax column. Helium was used as the carrier gas at a flow rate of 1.15 mL·min⁻¹ with a split ratio of 5 : 1. The temperature program was as follows: initial hold at 40 $^{\circ}\text{C}$ for 2 min, ramp up at 6 $^{\circ}\text{C}\cdot\text{min}^{-1}$ to 100 $^{\circ}\text{C}$, then increase at 5 $^{\circ}\text{C}\cdot\text{min}^{-1}$ to 200 $^{\circ}\text{C}$, followed by a ramp at 10 $^{\circ}\text{C}\cdot\text{min}^{-1}$ to 240 $^{\circ}\text{C}$, which was held for 6 min. Using 3-octanol as the internal standard, quantification was performed via peak area normalisation. Aroma compounds were identified by comparison with vola-

tile compound spectra in the NIST database, enabling both qualitative and quantitative analysis.

Statistical analysis. Qualitative and quantitative analyses of aroma substances were performed using Excel (2021, Microsoft Corporation). One-way ANOVA was conducted with IBM SPSS Statistics 26 to assess statistical significance. Origin Pro 2023 (OriginLab) was used for plotting histograms and scatterplots, as well as for conducting principal component analysis (PCA) and generating clustering heatmaps.

RESULTS AND DISCUSSION

The impact of ageing time on physicochemical indicators of brandy

Analysis of the change of alcohol content in the ageing process. During the ageing and blending processes, water was gradually added to reduce the alcohol content, ultimately adjusting the alcohol strength of the brandy to approximately 40% vol. The rate of alcohol reduction directly affects the interactions between ethanol and water molecules, which play a crucial role in the taste profile of the brandy. As shown in Figure 1, the alcohol content of the three distilled spirits exhibited a decreasing trend during oak barrel ageing, consistent with the findings of Tarko et al. (2023), who observed a gradual decline in alcohol content across six Chardonnay brandies with varying ageing times.

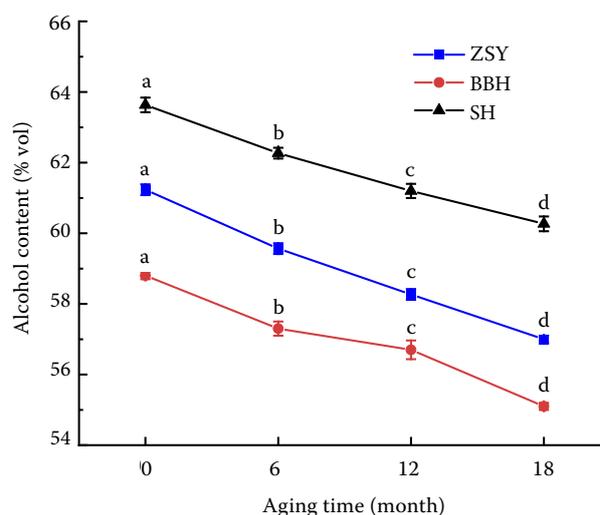


Figure 1. Changes in alcohol content during ageing of brandy

^{a,b} different letters indicate significant differences of the same product between the same index and different ageing time ($P < 0.05$)

BBH – Beibing Hong; SH – Shuanghong; ZSY – Zuoshanyi

This decrease in alcohol content is primarily attributed to the volatilisation of ethanol during ageing. Specifically, the alcohol content of BBH decreased slowly from 6 to 12 months of ageing, followed by a more rapid reduction from 12 to 18 months. Following 18 months of oak barrel ageing, SH brandy retained the highest alcohol content, whereas BBH exhibited the lowest alcohol content among the three varieties.

Analysis of the change of total acid and pH in the ageing process. Acid substances significantly influence the taste and aroma of brandy. These acids originate from the oxidation of alcohols and are extracted from

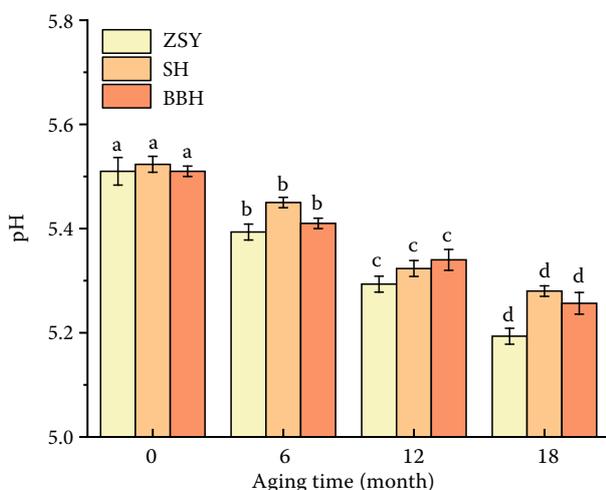


Figure 2. Changes in total acid during ageing of brandy

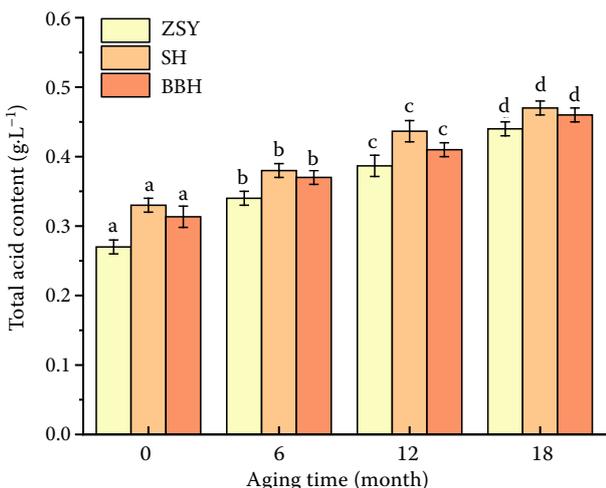


Figure 3. Changes in pH during ageing of brandy

^{a,b}different letters indicate significant differences of the same product between the same index and different ageing time ($P < 0.05$)

BBH – Beibing Hong; SH – Shuanghong; ZSY – Zuoshanyi

oak barrels during the ageing process. As illustrated in Figure 2, the total acid content of all three brandy types increased over time, with the levels ranking from highest to lowest as SH, BBH, and ZSY.

Correspondingly, as the total acid content rose, the pH values decreased. Figure 3 shows that the pH of all three brandies declined during ageing, with the order from highest to lowest pH being SH > BBH > ZSY.

The impact of storage time on nutritional substances in brandy

Analysis of the change of organic acid. Tartaric acid, malic acid, and adipic acid were detected in the brandy samples, while other organic acids were not observed. Some low-boiling-point organic acids, such as acetic acid and propionic acid, volatilises along with ethanol during the ageing process. Additionally, organic acids reacted with alcohols to form esters, a key contributor to brandy's aroma, leading to a decrease in the content of free organic acids (Comuzzo et al. 2015). The tartaric acid content showed a slight increase at 12 months of ageing, followed by a gradual decline (Figure 4a). This pattern may be explained by the transfer of organic acids from the oak barrels exceeding the amount of tartaric acid consumed in esterification during the 6–12 month period (Sánchez-Guillén et al. 2019). Malic acid content in all three brandies decreased throughout the ageing period (Figure 4b). After 18 months, SH brandy had the highest malic acid content, BBH the lowest, and ZSY showed a relatively stable trend. As depicted in Figure 4c, adipic acid levels in SH brandy declined more rapidly after 6 months, with the highest adipic acid content found in BBH brandy after 18 months.

Analysis of the change in the polyphenolic compounds. During the ageing process of brandy in oak barrels, a series of physicochemical interactions occur between the brandy and oak components, such as tannin extraction and the degradation of cellulose and lignin (Valcárcel-Muñoz et al. 2021). It is well established that many polyphenols are introduced or formed during barrel ageing, primarily phenolic acids like gallic acid, protocatechuic acid, and vanillic acid (Zhang et al. 2018). For instance, vanillic acid was not detected in SH brandy immediately after distillation, but appeared after 6 months of ageing in oak barrels, which supports this established observation. Similarly, caffeic acid and *p*-hydroxybenzoic acid were absent post-distillation in SH and BBH brandies but emerged during ageing, possibly due to extraction from the oak barrels or the hydrolysis of tannins (Xiang et al. 2020). The detailed changes

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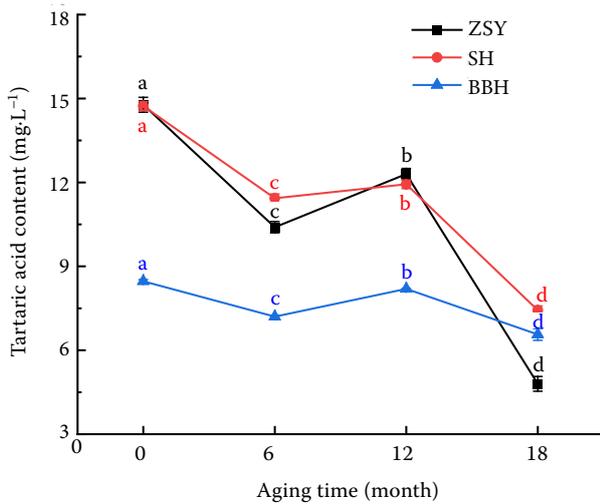


Figure 4a. Changes in tartaric acid during ageing of three brandies

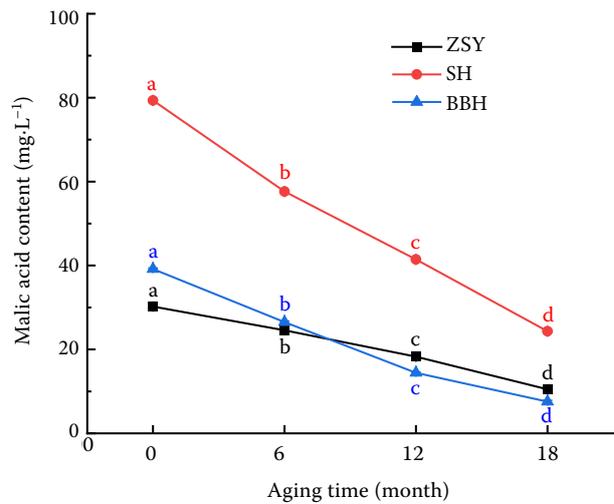


Figure 4b. Changes in malic acid during ageing of three brandies

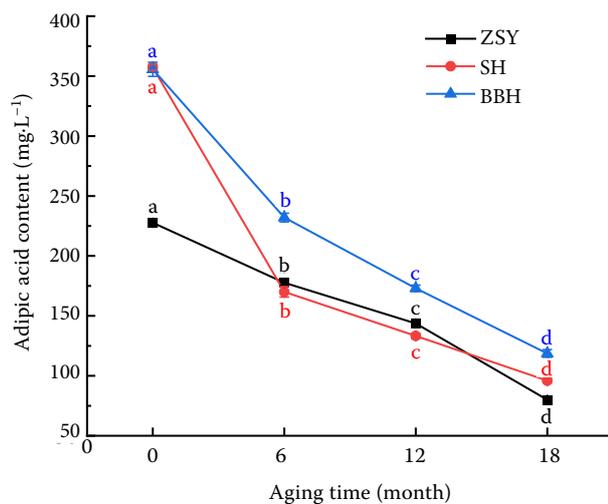


Figure 4c. Changes in adipic acid during ageing of three brandies

^{a,b}different letters indicate significant differences of the same product between the same index and different ageing time ($P < 0.05$)

BBH – Beibing Hong; SH – Shuanghong; ZSY – Zuoshanyi

of polyphenols throughout the ageing process of the three brandies are summarised in Table 1.

The increase of *p*-Coumaric acid and *o*-Coumaric acid in the three brandies was not significant with extended ageing time, which may be related to chemical transformations involving furans or furfurals. As shown in Table 1, ten types of phenolic acid compounds in the three

brandies generally increased as ageing time progressed, consistent with the trends reported (Valcárcel-Muñoz et al. 2021). This indicates that the ageing process has a substantial impact on brandy quality; the longer the ageing period, the greater the diversity of phenolic acid compounds present, thereby enhancing the overall quality of the brandy (Winstel et al. 2022).

Table 1. Changes of polyphenols during the ageing process of three brandies

Polyphenolic compounds	Phenolic acid content ($\mu\text{g}\cdot\text{L}^{-1}$)											
	Zuoshanyi				Shuanghong				Beibing Hong			
	6 months	12 months	18 months	6 months	12 months	18 months	6 months	12 months	18 months	6 months	12 months	18 months
Epicatechin	33.51 ± 0.43 ^a	46.92 ± 0.26 ^b	57.51 ± 0.53 ^c	35.21 ± 0.64 ^a	54.22 ± 0.52 ^b	74.51 ± 0.69 ^c	21.32 ± 0.12 ^a	56.32 ± 0.33 ^b	89.6 ± 0.95 ^c	21.32 ± 0.12 ^a	56.32 ± 0.33 ^b	89.6 ± 0.95 ^c
Syringic acid	21.24 ± 0.44 ^a	42.35 ± 0.24 ^c	44.26 ± 0.29 ^b	21.10 ± 0.28 ^a	42.33 ± 0.42 ^b	64.36 ± 0.39 ^c	31.22 ± 0.35 ^a	100.61 ± 0.41 ^b	111.32 ± 0.39 ^c	31.22 ± 0.35 ^a	100.61 ± 0.41 ^b	111.32 ± 0.39 ^c
Ferulic acid	70.37 ± 0.86 ^a	89.65 ± 0.74 ^b	100.21 ± 0.63 ^c	38.62 ± 0.66 ^a	67.32 ± 0.37 ^b	85.63 ± 0.19 ^c	42.22 ± 0.55 ^a	75.32 ± 0.42 ^b	81.61 ± 0.39 ^c	42.22 ± 0.55 ^a	75.32 ± 0.42 ^b	81.61 ± 0.39 ^c
Caffeic acid	53.63 ± 0.36 ^a	78.66 ± 0.27 ^b	89.42 ± 0.52 ^c	4.92 ± 0.14 ^a	16.72 ± 0.21 ^c	18.43 ± 0.26 ^b	11.22 ± 0.22 ^a	52.33 ± 0.39 ^b	69.42 ± 0.37 ^c	11.22 ± 0.22 ^a	52.33 ± 0.39 ^b	69.42 ± 0.37 ^c
Gallic acid	99.67 ± 0.22 ^a	142.31 ± 0.51 ^b	153.62 ± 0.26 ^c	89.53 ± 0.52 ^a	124.42 ± 0.37 ^b	142.13 ± 0.34 ^c	21.32 ± 0.64 ^a	96.82 ± 0.34 ^b	124.52 ± 0.59 ^c	21.32 ± 0.64 ^a	96.82 ± 0.34 ^b	124.52 ± 0.59 ^c
<i>p</i> -Coumaric acid	123.43 ± 0.63 ^a	132.12 ± 0.27 ^b	147.44 ± 0.67 ^c	11.72 ± 0.51 ^a	24.53 ± 0.31 ^b	36.43 ± 0.25 ^c	5.91 ± 0.37 ^a	19.52 ± 0.69 ^b	21.32 ± 0.64 ^c	5.91 ± 0.37 ^a	19.52 ± 0.69 ^b	21.32 ± 0.64 ^c
<i>o</i> -Coumaric acid	132.43 ± 0.35 ^a	141.15 ± 0.45 ^b	163.23 ± 0.96 ^c	21.41 ± 0.21 ^a	35.40 ± 0.29 ^b	44.93 ± 0.37 ^c	10.32 ± 0.34 ^a	24.82 ± 0.39 ^b	31.21 ± 0.28 ^c	10.32 ± 0.34 ^a	24.82 ± 0.39 ^b	31.21 ± 0.28 ^c
Protocatechuic acid	85.63 ± 0.24 ^a	96.32 ± 0.38 ^b	112.31 ± 0.33 ^c	27.62 ± 0.41 ^a	39.52 ± 0.38 ^b	46.82 ± 0.46 ^c	13.22 ± 0.33 ^a	25.67 ± 0.19 ^b	33.23 ± 0.61 ^c	13.22 ± 0.33 ^a	25.67 ± 0.19 ^b	33.23 ± 0.61 ^c
<i>p</i> -hydroxybenzoic acid	52.32 ± 0.36 ^a	74.21 ± 0.47 ^b	78.43 ± 0.25 ^c	15.72 ± 0.36 ^a	29.51 ± 0.28 ^b	37.43 ± 0.33 ^c	7.62 ± 0.13 ^a	12.10 ± 0.21 ^b	15.32 ± 0.19 ^c	7.62 ± 0.13 ^a	12.10 ± 0.21 ^b	15.32 ± 0.19 ^c
Vanillic acid	21.32 ± 0.39 ^a	36.21 ± 0.45 ^b	42.3 ± 0.49 ^c	8.91 ± 0.21 ^a	21.42 ± 0.19 ^b	15.92 ± 0.34 ^b	14.62 ± 0.21 ^a	35.43 ± 0.38 ^b	41.33 ± 0.31 ^c	14.62 ± 0.21 ^a	35.43 ± 0.38 ^b	41.33 ± 0.31 ^c

^{a-c}different letters indicate significant differences of the same product between the same index and different days ($P < 0.05$)

The impact of ageing time on aroma compounds in brandy

Relationship between aroma components and ageing time of ZSY brandy. A total of 88 aroma substances were detected in ZSY brandy aged for 6, 12, and 18 months, including 43 esters, 18 alcohols, 4 acids, 4 terpenes, 9 aldehydes and ketones, and 10 other compounds. To more intuitively illustrate the changes in aroma components of ZSY brandy throughout the 6- to 18-month ageing process, a clustering correlation heatmap of the 35 main aroma substances is presented in Figure 7.

As shown in Figure 7, most aroma substances exhibited a positive correlation with increasing ageing time, with the highest concentrations observed in the brandy aged for 18 months, highlighted in red on the heatmap. During the ageing process, the levels of diethyl oxalate and *sec*-butyl nitrite showed a negative correlation with ageing time. Diisobutyl oxalate was not detected after 18 months of ageing, while ethyl isocaproate appeared only after 12 months. Other esters generally showed positive correlations with ageing duration.

The contents of several alcohol-related aroma substances, including *cis*-cyclopentane-1,3-diol, isobutanol, 1,9-nonanediol, 1-octanol, *DL*-1,2-hexanediol, phenylethanol, and epicucalyptus oil, were negatively correlated with ageing time. Conversely, the levels of 2,6-dimethyl-5,7-octadien-2-ol, 3-pyrrolidinol, 3-methyl-1,5-pentanediol, and 5-methyl-3-heptanol increased as ageing progressed. Regarding acids, the aroma content of *n*-decanoic acid, 2,2-dimethylsuccinic acid, and *n*-butylboronic acid decreased with ageing, while diethylboronic acid showed a positive correlation with ageing time. Terpene levels remained largely unchanged, with 7-methyl-1-octene detected only after 12 months of ageing. Among aldehydes and ketones, the contents of 3-octanone and benzaldehyde increased with ageing, whereas other compounds remained stable. Cluster analysis indicated that the aroma profiles of ZSY brandy aged 12 and 18 months grouped into the same category.

With increasing ageing time, the concentration of volatile esters in brandy tends to rise, while the total amount of higher alcohols remains relatively stable, showing only a slight increase. This slight increase is primarily due to the evaporation of ethanol and water; since higher alcohols evaporate less readily, their relative proportion increases (Duan et al. 2024). Aldehydes, ketones, and terpenes constitute a relatively small fraction of the total aroma components, less than 1%. During ageing, alcohols are oxidised to aldehydes by oxygen exposure. These aldehydes can then react with ethanol to form acetals, and with longer

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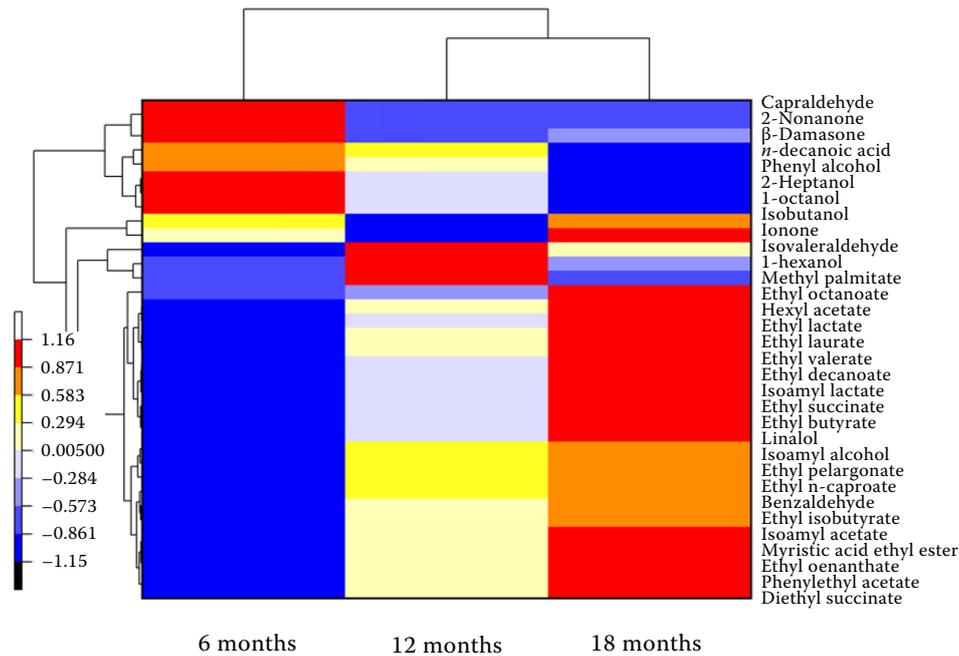


Figure 7. Thermogram of changes in aroma substance content of Zuoshanyi brandy with ageing time

ageing times, the levels of both aldehydes and acetals increase (Caldeira et al. 2016; Yan et al. 2024).

Relationship between aroma components and ageing time of SH brandy. A total of 98 aroma substances were detected in SH brandy aged for 6, 12, and 18 months, including 50 esters, 13 alcohols, 6 acids, 3 terpenes, 10 aldehydes and ketones, and 16 other

compounds. To more intuitively illustrate the changes in aroma components of SH brandy over the ageing process, a clustering correlation heat map of 37 main aroma substances was generated (Figure 8).

From Figure 8, it is evident that the SH brandy aged for 12 months contains a greater variety of aroma substances, represented by deeper red colours on the heat map.

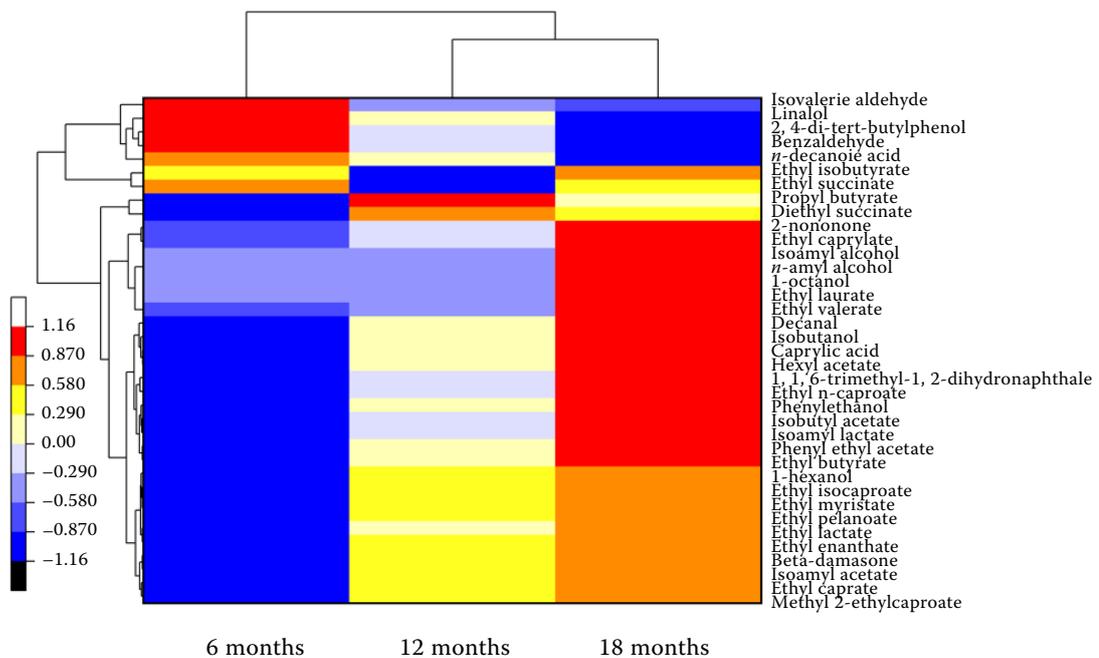


Figure 8. Heat map of the change of aroma substance content of Shuanghong brandy with ageing time

The contents of trimethylhexamethylene diisocyanate and *sec*-butyl nitrite showed a negative correlation with ageing time. In contrast, compounds such as pentadecyl acrylate, 2,8-dimethylmethyl undecanoate, isobutyl acetate, ethyl acetoacetate, and glyceryl monocaprinate were detected only after 18 months of ageing. Other esters exhibited either positive correlations with ageing time or showed no significant change. Among the alcohols, the contents of phenylethanol, isobutanol, and *n*-pentanol decreased with ageing time, while other alcohols either increased or remained stable. Vederol was detected exclusively after 18 months of ageing. For acids, caprylic acid and *n*-capric acid contents declined with ageing time, whereas guanidine acetic acid increased. Terpenes remained largely unchanged, with hexahydro-2,2-dimethyl-1,3-benzodioxole detected only after 6 months of ageing. In the aldehydes and ketones group, benzaldehyde and damascone showed positive correlations with ageing time, while others showed no significant variation.

Cluster analysis indicated that the aroma profiles of SH brandy aged for 12 and 18 months belong to the same category.

Relationship between aroma components and ageing time of BBH brandy. A total of 117 aroma substances were detected in BBH brandy aged for 6, 12, and 18 months, including 51 esters, 27 alcohols, 5 acids,

2 terpenes, 14 aldehydes and ketones, and 18 other compounds. To more clearly illustrate the changes in aroma components of the BBH brandy during the ageing process, a clustering correlation heat map of 34 main aroma substances was generated (Figure 9).

As shown in Figure 9, the variety of aroma substances increased with ageing time, particularly after 12 months, with deeper red colours on the heat map indicating higher concentrations. Throughout the ageing process, all ester aroma substances exhibited either a positive correlation or no significant change with ageing time; no esters showed a negative correlation. The content of isobutanol showed a negative correlation with ageing time, while other alcohols were either positively correlated or showed no obvious trend. Among acids, only 2,2-dimethylsuccinic acid displayed a negative correlation with ageing time; the others either increased or remained stable. β -Hydroxyisovaleric acid was detected starting from 6 months of ageing. In the terpene category, the content of β -damascone showed a positive correlation with ageing time, whereas hexadiethyldicyclopentadiene was no longer detected after 12 months of ageing. For aldehydes and ketones, compounds such as furfural, 3-octanone, damascenone, isovaleraldehyde, and ionone exhibited positive correlations with ageing time, while the remaining substances showed no significant changes.

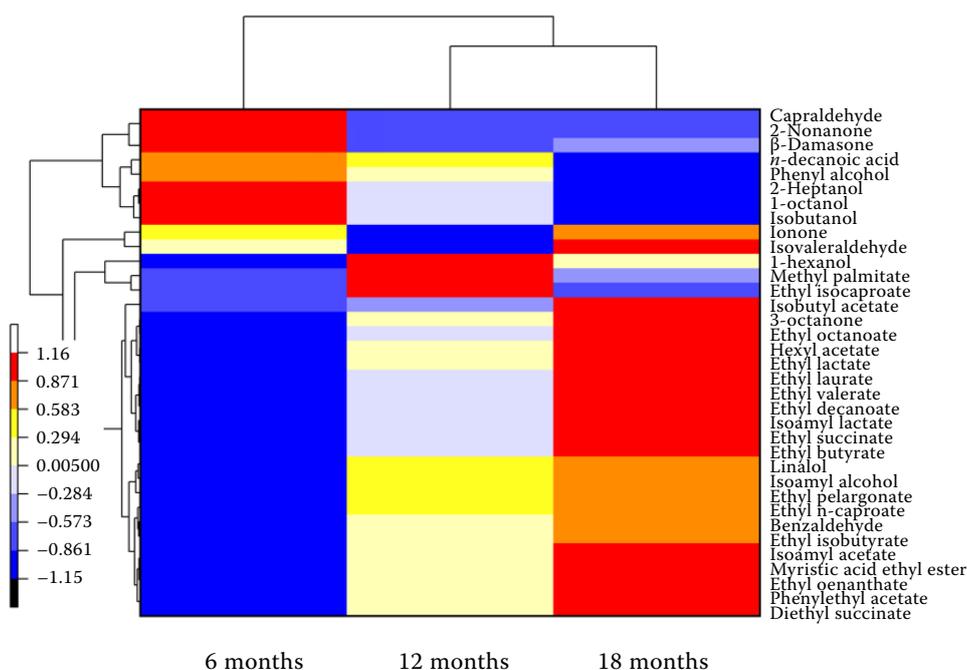


Figure 9. Heat map of variation of aroma substance content of Beibing Hong brandy with ageing time

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Cluster analysis indicated that the aroma profiles of the BBH brandy aged for 12 and 18 months belonged to the same category.

PCA of three kinds of brandy aged for 18 months by PCA. The influence of aroma substances on the evolution of various quantified parameters was analysed using PCA on the dataset encompassing all aroma variables from the three types of brandy aged for 18 months. The results are presented in Figure 10.

The first principal component (PC1) accounted for 55.9% of the variance, while the second principal component accounted for 27.1%.

BBH brandy was positioned in the first quadrant, with its aroma primarily influenced by compounds such as benzaldehyde, 1-octanol, rose ether, β -damascenone, butyl octanoate, 2-heptanol, and isoamyl decanoate. In contrast, SH and ZSY brandies were located in the fourth quadrant, sharing similar dominant aroma contributors, including ethyl myristate, ethyl succinate, ethyl butyrate, ethyl isobutyrate, ethyl phenylethyl acetate, isoamyl lactate, ethyl nonanoate, and ethyl enanthate.

PC1 had a significant impact on the aroma profiles of all three brandies aged for 18 months. The loading values for key aroma compounds on PC1 were: ethyl butyrate (2.943), ethyl succinate (2.761), benzaldehyde (2.696), ethyl myristate (2.448), 1-octanol (1.605), ethyl heptanoate (1.039), ethyl valerate (2.168), 2-heptanol (0.742), ethyl nonanoate (0.715), isoamyl lactate (0.646), ethyl lactate (0.634), phenethyl acetate (0.273), 3-dodecyl acetate (0.197), ethyl isobutyrate (0.124), and 2,2-dimethylsuccinic acid (−0.292).

CONCLUSION

Using GC-MS, HPLC, and other analytical techniques, this study characterised the physicochemical properties, nutrient composition, and aroma compounds of cold mountain grape brandy (cultivars BBH, ZSY and SH) at different ageing periods. Combined with cluster analysis and other statistical methods, the study revealed the dynamic changes during alcohol content, pH, total acidity, major organic acids, and polyphenol compounds

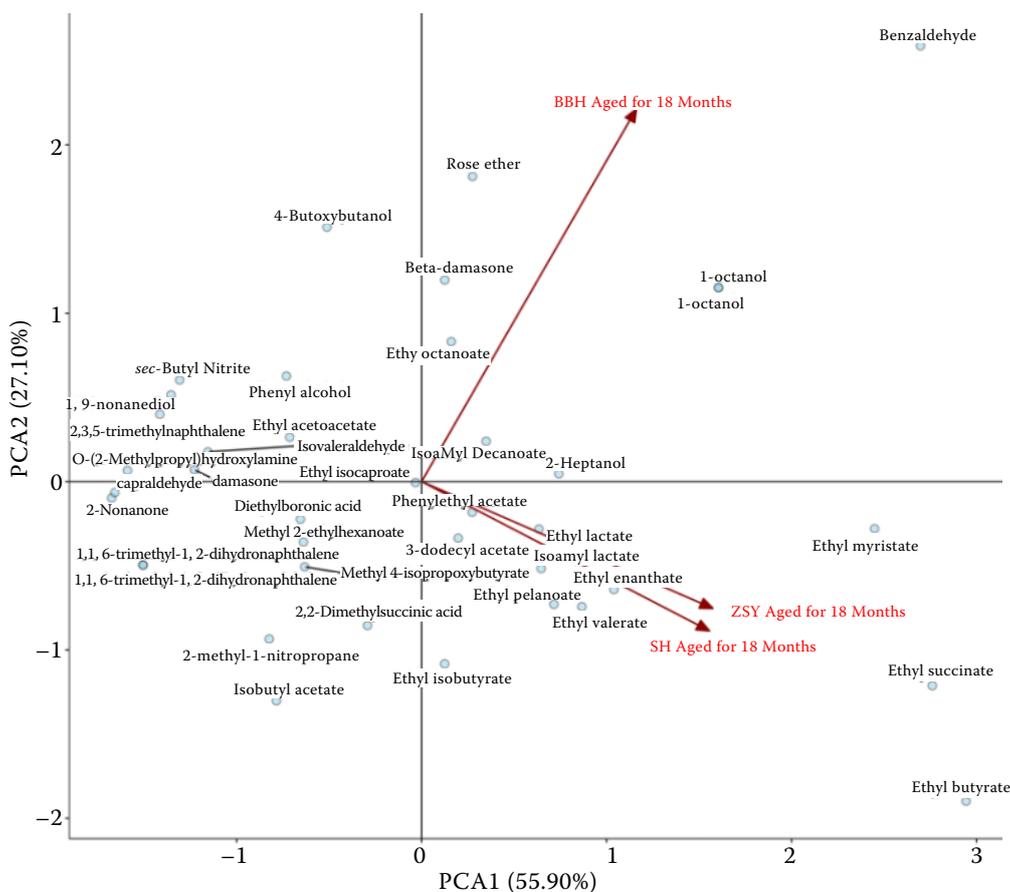


Figure 10. Analysis of the main components of three kinds of 18-month-aged brandies

BBH – Beibing Hong; SH – Shuanghong; ZSY – Zuoshanyi

throughout the ageing process. These findings provide a scientific foundation and technical guidance for the development for the cold mountain grape industry, with the potential to enhance the economic value of mountain grapes in Northeast China.

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