Received: 13/02/2025 Revised: 10/08/2025 Accepted: 06/11/2025 Published: 04/12/2025

Journal of the Institute of Brewing
EST 1895

ORIGINAL ARTICLE

DOI 10.58430/jib.v131i4.82

Impact of the rate of spirit distillation on floral aromas in single malt whisky

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Abstract

Why was the work done: The distillation rate in pot stills shapes the aromatic profile of single malt whisky. Floral notes are prized for their contribution to complexity and elegance, yet the impact of natural reflux in Charentais stills and modulation of flow rate during the second distillation remains poorly understood. This study considers how variations in distillate flow rate affect the floral aroma of new make spirit and assesses the effect of a stepwise distillation process on aromatic complexity. Also, the role of floral-related volatiles (monoterpenes, C13-norisoprenoids, and esters) is evaluated in floral perception together with potential synergistic interactions.
How was the work done: Five distillation profiles were tested, varying heating power during foreshots collection to influence reflux and applying stepwise flow rate changes during heart distillation. Here, 'reflux' refers to passive condensation of vapours in the swan neck and head, induced by temperature gradients and inferred from operational conditions rather than being directly measured. Sensory evaluation assessed the aromatic complexity and intensity of floral, fruity, and solvent notes. Targeted GC–MS quantified floral-related compounds, while sensory reconstitution in a model spirit examined interactions between monoterpenes, C13-norisoprenoids, and esters.
What are the main findings: Reduced natural reflux during foreshots, combined with gradual, stepwise heart distillation, significantly enhanced the floral intensity and aromatic complexity. Optimised conditions increased the levels of linalool, β-citronellol, β-damascenone, and some esters. Sensory reconstitution confirmed the synergism between esters, monoterpenes, and β-damascenone, amplifying floral perception beyond individual contributions.
Why is the work important: The results show that modulation of distillation rate can be strategically applied to refine the aroma balance and improve whisky quality, while highlighting the importance of aroma-aroma interactions in enhancing floral expression.

Keywords

single malt whisky, spirit distillation, floral aromas, monoterpenic compounds, C13-norisoprenoid compounds, esters, perceptual interactions.

Introduction

Like fine wines (Picard et al. 2015), floral notes in single malt whiskies contribute aromatic complexity and are often described as the 'backbone of freshness', adding elegance and subtlety to the sensory profile of high quality whiskies (Jackson 1994; Mac Lean 2003; Paterson 2011; Broom 2014). These delicate aromas, reminiscent of jasmin, rose, or violet, arise from the interaction of esters, terpenes, and norisoprenoids produced during fermentation and distillation (Christoph and Bauer-Christoph 2007). In whisky, terpenes and C13-norisoprenoids are of particular interest given their contribution to floral, citrus, and fruity notes. These compounds originate in the raw material which are released and transformed across the process, from fermentation to distillation and cask aging. While previous work (Picard et al. 2023) has emphasised oak as a source of terpenoids in matured whisky, Miller (2024) reported that a significant proportion of these compounds are already present in the distillate prior to maturation.

Distillation plays a pivotal role in shaping the flavour balance, texture, and complexity of new make spirit (Stewart and Russell 2014). Chemically, the distillation process involves separating alcohol from water and other components of the fermented wort (or ‘wash’) by heating it. Since alcohol has a lower boiling point than water, it vaporises first, carrying with it desirable volatile compounds responsible for the aroma and flavour that contribute to the complexity of the whisky. The vapours condense to liquid, resulting in a more concentrated alcohol, which forms the base of the whisky. Double distillation (or discontinuous distillation) in copper pot stills is a traditional technique used in whisky production, involving two separate and successive distillations (Piggott and Conner 2003; Vriesekoop and Ostrowski 2017). While the first distillation provides a rough separation of alcohol from the wash, the second distillation provides greater precision, enabling a new make spirit that is clean and flavourful (Léauté 1990). Spirit distillation separates the ‘heart’ containing desirable compounds from the ‘foreshots’ (undesirable lighter volatiles) and ‘feints’ (heavier compounds). This method strikes a balance between refining the alcohol content and concentrating the desirable aroma compounds that contribute to the profile of the whisky.

The behaviour of aroma compounds during distillation and their concentration in the different fractions depends on their physical-chemical characteristics, such as boiling point, volatility and solubility (Puentes et al. 2018). Using simulation, several studies have sought to develop optimised strategies during discontinuous distillation for controlling the composition of the spirit (Cantagrel et al. 1989; Awad et al. 2017; Douady et al. 2019; Zanghelini et al. 2024b). Esters are mostly concentrated in foreshots (due to their volatility being higher than ethanol and their low solubility in water), terpenes are more present in the heart, despite their behaviour mainly depending on their respective volatilities and solubilities, which in turn depend on the ethanol content (Matias-Guiu et al. 2016; Esteban-Decloux et al. 2022; Zanghelini et al. 2024a). An increase in the concentration of norisoprenoids is observed during distillation, due to their relatively high boiling point, together with the gradual hydrolysis of multiple norisoprenoid precursors (Mendes-Pinto 2009; Lukić et al. 2011).

The alcoholic strength of the distillate influences the separation of volatile aroma compounds and must be carefully managed in order to retain the desired aroma compounds in new make spirit. During distillation, reflux affects the aromas in whisky by controlling which volatile compounds are retained or re-condensed in the still. This affects the rate of distillation, since a high reflux creates a cycle where vapours are repeatedly condensed and re-evaporated, increasing the interaction with the surfaces inside the still and requiring more time. In Charentais-style stills, natural reflux occurs because of the geometry of the still, particularly in the head and swan neck, where internal temperature gradients cause partial condensation of rising vapours (Léauté 1990; Bougas 2009). In more controlled systems, this reflux cannot be directly adjusted, as the only parameter during distillation that can adjusted is the heating power applied to the boiler.

In this study, the term ‘reflux’ is used to describe an inherent, unmeasured phenomenon, whose intensity was not quantified but inferred from slower distillate flow rates and visible condensation behaviour observed during the process. This type of reflux is known to affect the transfer and retention of aroma compounds in traditional pot stills (Madrera

2003). Yet, despite its presumed impact on aroma development, the specific influence of natural reflux during spirit distillation remains poorly understood, particularly in relation to the expression of floral aroma in new make spirits.

The aim of this study was to assess the impact of the rate of spirit distillation on the sensory profile of new make spirit. By combining sensory and chemical approaches, optimal conditions were sought to enhance floral perception during the spirit distillation. Additionally, to further explore the organoleptic impact of key aroma compounds, specific sensory tests were developed at concentrations found in new make spirit to evaluate their contribution to the floral aroma through perceptive interaction phenomena.

Materials and methods

Industrial process

The process for the production of new make spirit, from mashing to distillation, was performed at the distillery Maison Lineti (Artigues de Lussac, France).

Wash production

Fermentations were performed in thermo regulated 70 hL egg shaped vessel using barley malt wort pitched with 0.67 g/L DistillaMax® yeast (*Saccharomyces cerevisiae*) (Lallemand Biofuels & Distilled Spirits, Montreal, Canada). As recommended by the supplier, the yeast (3 kg) was suspended in 30 L of water for 15 min at 37°C with 10 L of wort added to lower the temperature to approximately 30°C before pitching. Fermentation was performed for 7 days at 28°C. At the end of the fermentation, the final gravity was 0.998 g/mL (DensitoPro Handheld Density Meter, Mettler Toledo) with a pH of 3.5 (Orion Star pH meter, Thermo Fisher Scientific). The ABV (alcohol by volume) of the wash was 8-9% (v/v).

Production of new make spirit

To produce the new make spirit, a double batch distillation was performed at atmospheric pressure in copper pot stills (Satif, Salles-d'Angles), with a direct fired boiler and a maximum capacity of 25 hL. The cooling temperature was 20°C. Wash (23 transferred to the still for the first distillation (wash distillation), with a temperature range of between 80 and 100°C. This process produced low wines and foreshots in the first litres of distillate. This distillation lasted approximately six hours and proceeded until the ABV of distillate was 1% v/v, with the ABV of the low wines at 25% ABV. For the second distillation (the 'spirit distillation'), two wash distillations were required to load the boiler. Accordingly, 23 hL of low wines were transferred to the boiler, with a boiling temperature range of between 80 and 100°C. During the process, three fractions were collected, the foreshots, the heart - comprising the new make spirit - and the feints. All cut points (foreshots/low wines for the wash distillation; foreshots/heart and heart/feints for the spirit distillation) were determined by nosing.

Depending on the heating power, the distillation took between 9 and 11 hours, and was stopped when the ABV of the distillate was 1%. The foreshots from the first and second distillations, as well as the feints from the second distillation, were recycled into the next wash distillation (same wash batch). The heart (new make spirit) was transferred into an intermediate spirit receiving vessel before barrelling. The ethanol content of the new make spirit was 70-73% ABV. Distillations were monitored using the Pro-face interface and the Pro-face Remote HMI software (Schneider Electric). Flow rate, alcoholic strength and cooling temperature, were recorded every 10 seconds by a Coriolis flow meter (Proline Promass 100, Endress + Hauser) attached to the hydrometric port of the still. Gas pressure and boil temperature were recorded every 10 seconds.

All data were treated using Excel software (16.88 version).

Spirit distillations - experimental design

Four spirit distillation strategies (SD-A, SD-B, SD-C, and SD-D) were designed to vary the heating power applied to the boiler, which in turn modified gas pressure and affected the distillate flow rate. These adjustments were used to change the presumed level of natural reflux inside the Charentais still, although reflux was not directly measured. The flow rate combinations during the distillation of the heads and heart are presented in Table 1. This made possible the adjustment of heating power that was

fixed by the gas pressure. The feints fraction was distilled under identical conditions in all regimes and recycled into the next first distillation (wash run). As this did not vary between treatments, it was not included in the comparative analysis. A further profile (SD-E) was performed combining several stages of the flow rate during the heart distillation (Table 2).

To minimise the influence of any fluctuations in ambient temperature on the management of the distillation process, experiments were performed over a two week period in March 2023.

Sampling of wash, low wines and new make spirit

Wash samples (200 mL) were collected at the end of fermentation, stored in brown bottles at 4°C and analysed within 24 hours.

Low wines and new make spirit samples were collected at the end of wash and spirit distillations, respectively. New make spirit was sampled prior to dilution and cask filling. Samples of low wines and new make spirits (100 mL) were stored at room temperature in brown bottles.

Chemicals and reagents

Absolute ethanol (purity > 99.9%) and anhydrous sodium sulphate were supplied by Merck (Fontenay-sous-Bois, France). Dichloromethane (purity > 99.9%) was provided by VWR Chemicals (Fontenay-sous-bois). Microfiltered water was obtained using a Milli-Q purification water system (resistivity = 18.2 MΩ cm, Millipore, Saint-Quentin-en-Yvelines, France). Compounds were obtained from commercial sources as follows: ethyl propanoate (99%) from Alfa Aesar (Kandel, Germany); propyl acetate (99%), 2-methylpropyl acetate (99%), ethyl butanoate (99%), ethyl hexanoate (99%), hexyl acetate (99%), ethyl octanoate (98%), ethyl 2-methylpropanoate (99%), ethyl 2-methylbutanoate (99%), ethyl 3-methylbutanoate (98%), 2-methylbutyl acetate (99%), ethyl 2-hydroxy-3-methylbutanoate (99%), ethyl 6-hydroxyhexanoate (97%), 2-Phenethyl acetate (99%), ethyl decanoate (99%), ethyl dodecanoate (≥98.0%), terpinolene (95%), β-citronellol (95%), geraniol (98%), and β-damascenone (99%) from Sigma-Aldrich (Saint-Quentin Fallavier, France); 3-methylbutyl acetate (isoamyl acetate) (98%), ethyl 2-hydroxy-4-methylpentanoate (98%), linalool (96%), and α-terpineol (95%) from TCI Europe (Zwijndrecht, Belgium); ethyl butyrate - 4,4,4-d_3, ethyl hexanoate-d_{11}, ethyl octanoate-d_{15}, and linalool-d_3, β-ionone-d_3 from Cluzeau (Sainte Foy la Grande, France). Aroma compounds were prepared in absolute ethanol.

Table 1.

Operating parameters for spirit distillation (foreshots and heart).

The 'reflux pattern' refers to the presumed levels of natural reflux, inferred from gas pressure and distillate flow rate, but not directly measured.

	SD-A	SD-B	SD-C	SD-D
Total Distillation time (h)	10.58	9.50	11.13	9.10
Foreshots				
Distillation time (h)	0.50	0.46	0.16	0.20
ABV in the fraction (%)	77.2	76.4	77.3	77.7
Volume (L)	17.6	16.3	17.5	16.3
Gas pressure (mbar)	980	980	1961	1961
Flow rate (L/h)	60	60	100	100
Reflux pattern	high	high	low	low
Heart				
Distillation time (h)	4.43	3.33	5.28	0.70
ABV in the fraction (%)	72.9	72.2	73.1	72.4
Volume (hL)	5.1	5.0	6.2	5.5
Gas pressure (mbar)	6933	9806	6933	9806
Flow rate (L/h)	120	160	120	160
Reflux pattern	high	low	high	low

Table 2.

Parameters of spirit distillation SD-E (foreshots and heart) using multi-stage heart distillation.

The 'reflux pattern' refers to the presumed levels of natural reflux, inferred from gas pressure and distillate flow rate, but not directly measured.

	SD-E	
	Total Distillation time (h)	10.26
	Foreshots distillation	
	Distillation time (h)	0.50
	Total ABV in the fraction (%)	77.7
	Volume (L)	17.0
	Gas pressure (L/h)	980
	Flow rate (L/h)	60
	Reflux pattern	high
	Heart distillation	
	Distillation time (h)	3.93
	Total ABV in the fraction (%)	72.5
	Volume (hL)	5.4
Stage 1	Ramp time (min)	7
	Ramp gas pressure (mbar/min)	1260
	Ramp Flow rate (L/h/min)	8.6
	Stage time (min)	65
	Stage gas pressure (mbar)	9806
	Stage Flow rate (L/h)	160
	Reflux pattern	low
Stage 2	Ramp time (min)	1
	Ramp gas pressure (mbar/min)	1471
	Ramp Flow rate (L/h/min)	20
	Stage time (min)	65
	Stage gas pressure (mbar)	8334
	Stage Flow rate (L/h)	140
	Reflux pattern	medium
Stage 3	Ramp time (min)	1
	Ramp gas pressure (mbar/min)	1176
	Ramp Flow rate (L/h/min)	20
	Stage time (min)	65
	Stage gas pressure (mbar)	6865
	Stage Flow rate (L/h)	120
	Reflux pattern	high

All the compounds used in this study were olfactively pure, as confirmed by gas chromatography analysis with olfactometric detection (GC-O) of reference compounds. Flame ionisation detector analysis confirmed very high purity (> 95%).

Sensory analyses

General conditions

Samples were assessed at a controlled room temperature (20°C) in individual booths (ISO 8589:2010, 2010). Covered ISO glasses (ISO 3591:1977, 1977) were used, containing 20 mL of diluted sample at 40% ABV and coded with 3-digit random numbers. In this work, all sensory analyses were performed using orthonasal perception. All panellists were informed of the context of the study before starting data collection.

Sensory panels

Panel 1 consisted of five experts - two female and three male - in wine and spirit tasting, one from Institute of Vine and Wine Science (ISVV) and four from Maison Lineti. This Panel generated olfactory descriptors for all samples.

Panel 2 consisted of 15 individuals, 10 female and 5 male from the research laboratory staff at ISVV. Using the method of Pelonnier-Magimel et al (2020), they were trained to recognise specific olfactory descriptors of spirits ('aromatic complexity', 'aromatic intensity', 'white flowers', 'fresh fruits (apple, pear)', 'fresh rose', and 'solvent').

Descriptive testing methods

The aromatic properties of the samples were established by Panel 1 via orthonasal perception, using a generation of descriptors methodology. During the panel discussion phase, the panellists produced a maximum of five descriptors for sample to evaluate their respective aromatic qualities (NF ISO 11035: 1995).

Sensory profiles were performed by Panel 2 using the six descriptors: 'aromatic complexity', 'aromatic intensity', 'white flowers', 'fresh fruits (apple, pear)', 'fresh rose', and 'solvent' (NF ISO 13299: 2016). The objective of this comparative sensory profile was to assess the qualitative differences between samples derived from different spirit distillation parameters. Participants evaluated samples using a 0 to 100 mm unstructured scale, where '0' indicated no odour was perceived and '100' indicated an odour of high intensity.

To evaluate the specific impact of aroma compounds, aromatic reconstitutions based on the concentration found in new make spirit SD-E, were made using new make spirit SD-A as the matrix. Three glasses were presented to the panellists for sensory evaluation. The first glass contained the new make spirit SD-A diluted to 40% ABV, with the second and third glasses contained the same sample, but with the addition of either (i) monoterpenic and C13-norisoprenoid compounds, or (ii) esters, adjusted to the concentration in the new make spirit SD-E (Figure 1).

Each panellist evaluated the samples using the six descriptors (aromatic complexity, aromatic intensity, white flowers, fresh fruits, and fresh rose) using a 0 to 100mm unstructured scale, where 0 indicated no odour and 100 indicated an odour of high intensity.

Quantitative chemical analyses

Sample preparation and extraction of aroma compounds

With the exception of wash samples at 7% ABV, samples were diluted to 12% ABV and prepared as described by Garbay et al (2023). Samples (50mL) were spiked with different internal standards ('IS'): 374 µg/L of ethyl butyrate - 4,4,4-d_3 (IS for the non-polar esters ethyl propanoate and 2-methylpropyl acetate), 171 µg/L of ethyl hexanoate-d_{11} (IS for the non-polar esters ethyl hexanoate and hexyl acetate), 263 µg/L of ethyl octanoate-d_{15} (IS for ethyl octanoate), 300µg/L of ethyl 2-hydroxy-2-methylpropanoate (IS for substituted and hydroxylated polar esters and ethyl decanoate and ethyl dodecanoate), 64 µg/L of linalool-d_3 for monoterpenic compounds, and 40 µg/L of β-ionone-d3 for β-damascenone.

The sample was then serially extracted using dichloromethane (4 mL, 4 mL, and 2 mL) at room

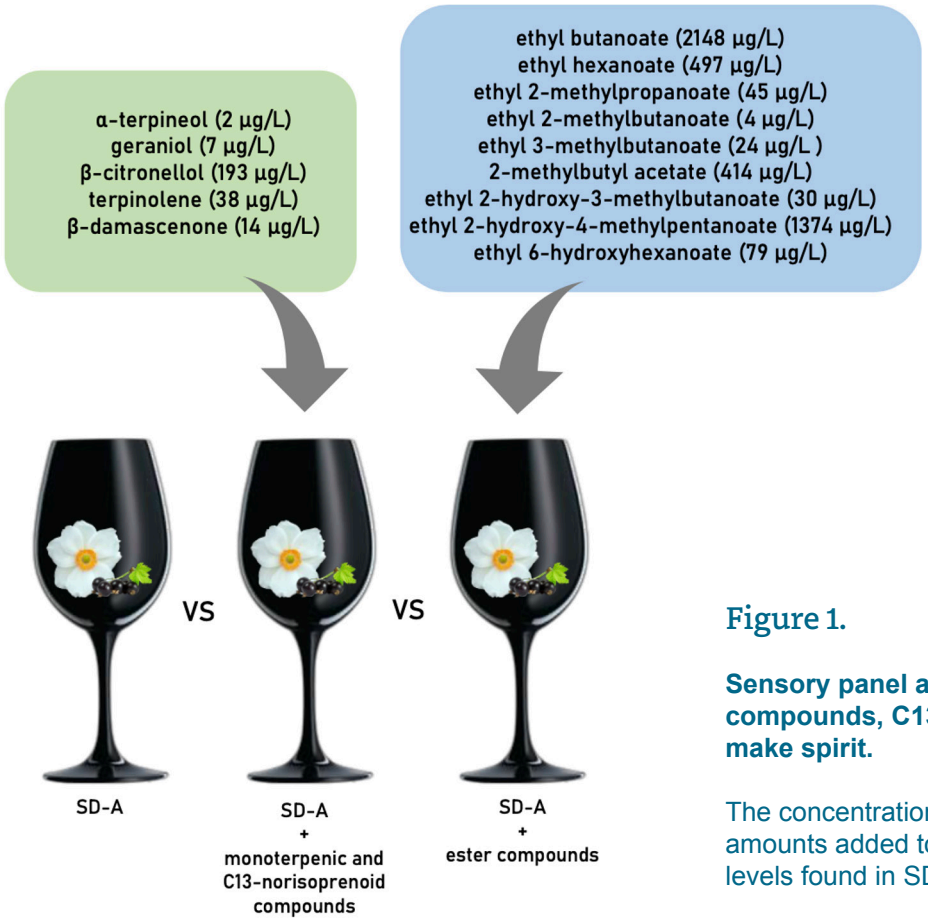


Figure 1.

Sensory panel analysis of monoterpenic compounds, C13-norisoprenoids, and esters in new make spirit.

The concentration in brackets correspond to the amounts added to the SD-A sample to achieve the levels found in SD-E.

temperature (around 25°C), with stirring (700 rpm), for 10 min. The organic phase was separated from the aqueous phase, collected, blended, dried over sodium sulphate and concentrated under a nitrogen flow (100 mL/min) to a final volume of 250µL.

Gas Chromatography–Mass Spectrometry analyses (GC-MS)

Targeted GC-MS analyses was as described by Garbay et al (2023) using an HP 5890 GC system coupled to an HP 5972 quadrupole mass spectrometer (Hewlett-Packard) equipped with a Gerstel MPS2 autosampler. Aroma extract (2 µL) from diluted samples were injected in split-splitless mode (injector temperature: 200°C, interface temperature: 240°C, split flow: 60 mL/min, split ratio: 30:1, total flow: 60 mL/min, saver flow: 20 mL/min, saver time: 2 min). The capillary column was a BP21 (SGE 054469, Ringwood, Australia), 50 m × 0.320 mm i.d., film thickness: 0.25 µm. The oven temperature was programmed as follows: 40°C for 1 min, then increased to 100°C at a rate of 1°C/min and to a final isotherm of 180°C at 3°C/min, which was maintained at 180°C for 3 min. The run lasted 90 minutes. The carrier gas was helium N55 (Air Liquide, France) with a column head pressure of 9.48 psi, and an initial flow of 2 mL/min. The MSD transfer line heater was set to 280°C. The mass spectrometer was operated with electron ionisation at 70 eV and in selected-ion monitoring (SIM) mode (dwell = 20 msec). The ions were selected according to the previous study by Garbay et al (2023).

Statistical analyses

Statistical analyses was performed using XLSTAT software (Addinsoft, Paris, France). The STATIS method was used to analyse and compare the sensory profiles of the distillates across the descriptors. This is a multivariate statistical approach designed to handle datasets structured in blocks, allowing the integration of multiple individual evaluations into a consensus analysis (Lavit et al. 1994). Each block corresponded to a sensory evaluation matrix provided by an individual assessor, with rows representing the samples and columns representing the studied descriptors. A partial projection plot was produced to evaluate the contribution and alignment of each individual

dataset with the compromise matrix. In this plot, each sample is represented by several points, corresponding to its positioning as assessed by each panellist. The proximity of these points indicates the degree of agreement among assessors. The global positioning of the samples in the consensus space highlights the overall relationships between the distillate samples and their sensory descriptors, while the dispersion of points in the partial projection plot provides insight into the consistency of the panellists.

The STATIS method also enabled the evaluation of the consensus between panellists by calculating Redundancy Vector (RV) coefficient. This is an key tool for evaluating the reliability and consistency of individual data compared to the group in multiblock studies. The RV coefficient calculated for each assessor indicates (i) their level of agreement with the group consensus, and (ii) their contribution to the collective sample sensory representation. A high RV coefficient means a strong alignment between the evaluation of an panellist and the group consensus, ensuring robust and reliable interpretation of the sensory data.

Discrimination between the sensory data in terms of intensity for each descriptor were performed using non-parametric tests. The Kruskal-Wallis test, followed by Dunn's post hoc test with a significance level of $\alpha = 5\%$ ($p < 0.05$), was used for comparisons involving more than three samples. For comparisons between two samples, the Wilcoxon signed-rank test with a significance level of $\alpha = 5\%$ ($p < 0.05$) was applied.

A heatmap was generated to visualise the relative concentrations of volatile compounds and their distribution among the samples. The dataset consisted of quantitative measurements of the 24 volatile compounds. A chromatic scale was used in which red and blue colours represent either high and low concentrations of each compound. Hierarchical clustering was performed using Ward's method, based on Euclidean distances, to group both samples and compounds according to their similarity in chemical profiles. The Pearson correlation test was used to evaluate the relationship between the sensory data and the concentration of the compounds in the spirit.

Results and discussion

Validation of the reproducibility of the industrial process

In a study on pear spirits, García-Llobodanin et al (2011) found that distillation using traditional alembic stills were more reproducible than batch distillation columns. However, even under similar conditions, inconsistencies were observed in the composition of final spirit regardless of the distillation method. In the absence of chemical data on the 'wash' and low wines, it was not possible to confirm that the lack of reproducibility is exclusively due to the distillation process.

To address this concern, the repeatability of the production process across fermentation, first distillation, and second distillation was explored. Three identical fermentation batches were produced and distilled using the same method. For the spirit distillation, the SD-A profile was chosen (Table 1). The concentration of compounds in the triplicate washes, corresponding low wines, and new make spirits (Table 3) were measured. Reproducibility was assessed by the relative standard deviation (RSD) of each compound in each matrix. For the different chemical families (ethyl esters, ester acetates, hydroxy ethyl esters, monoterpenic compounds, and C13-norisoprenoids), the results show that the reproducibility was good, since the relative standard deviations were below 15%. Four compounds (ethyl butanoate, 2-methylpropyl acetate, hexyl acetate, and phenethyl acetate) had an RSD higher than 15% in the washes, compared to one in the low wines (ethyl octanoate), and two in the new make spirits (ethyl 6-hydroxyhexanoate and linalool).

In the context of this study, this step validated the reproducibility of the different production stages, supporting the hypothesis that the sensory and chemical differences observed in the spirit distillation were solely related to this process, and not from variations in the wash and low wines produced under similar conditions.

Table 3.

Compounds (µg/L) in the fermented washes, corresponding low wines and new make spirits (n=3)

	Wash					Low wines					New make spirit				
	Wash 1	Wash 2	Wash 3	*Mean*	*RSD (%)*	LW 1	LW 2	LW 3	*Mean*	*RSD (%)*	NMS 1	NMS 2	NMS 3	*Mean*	*RSD (%)*
Total ethyl esters	**1608.7**	**1307.5**	**1280.8**	**1399**	**11%**	**8447.9**	**9371.3**	**10162.4**	**9327.2**	**8%**	**27716.6**	**25430.3**	**24644.7**	**25930.5**	**5%**
Ethyl propanoate	102.3	83.7	72.7	86.2	14%	123	114.5	138.4	125.3	8%	348.6	350.8	345.3	348.2	1%
Ethyl 2-methylpropanoate	30.1	24.4	25.8	26.8	9%	151.3	170.7	148.9	157	6%	515.3	537.2	478.2	510.3	5%
Ethyl butanoate	147.5	148.5	102.2	132.8	16%	644.6	600.3	692.9	645.9	6%	2086.1	1929.9	2108	2041.3	4%
Ethyl 2-methylbutanoate	2.2	1.7	2	2	10%	10.4	10.7	9.5	10.2	5%	43.8	42.9	42.4	43.1	1%
Ethyl 3-methylbutanoate	2.9	3.3	3.2	3.2	5%	22	24.4	20.2	22.2	8%	98.4	89.4	67.2	85	15%
Ethyl hexanoate	781.6	628.4	619.2	676.4	11%	1673.6	1772.9	2150.5	1865.7	11%	6644	6044.3	6489.5	6392.6	4%
Ethyl octanoate	308.3	218.4	245.5	257.4	15%	704.9	807	1212.7	908.2	24%	5859.4	4551.1	5469	5293.2	10%
Ethyl decanoate	177.1	154	157.6	162.9	6%	3008.2	3409	3502.6	3306.6	6%	8682.3	8453.9	6545.2	7893.8	12%
Ethyl dodecanoate	56.6	45.1	52.6	51.4	9%	2109.8	2461.8	2286.8	2286.1	6%	3438.6	3430.8	3100	3323.1	5%
Esters acetates	**6214.5**	**5975.6**	**5658.1**	**5949.4**	**4%**	**13870.4**	**12650.9**	**13702.5**	**13407.9**	**4%**	**45752**	**41478.2**	**43315.8**	**43515.3**	**4%**
Propyl acetate	1.4	1.3	1.2	1.3	7%	6	5	4.5	5.2	12%	31.9	29.8	23	28.2	13%
2-methylpropyl acetate	297.1	292	205.3	264.8	16%	703.1	679.1	797.9	726.7	7%	1961	2176.3	2333	2156.7	7%
2-methylbutyl acetate	215.9	172.1	238.3	208.8	13%	2887	2495	2276.7	2552.9	10%	11775.1	9842.5	7877.4	9831.7	16%
Isoamyl acetate	5182.9	5152	4750.9	5028.6	4%	9124.3	8290.7	9581.9	8998.9	6%	28564.6	26325.6	30176.3	28355.5	6%
Hexyl acetate	31	29.3	17.4	25.9	23%	80.4	65.3	92.8	79.5	14%	306.3	275.8	288.5	290.2	4%
Phenethyl acetate	486.1	328.9	444.9	420	16%	1069.7	1115.7	948.6	1044.7	7%	3113.1	2828.4	2617.6	2853	7%
Hydroxy ethyl esters	**60.8**	**55.7**	**63.1**	**59.8**	**5%**	**267**	**262.1**	**239**	**256.1**	**4%**	**1 120.8**	**900**	**886.5**	**969.1**	**7%**
Ethyl 2-hydroxy-3-methylbutanoate	nd	nd	nd	nd	-	10.3	13.8	12	12	12%	21.9	22.9	20.5	21.8	5%
Ethyl 2-hydroxy-4-methylpentanoate	10.8	15.4	13.8	13.3	14%	166.8	156.3	149.1	157.4	5%	223.7	220.9	263.6	236.1	8%
Ethyl 6-hydroxyhexanoate	50	40.3	49.3	46.5	9%	89.9	92.1	77.9	86.6	7%	875.2	656.2	602.4	711.3	17%
Monoterpenic compounds	**80**	**76**	**83.4**	**79.8**	**4%**	**160.5**	**162.8**	**151.9**	**158.4**	**3%**	**888.5**	**922.5**	**797**	**869.3**	**6%**
Terpinolene	nd	nd	nd	nd	-	nd	nd	nd	nd	-	92.6	80	72.7	81.8	10%
Linalool	nd	nd	nd	nd	-	37.9	35.9	27.6	33.8	13%	195.4	164.7	130.4	163.5	16%
α-Terpineol	nd	nd	nd	nd	-	6.1	7.3	5.7	6.4	11%	22.7	19.9	16.5	19.7	13%
β-Citronellol	17.9	16.3	18.9	17.7	6%	68	60.6	63.9	64.2	5%	494	586.9	502.2	527.7	8%
Geraniol	62.1	59.7	64.4	62.1	3%	48.4	58.9	54.6	54	8%	83.8	71	75.2	76.7	7%
C13-norisoprenoids	**0.8**	**0.9**	**1**	**0.9**	**6%**	**26.9**	**27**	**25.7**	**26.5**	**2%**	**114.6**	**119.6**	**110.2**	**114.8**	**3%**
β-damascenone	0.8	0.9	1	0.9	6%	26.9	27	25.7	26.5	2%	114.6	119.6	110.2	114.8	3%

Impact of the spirit distillation rate on new make spirit: identification of sensory characteristics by the expert panel

The description of the four spirit samples highlighted six dominant sensory descriptors: 'aromatic complexity', 'aromatic intensity', 'white flowers', 'fresh fruits (apple, pear)', 'fresh rose' and 'solvent'. These descriptors were selected for further development. Notable differences were observed (Table 4) between samples distilled with low or high reflux during the foreshots distillation (Table 1). Samples SD-C and SD-D, with low reflux, received negative feedback due to the aggressive aroma, reduced elegance, and lack of complexity. In particular, SD-D was described as lacking expression, being simplistic, and having slight 'wet mop' off-notes. By contrast, samples SD-A and SD-B, produced with high reflux during the foreshots distillation, received a more positive evaluation. These samples were described as complex, fruity, and floral. The SD-A sample was particularly liked for its pronounced floral and grain characteristics and was the favourite overall of the panel.

Table 4.

Sensory characteristics of the spirit distillation samples (SD-A, SD-B, SD-C, and SD-D)

	Foreshots		Heart		Tasting Comments
	Reflux pattern	*Flow rate (L/h)*	*Reflux pattern*	*Flow rate (L/h)*	
SD-A	High	60	High	120	• Dominant aroma of cereal grains and creamy notes. • Fruity notes, including pear and hints of blackcurrant. • Floral characteristics of white flowers and hints of apple compote. • The overall profile is structured, complex, and precise
SD-B	High	60	Low	160	• Subtle fruity notes - nuances of stone fruits and caramelised apple. • Delicate floral undertones, though less expressive overall. • The profile is softer and less intense.
SD-C	Low	100	High	120	• Sharp mineral notes reminiscent of flint, with aromas of boiled malt. • Hints of fresh cut grass. • Structured but less refined profile and lacks elegance.
SD-D	Low	100	Low	160	• Low aromatic intensity with fresh fruit notes, such as apricot. • The aroma is aggressive and lacks complexity. • Undesirable off notes ('wet mop' aroma).

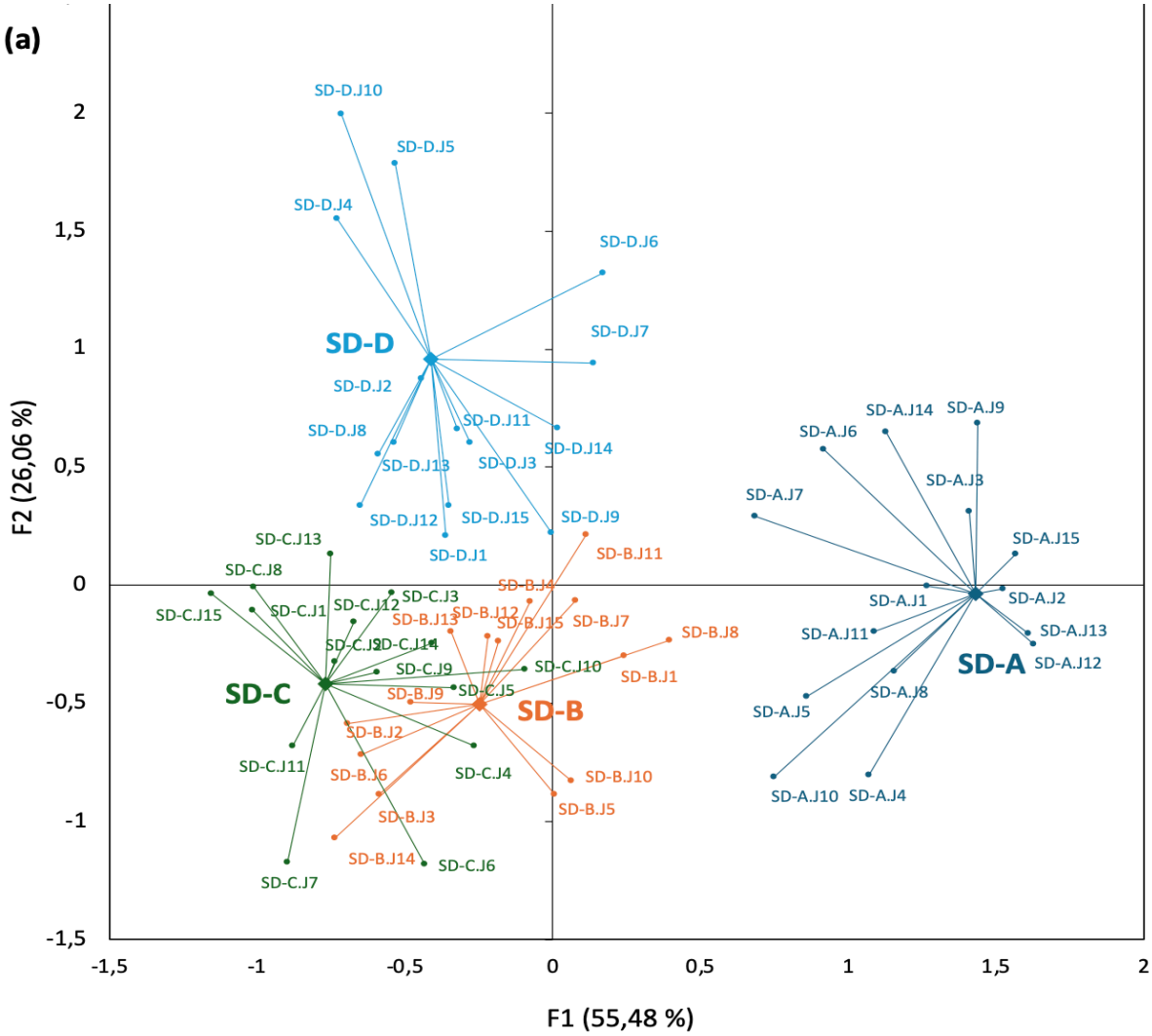
Overview, and consensus of the sensory panel

The partial projection plot from the STATIS analysis (Figure 2a) visualises the variability of evaluation by individual and the consensus among panellists (J1 to J15) for the sensory analysis of the spirit samples (SD-A, SD-B, SD-C, and SD-D). The axes (F1 and F2) represent the main factors derived from the STATIS analysis, explaining 55.48% and 26.06% of the variance for a total of 81.55%. Each sample was represented by a central point (the consensus position) surrounded by individual evaluations (eg SD-A.J1, SD-A.J2, etc.), corresponding to the assessments of each panellist. The lines connecting the central point to individual points represent the variability in the perception of each assessor. The positions of the central points in the factor space show the general relationships between the samples. The SD-A sample was clearly distinct from the other samples, located on the positive side of F1 axis. SD-B and SD-C samples were closer to each other, indicating a similar sensory profile, while SD-D sample occupied a unique position with less overlap with the other samples.

With regard to the consensus between panellists, samples with tightly clustered points around their centre (SD-A and SD-D) exhibited a high level of consensus, suggesting these samples have clear and distinct sensory attributes. By contrast, samples with more dispersed points (SD-B) indicated greater variability in the individual assessment, with more complex or ambiguous sensory characteristics. Interestingly, samples SD-A and SD-D were positioned on opposite sides of the sensory space,

Figure 2a.

Partial projection plot of samples - individual evaluation and consensus among panellists



highlighting clear sensory differences. These differences may be attributed to variations in the distillation process, with SD-A produced under high reflux conditions and SD-D under low reflux conditions.

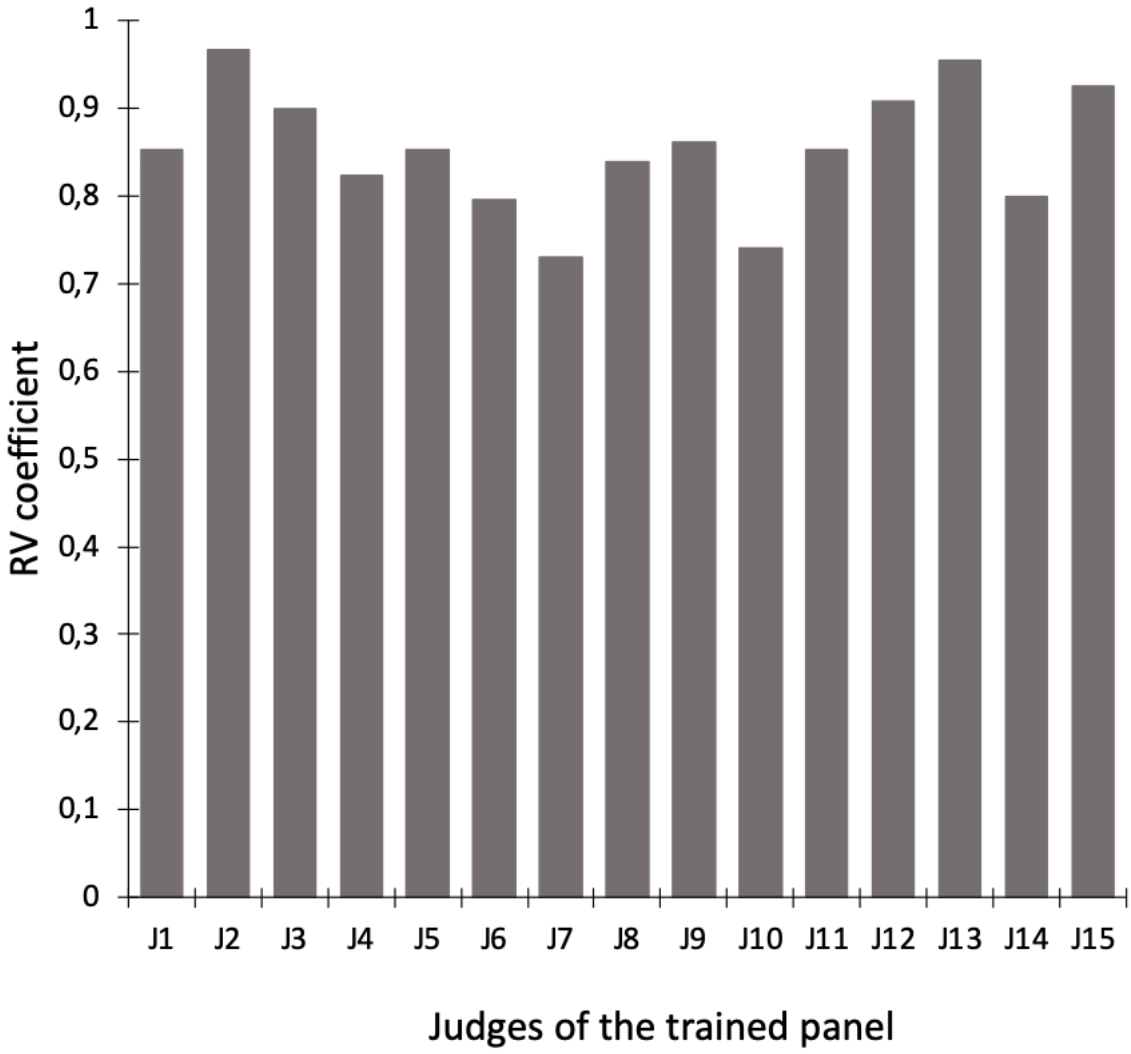
Figure 2b presents the RV coefficients between the configuration of each assessor (J1 to J15) and the consensus obtained through the STATIS method. The RV coefficient ranged from 0 to 1, with values closer to 1 indicating a stronger alignment between the evaluation of an individual assessor and the group consensus. Interestingly, most assessors exhibited RV coefficients above 0.8, suggesting that the panellists shared a similar perception of the sensory characteristics of the samples. Specifically, panellists J2, J12, J13, and J15 showed RV coefficients close to 1, showing their evaluations to be highly aligned with the consensus and strongly contribute to the compromise configuration. Other panellists - J7 and J10 - have lower RV coefficients (around 0.7), indicating some divergence from the consensus. This may reflect differences in individual perception or interpretation of the sensory descriptors. Here, additional training or clarification on the sensory descriptors to improve alignment with the group could be required.

Sensory discrimination

The sensory evaluation of the samples revealed marked differences of some of the descriptors selected by the expert panel, with findings replicated consistently by the 15 panellists of the trained panel. Descriptors, such as aromatic complexity and notes of white flowers, showed significant variation

Figure 2b.

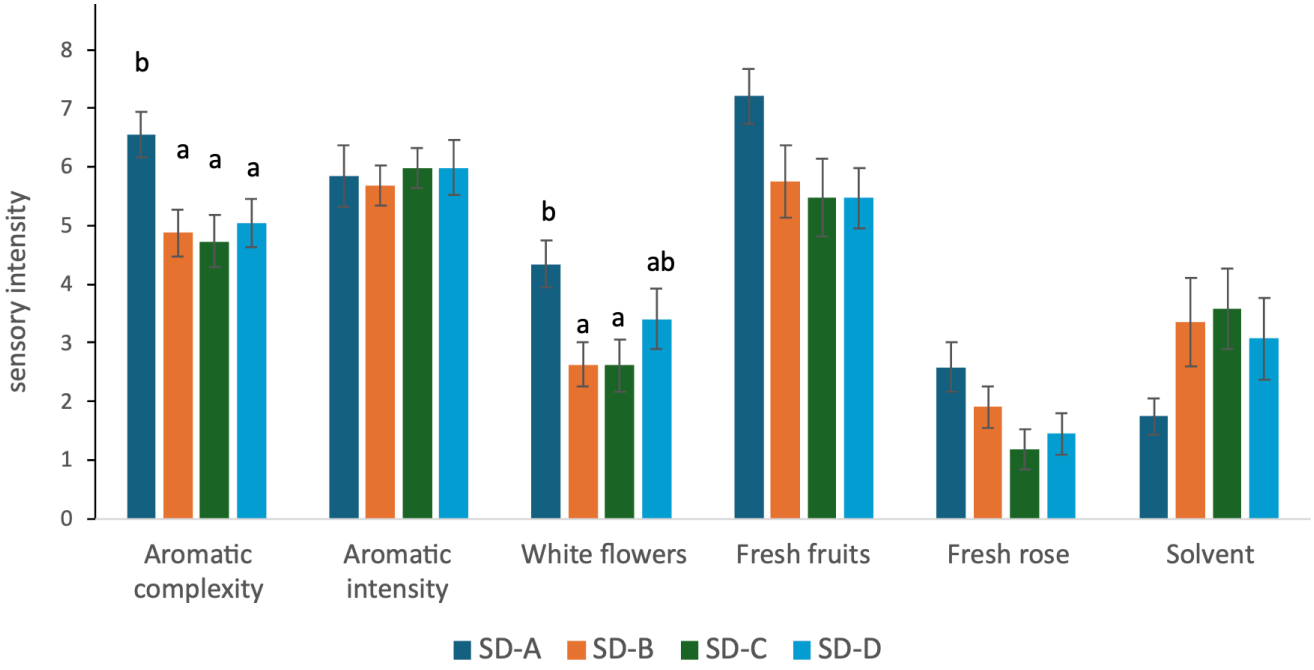
RV coefficients between individual configuration and consensus for panellists in the trained panel.



among the samples. Clear patterns were seen in the intensity of fresh fruit (apple, pear), fresh rose, and solvent-like notes. Sample SD-A stood out, exhibiting greater aromatic complexity and floral notes. The intensity of fresh rose notes was higher for the SD-A sample, whereas SD-B, SD-C, and SD-D displayed more pronounced solvent-like characteristics. This suggests that SD-A offered a complex aromatic profile, with minimal solvent-like attributes (Figure 3). These results align closely with the descriptive analyses provided by the expert panel, confirming the consistency and reliability of the sensory evaluation. Moreover, the dual interpretation of sensory results from both panels highlighted that low reflux during foreshots distillation should be avoided, as it compromises the notes of the heart fraction. These findings are consistent with other studies with rectification packed columns, where a high reflux rate at the start of distillation enhanced the floral expression and reduced undesirable aromas in the heart fraction (Rodríguez-Bencomo et al. 2016; Matias-Guiu et al. 2016; Matias-Guiu et al. 2018). Although 'reflux' is used here to describe condensation phenomena inside the Charentais still, the natural reflux resulting from temperature gradients and flow conditions, is not a directly controlled or measured variable. Its effect is likely intertwined with residence time and heating rate and may not be interpreted independently from these operational factors.

Figure 3.

Mean intensities of aromatic descriptors. Different letters indicate statistically significant differences among samples (p-value ≤ 0.05, significance levels determined by Kruskal-Wallis statistical test).



Multi-stage spirit distillation for increasing the floral character in new make spirit

The results showed that high reflux conditions during the distillation of the heart fraction enhance the quality of the spirit, imparting desirable floral aromas and overall complexity. These results pave the way for advancing the approach used in the production of the SD-A spirit (Table 1) sample. To achieve this, a specific profile, SD-E (Table 2) was developed by maintaining a high reflux during foreshots distillation and incorporating multiple flow rate adjustments during the heart distillation phase. With the incorporation of multiple flow rate adjustments during the heart distillation phase, the sensory panel reported superior sensory comments for SD-E compared to SD-A (Table 5) confirming that these multiple flow rate adjustments increase aromatic complexity.

The sensory characteristics of SD-A and SD-E, across six attributes - aromatic complexity, aromatic intensity, white flowers, fresh fruits (apple, pear), fresh rose, and solvent - were evaluated by Panel 2 (Figure 4). The intensity of aromatic complexity, white flowers, and fresh rose notes showed a significant difference, with SD-E perceived as having higher scores for the intensity of these attributes. These results validated that the multiple flow rate gradient (120, 140 and 160L/h) increased aromatic complexity, white flower notes, and fresh rose notes compared to the sinle and lower slow flow rate (120L/h) of SD-A.

Chemical profiling of distillates

Heatmaps are a powerful tool for exploring the chemical complexity of alcoholic beverages, having been used to differentiate wines according to the volatile profile influenced by grape origin, yeast diversity, and fermentation conditions (Binati et al. 2020; Luzzini et al. 2021; Ferremi et al. 2024; Plantevin et al. 2024). In the context of spirits, heatmaps have been used in the identification of key volatiles and aroma markers, such as esters and aldehydes, which define the sensory profiles and chemical diversity of whiskies, brandies, and baijiu (Liu et al. 2023; Huang et al. 2024). By visually representing the relative concentrations of chemical compounds, heatmaps enable rapid identification of patterns, differences, and similarities among samples.

Table 5.

Sensory characteristics of the spirit sample SD-E

	Foreshots		Heart		Tasting Comments
	Reflux pattern	*Flow rate (L/h)*	*Reflux pattern*	*Flow rate (L/h)*	
SD-E	High	60	3 successive stages	160/140/120	• Complex and balanced profile, with malt, fruit, and floral notes. • Fresh pear fruit characteristics. • The floral element is subtle, contributing to the overall fineness. • A slight nutty note, particularly hazelnuts, enhances the complexity.

Figure 4.

Mean intensities of aromatic descriptors in SD-A and SD-E samples. Significance levels at 5% (*) and 1% (**) determined by Wilcoxon signed-ranked statistical nonparametric test.

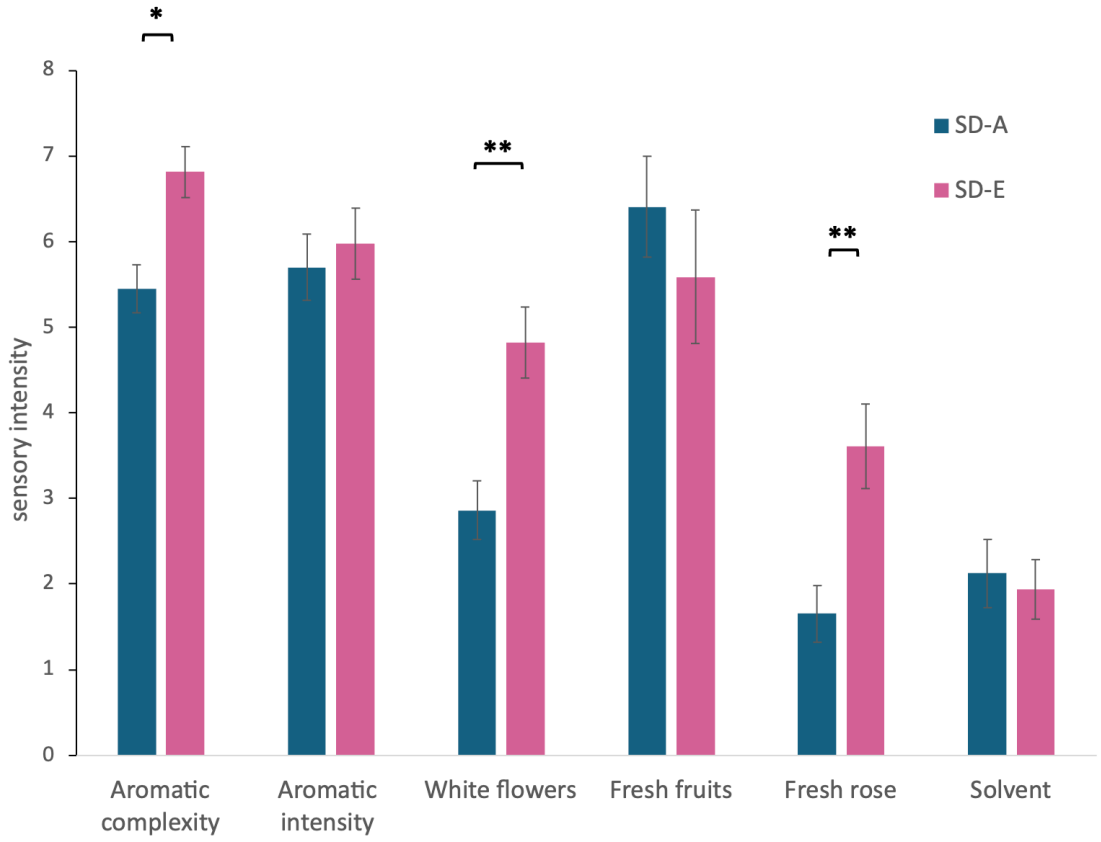
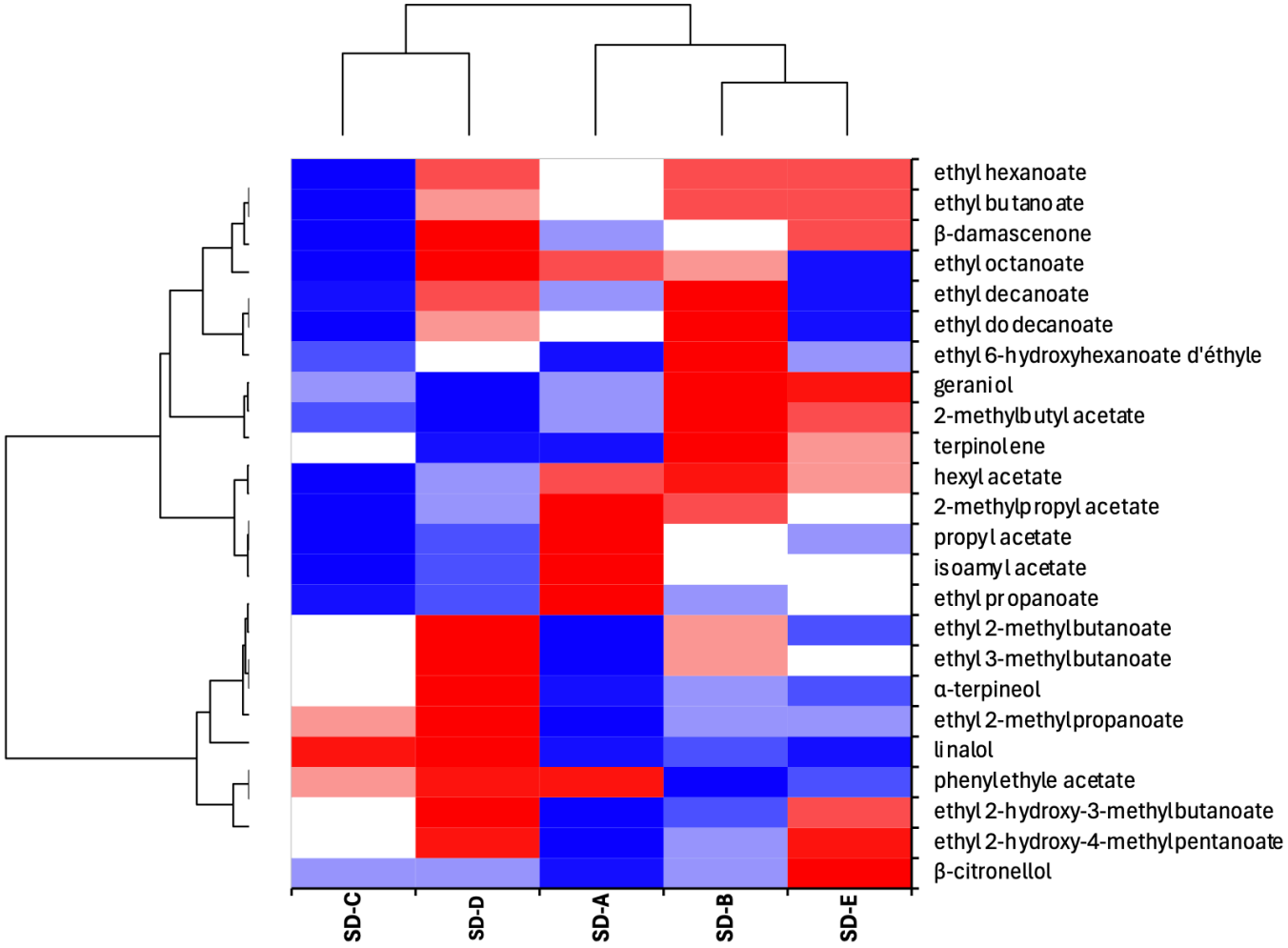


Figure 5 shows the heatmap of volatile patterns of the five distillate samples (SD-A, SD-B, SD-C, SD-D, and SD-E) corresponding to the chemical data reported in Supplementary Information Table S1. The chromatic scale of the heatmap reflects the relative abundance of each volatile compound, ranging from dark blue (minimum concentration) to dark red (maximum concentration). Hierarchical clustering revealed two main groups: SD-C and SD-D, which shared similar chemical profiles with generally low concentrations for dominant compounds, and SD-A, SD-B, and SD-E, which

Figure 5.

Heatmap and dendrogram of the 24 volatile compounds quantified in the five distillate samples. The relative content of each compound is depicted using a chromatic scale ranging from dark blue (minimum) to dark red (maximum). The dendrogram, generated through hierarchical cluster analysis (HCA) using Ward's clustering algorithm, is included to illustrate the grouping of compounds (on the left) and samples (at the top). Detailed data are provided in Supplementary Information Table S1.



displayed greater variability with elevated levels of specific compounds, such as 2-methylpropyl acetate.

When comparing the monoterpenic profiles of SD-A, SD-B, SD-C and SD-D, under the same distillation pattern for the head fraction (either high or slow), higher concentrations of monoterpenic compounds were observed in samples with lower reflux during the heart distillation (SD-B and SD-D). These findings are consistent with those of Matias-Guiu et al (2016), who reported that a significant reduction in internal reflux during the heart distillation enhanced the recovery of terpenic compounds.

SD-B and SD-E were found to be more closely related suggesting they shared similarities in their volatile profiles, likely to be due to overlapping concentrations of specific compounds. However, SD-E had distinctive characteristics, notably the higher concentrations of β-damascenone and β-citronellol (more than double the concentration found in other samples). Interestingly, β-damascenone, beyond its contribution to floral notes, has been identified as a key compound in enhancing the fruity character of esters (Pineau et al. 2007). Further, research by Garbay et al (2023) highlighted the role of β-citronellol, a compound characterised by rose, citronella, and citrus aromas, in modulating fruity aroma. When combined with other monoterpenic compounds, β-citronellol has been shown to enhance the intensity of fresh blueberry and blackcurrant notes in model mixtures of esters representing the fruity aroma of red wine.

Additionally, the presence of substituted esters - ethyl 2-hydroxy-4-methylpentanoate and ethyl 2-hydroxy-3-methylbutanoate - differentiated SD-E from the SD-B sample. These esters contribute to the blackberry fruit aroma in a model hydroalcoholic mixture and a red wine matrix along with other esters (Lytra et al. 2012).

Conversely, long chain ethyl esters (ethyl octanoate, ethyl decanoate, and ethyl dodecanoate), are associated with fruity, waxy and soapy notes (Antalick et al. 2010). These compounds were found at lower concentrations in SD-E compared to SD-B. This nuanced relationship highlights that while there were commonalities, SD-E possessed features that differentiate it from SD-B.

Perceptual interaction of esters and monoterpenic compounds: impact on the floral perception of new make spirit

Despite the variations in the concentration of some compounds, and the intensities of the selected descriptors among the samples, Pearson correlation tests failed to establish a significant relationship between the concentration of compounds and the intensity of the aromatic notes (data not shown). This lack of correlation underscores the complexity of aroma perception, suggesting that factors beyond the concentration of individual compounds, such as perceptual interactions between volatiles, play a critical role.

Consequently, a study on perceptual interactions is important for understanding how these compounds interact to shape the perception of aromas. Research has highlighted the role of perceptual interactions in odour mixtures, particularly in perfumery and food flavouring. Models have demonstrated how odorant interactions can alter the perception of blends (Cain and Drexler 1974; Thomas-Danguin et al. 2014). In oenology, studies confirm the impact of these interactions in complex aroma systems like wine. Compounds, such as monoterpenes, norisoprenoids, and esters - which are included in this work - provoke synergistic effects, often involving molecules below sensory thresholds (Lytra et al. 2013; Gabray et al. 2024). These findings highlight the intricate nature of aroma perception in complex matrices, where volatile interactions shape the sensory identity of the final product.

The sensory and chemical results reported here demonstrate that, compared to SD-A, new make spirit SD-E exhibited superior quality, characterised by more intense floral aromas, significantly higher concentrations of esters, monoterpenic compounds, and C13-norisoprenoids. To evaluate whether the enhanced floral aroma in SD-E is derived from these compounds, aromatic reconstitutions were made.

New make spirit SD-A was used as the base matrix which was supplemented with mixtures of either esters or monoterpenic compounds together with C13-norisoprenoids at concentrations corresponding to those in SD-E. These reconstituted samples were compared to each other to evaluate the specific impact of these aroma compounds on the sensory profile. Sensory profiles applied on a 100 mm scale of intensity scale revealed significant differences in aromatic complexity between SD-A and SD-A supplemented with either (i) monoterpenic compounds and C13-norisoprenoids, or (ii) esters, at concentrations found in SD-E. Specifically, monoterpenic compounds and C13-norisoprenoids, such as β-citronellol, terpinolene, α-terpineol, geraniol, and β-damascenone, were associated with enhanced aromatic complexity, with white flower and fresh rose notes (Figure 6a). Meanwhile, esters, including ethyl butanoate, ethyl hexanoate, ethyl 2-methylpropanoate, ethyl 2-methylbutanoate, and ethyl 6-hydroxyhexanoate, unexpectedly contributed to floral attributes rather than the fruity notes which they are typically associated with matices such as whisky and wine (Figure 6b).

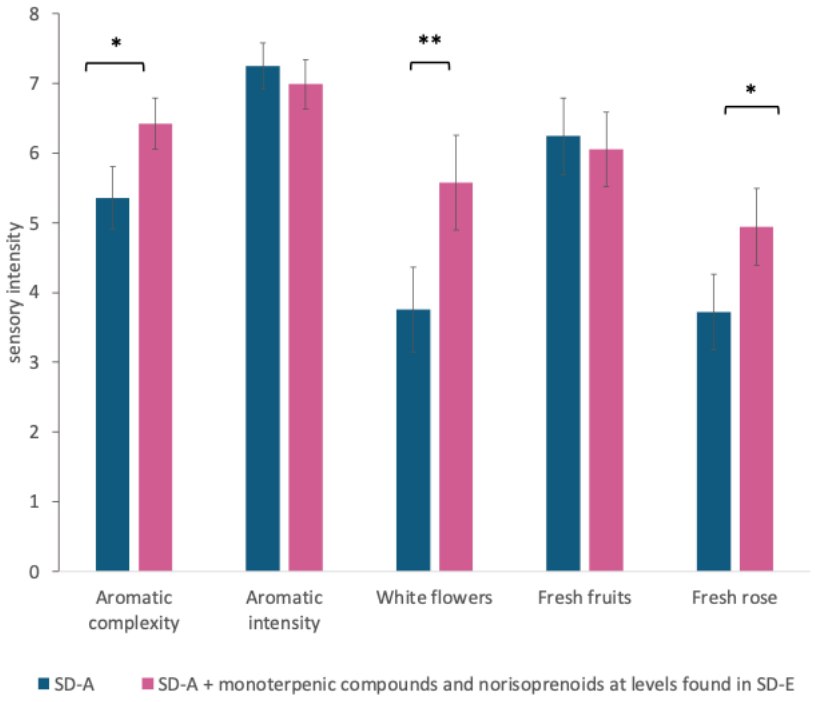
Recently, linalool, geraniol, and citronellol have been associated with flowery notes (such as rose-like attributes) in whisky (Shen et al. 2024). Moreover, Guo et al (2024) found that β-damascenone correlates with floral and honey-like notes. The chemical family of ethyl esters constitute key molecular markers in whisky, and their direct impact on the fruity perception of whiskies is well known, due to their comparatively high concentration (mg/L) (Salo et al. 1972; Poisson et al. 2008). However, considering the olfactory impact on the flowery perception of whisky, only phenylethyl acetate has been associated with rose-like aromas

Figure 6.

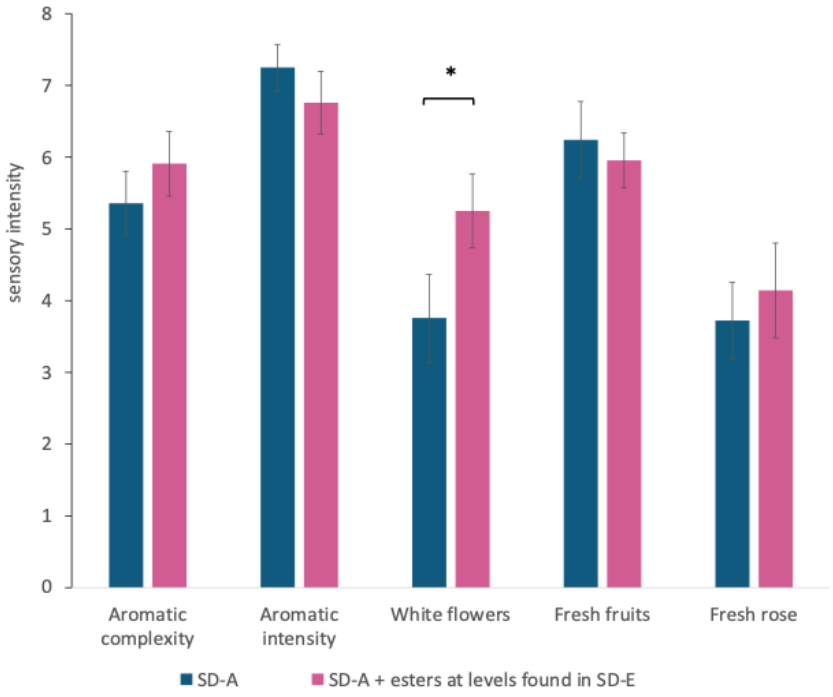
Olfactory impact of (a) monoterpenic compounds and C13-norisoprenoids and (b) esters added to SD-A at levels found in SD-E.

* 5% and ** 1% significance levels determined by Wilcoxon signed-ranked statistical nonparametric test.

(a)



(b)



has been shown (Kelly et al. 2023). Indeed, this is the first time that ethyl esters have been linked to floral perception through synergistic effects in complex matrix has been shown. These findings reiterate the complex interplay of these compounds in shaping the sensory profile of spirit and align with prior studies on their aromatic contributions.

This work highlights the importance of monitoring floral markers alongside other aroma active compounds that may negatively affect perception. Such effects may arise when these compounds exceed their perception thresholds or through sensory masking, where the presence of volatiles suppress or modify the perception of desirable aromas. Future research should involve the quantification of a broader range of potential off-flavour compounds, including, but not limited to, aldehydes and volatile sulphur compounds. The results can then be interpreted in relation to sensory thresholds and integration with analysis of any sensory-chemical correlation.

Conclusions

This study highlights the influence of controlling the distillation rate on the aromatic composition of single malt whisky produced in pot stills. Through combined sensory and chemical analyses, it was shown that a high reflux during the foreshots phase enhances the retention of complexity, intensity, and floral aromas in the heart, while limiting undesirable off notes. The innovative implementation of a multi-stage flow rate adjustment strategy, designed to modulate presumed reflux intensity and distillation dynamics during heart distillation, proved effective in amplifying the perception of floral notes and refined the balance of the new make spirit.

This approach optimised the gradual selection of volatile compounds, resulting in a more intricate and harmonised profile. This work also highlighted the importance of monoterpenes, C13-norisoprenoids, and esters in defining floral attributes, with perceptual interactions between these compounds contributing to an intensified aromatic profile. Notably, the contribution of esters to floral notes, rather than exclusively fruity, points to synergistic effects that merit further investigation. However, while this study provides useful insights into optimising distillation techniques for maximising floral expression, the complexity of aroma interactions in whisky suggests that additional unidentified compounds may also contribute. Finally, while this study focuses on the impact of distillation, it should be noted that the composition of the new make spirit also provides the foundation for subsequent maturation. Terpenes, esters, and related volatiles present after distillation persist and interact with oak derived compounds during aging, contributing jointly to the final aromatic profile of mature whisky. Further investigations are required to unravel these interactions and examine how additional distillation parameters, combined with cask ageing, shape the long term floral expression of whisky.

Author contributions

Magali Picard: experimental design, methodology, formal analysis, resources, investigation, data curation, validation, supervision, writing (original draft, review and editing), project administration, funding acquisition.
Esteban Muller: investigation, formal analysis, data curation.
Ivan Lainé: methodology, investigation, resources, data curation, writing (original draft).
Clément Chambolle: investigation, formal analysis, data curation.
Justine Garbay: investigation, formal analysis, data curation, writing (original draft, review and editing).
Georgia Lytra: experimental design, methodology, formal analysis, resources, investigation, data curation, validation, supervision, writing (original draft, review and editing), project administration, funding acquisition.

Acknowledgements

The authors thank Martine Esteban-Decloux for her helpful advice in writing this paper.

Conflict of interest

The authors declare no competing financial interest.

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