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Vacuum distillation of Muscaris pomace: temperature effects on aroma composition

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Abstract. The consumption of wine in traditional wine-producing countries like Italy, Spain, and France is decreasing. However, there is an increasing demand for non-alcoholic or low-alcohol wines, which has become a growing trend. This shift in consumption patterns has raised interest from both, companies and researchers. As the popularity of these products grows, so does the development of technologies to remove alcohol from wine. Currently, there are two main approaches for complete dealcoholization of wine for commercial use: membrane techniques, such as osmotic distillation, reverse osmosis (often coupled with another method), and dialysis, or distillation techniques, such as SCC (Spinning Cone Column) or vacuum distillation.

One of the major challenges with these technologies is the loss of aromatic compounds during processing. Several strategies have been developed to either reduce this aroma loss or to enhance aromas using other materials.

One potential solution for improving aroma compounds in dealcoholized wine is to use by-products of the winemaking process, namely pomace, which consists of grape skins, seeds, and stems. Pomace can make up 15-30% of the total weight of the processed grapes. There are three types of pomaces: non-fermented pomace, semi-fermented pomace and fermented pomace. To extract aromas from pomace, various methods can be employed, such as distillation (at atmospheric or reduced pressure), pressing, supercritical fluid extraction, or solvent/enzyme extraction. The aim of this research is to investigate the differences in aroma extraction from pomace at different temperatures under vacuum conditions. For the experiment, Muscaris's pomace from the Rheingau (Germany) was used. The pomace was acidified and fermented (Lalvin EC1118), and then it was treated by vacuum distillation. A total of 2.5 kg of pomace was processed at 50 mbar, with extractions performed at four different temperatures: 25, 35, 45, and 90°C. The vacuum distillation process was stopped once 100 mL of distillate has been collected. All aroma extracts were analysed by gas chromatography (GC) to compare the differences in aroma composition across the various extraction methods. Moreover, GC analysis for methanol was also carried out to determine whether there was a possible increase in methanol, over the legal limit, in case of addition of the aroma in a dealcholised wine. This analysis will provide understandings into the best conditions for extracting aromas from pomace.

1. Introduction

In 2023, the wine industry in Europe produced around 237 million hectolitres of wine (significant decrease of nearly 25 mhl (-9.6%) compared to 2022), while consumption, in 2023, is only 221 million hectolitres (a decrease of 2.6% compared to 2022). The extra 16 million hectolitres were used for other purposes, such as producing vinegar, distillates like Cognac or Brandy, or aging in barrels [1]. However, wine consumption has been decreasing over the years. The decline in global wine consumption has followed a relatively stable path since 2018. A combination of elements has contributed to the decrease in wine consumption, including the significant reduction of consumption in China, the impact of the global pandemic, ongoing geopolitical and economic pressures, and the diminishing purchasing power of consumers [2]. As the market landscape evolves, many wineries have been forced to explore alternative strategies to sustain their businesses. One promising approach is the production of dealcoholized wine, which has the potential to expand market reach by attracting consumers who do not usually drink alcoholic beverages for religious, health-related, or medical reasons. Over the past five years, the market for dealcoholized wine has experienced notable growth. In 2021, the non-alcoholic wine market was valued at over US$ 1.6 billion, with projections indicating a compound annual growth rate (CAGR) of 10.4%, reaching an estimated US$ 4.5 billion by 2031. This marks an increase from the 8.8% CAGR recorded between 2016 and 2020 [3]. Given this trend, research must prioritise enhancing the quality of dealcoholized wine.

At present, all dealcoholization methods approved by the OIV result in the loss of aromatic compounds [6]. The removal of alcohol also modifies the wine's sensory balance, leading to increased acidity and reduced sweetness [4–6]. Depending on the technique employed—whether distillation or membrane-based processes—aroma losses can range from 35.54% to 93.62%, with the risk of complete aroma loss if the process is not carefully managed [7]. However, the extent of these losses varies significantly depending on the specific technology used. Therefore, preserving aromas is crucial, and an alternative approach involves reintegrating them into the wine. A promising solution lies in recovering aromas from grape pomace, the most significant by-product of winemaking [8]. Research has shown that pomace accounts for 15% to 30% of the total weight of processed grapes [9,10].

Pomace can be classified into three types:

- **Non-fermented pomace:** obtained after grape pressing.
- **Semi-fermented pomace:** subjected to short maceration, during which fermentation begins in the juice.
- **Fermented pomace:** fully fermented along with the juice, as occurs in red winemaking.

Pomace consists mainly of skin (73-91%), seeds (7.9-21%), and stems (0.54-1.84%) [11]. More specifically, its composition includes water (50-70%), cellulose (10-20%), sugars (6-8%), fatty compounds (2-4%), organic acids (1-2%), tannins (1-2%), and minerals (1-2%) [12].

One of the main challenges in pomace distillation, regardless of its type, is determining the optimal timing. Ideally, distillation should occur as soon as fermentation is complete, with minimal handling, as every movement can reduce ethanol levels by approximately 0.5% v/v. This issue is further complicated by the seasonal nature of grape production, which is concentrated within a few months each year. Depending on storage duration, conditions, and methods, pomace preservation poses several challenges, including susceptibility to bacterial or mould contamination. Such contamination can lead to the development of undesirable compounds that affect the sensory profile of the resulting spirits [13]. Notably, compounds such as ethyl acetate, acetic, butyric, and propionic acids, 2-butanol, and long-chain fatty acid ethyl esters have been identified as key contributors to these off-flavours [14]. Another problem with pomace is the high concentration of methanol.

Methanol, or methyl alcohol, is a toxic compound that harms human health through two primary mechanisms:

- central nervous system depression similar to ethanol intoxication, and
- metabolic conversion to formic acid via formaldehyde, which inhibits mitochondrial cytochrome c oxidase and leads to cellular hypoxia, metabolic acidosis, and other disturbances [15].

Table 1. Resolution OIV: OENO 19/2004 Maximum content limits of methanol in wines

Type of Wine	OIV Methanol limits (mg/L)
Red	400
White and Rose	250

The International Organisation of Vine and Wine (OIV), therefore enforces legal limits on methanol content in Table 1. Naturally occurring in all alcoholic beverages, methanol is particularly concentrated in fruit-based distillates owing to their high pectin content. Pectin—a branched heteropolysaccharide of galacturonan chains and neutral sugars such as arabinose, xylose, rhamnose, and galactose—provides structural integrity alongside cellulose and hemicellulose fibres [16,17]. During grape-juice extraction, pectin methyl esterase (PME) rapidly demethylates pectins, saponifying $–OCH_3$ groups within 24–48 hours, whereas polygalacturonase (PG) requires up to eight days to depolymerize the pectin backbone in the absence of active PG [18].

Nevertheless, pomace remains a valuable resource, rich in bioactive compounds and aromatic substances, making it crucial to minimise waste.

The objective of this study is to investigate aroma extraction from pomace under vacuum conditions at different temperatures. Identifying the optimal temperature for obtaining an aroma extract suitable for incorporation into dealcoholized wine could cover the way

for the development of aromatized dealcoholized wine. Future research should focus on evaluating the impact of these aroma extracts on the sensory characteristics of dealcoholized wine, as well as assessing whether different grape varieties respond similarly to their addition.

2. Materials and Methods

For this study, 100 kg of destemmed Muscaris (Solaris × Muskateller) white-grape pomace from the 2024 vintage grown at Hochschule Geisenheim University (Rheingau, Germany) were used.

2.1. Pomace Fermentation

The pomace was placed in a fiberglass container, and stratified in 20 cm layers. For each layer before compaction tartaric acid (2 g/kg) and commercial yeast EC 1118 Lalvin (Lallemand Oenology) (0.175 g/kg) were added. After that, the container was covered with a plastic layer and sand. The fermentation process lasted for three months at a temperature of around 8°C. Afterwards, the first 10 cm of the pomace were removed to ensure a better material quality and to avoid the distillation of the outer layer, which was more exposed to oxygen and spoilage. The grape pomace was collected from the bucket uniformly, to ensure a homogeneous starting material.

2.2. Aroma Extraction

The aroma extraction from the grape pomace was conducted using Hei-VAP Industrial Rotary Evaporators (Heidolph Instruments GmbH & Co. KG, 91126 Schwabach, Germany). This system comprised a heating bath capable of water temperature settings up to 180°C, a vacuum pump (Hei-Vac Valve Industrial) for evacuating, evaporating, and pumping out gases and vapours, and a chilling system (Hei-CHILL 3000) for condensation.

At each step, 2.5 kg of pomace and 1 liter of distilled water were distilled in a 20-L evaporating flask. The vacuum pressure was maintained at 50 mbar, with a water bath temperature initially set at the temperature target and a feed flask rotation speed of 80 rpm. The condensation was kept constant at 1.8°C throughout the experiments. After collecting 100 mL of the first pomace´s aromatic fraction, the machine was cleaned, and a new step began with a different target temperature. Extractions were performed at 25, 35, 45, and 90°C, each in triplicate. The aroma extract was conserved in two small glass tubes (50 mL), in a fridge with a temperature of 4°C and in a dark environment for three months, before being analyzed.

2.3. Analysis

2.3.1. Ethanol Determination

All ethanol analyses were carried out using DMA 4500M density meter, a Lovis 2000 M/ME rolling ball viscometer, and an Alcolyzer (Anton Paar GmbH, Graz, Austria), available at the Beverage Technologies laboratory of the Hochschule Geisenheim University.

2.3.2. Determination of Monoterpenes, Monoterpenoids and C13-Norisoprenoids in samples with high ethanol content

Free terpenes and C13-norisoprenoids in samples with high alcohol content were analysed with adapted concentrations of references substances by means of headspace solid phase microextraction (HS-SPME) in connection with gas chromatography and mass spectrometry (GC-MS) according to the method described by Brandt (2021) and Câmara et al. (2006) [20-21]. 5 mL of sample was transferred to a 20 mL amber headspace vial containing 1.7 g of NaCl. 10 µL of an internal standard mix was added. The vials were closed and kept at 7°C prior analysis. HS-SPME sampling was carried out with a polydimethylsiloxane-coated SPME fibre (100 µm, 23 ga, Supelco, Bellefonte, PA USA). After an incubation time of 10 min (agitation at 500 rpm, 40°C) samples were extracted for 20 min.

For GC-MS analysis an Agilent 6890 GC coupled to a quadrupole mass spectrometer Agilent 5973 N (Agilent Technologies, Palo Alto, CA, USA) was used. Thermal desorption of the fiber took place at 240°C for 4 min in splitless mode. For chromatographic separation of the compounds a 30 m DB-WAX column with an internal diameter of 0.25 mm and a film thickness of 0.5 µm (J & W Scientific, Agilent Technologies, Palo Alto, CA, USA) was used. The initial temperature of the GC was set to 40°C (4 min), raised to 190°C with 5°C/min, further ramped to 240°C with 10°C/min and held for 15 min. Helium was used as carrier gas at 1.2 mL/min in constant flow. The mass spectrometer was set in SIM Mode (temperature ion source: 230°C, temperature quadrupole: 150°C).

Data analysis was done with MassHunter Software (Agilent Technologies, Palo Alto, CA, USA).

2.3.3. Methanol Determination

For the quantification of methanol, samples with high ethanol concentrations are first diluted to 10 % EtOH, and the dilution factor has to be considered for evaluation. Wine and wine-containing beverages do not need to be diluted. For sample preparation, 5 mL of the (diluted) sample is added to a 10 mL glass vial and spiked with 10 µL of an ethanolic solution containing 103 g/L methanol-d_3 as an internal standard.

The measurements were performed using HS-GC-MS on an Agilent 7890B gas chromatograph (Agilent, Santa Clara, California, USA) equipped with a CIS4 Cooled Injection System and an MPS robotic autosampler (both from Gerstel, Mülheim an der Ruhr, Germany) and coupled to a 5977B mass selective detector (Agilent, Santa Clara, California, USA).

Sample extraction was carried out using a Headspace Sampler HSS (Gerstel) with the following method:

incubation temperature of 80°C, incubation time of 30 minutes, stirring speed of 300 rpm, and an injection volume of 400 µL per sample. Injection was performed in split mode (1:10) into the Cooled Injection System CIS 4 (Gerstel) at a starting temperature of 10°C with a heating rate of 12°C/min. The chromatographic separation was achieved using a Stabilwax-DA column (30 m × 0.25 mm × 0.25 µm, Restek GmbH, Bad Homburg, Germany) connected to an IP Deactivated Guard Column (5 m × 0.25 mm ID, Restek GmbH, Bad Homburg, Germany). The chromatographic conditions were 30°C (10 min) followed by a ramp of 20°C/min to 240°C (10.5 min) with helium as the carrier gas at a constant flow of 1.2 mL/min. The temperature of the ion source operating in EI (electron impact) mode at 70 eV was maintained at 230°C, and the temperature of the quadrupole was set at 150°C. For this method, the solvent delay was 2.2 min, and the detector was switched off at 3.63 min.

Detection was carried out in SIM with the following masses and retention times: methanol (m/z = 29, 31, **32**), methanol-d_3 (m/z = 30, 33, **35**). Data analysis was performed with Agilent MassHunter Workstation Software (version B.08.00).

2.3.4. Statistical Analysis

All measurements were conducted in triplicate, and results are expressed as mean values with standard deviations. The normality of data was assessed using the Shapiro–Wilk test. All aroma compounds passed the normality test, except for β-damascenone. One-way ANOVA followed by Tukey's post-hoc test ($p < 0.05$) was applied to evaluate the effect of temperature on normally distributed parameters. For β-damascenone, which did not follow a normal distribution, the Kruskal–Wallis test was used instead. All statistical analyses were performed using R Commander (version 2.8-0), and graphical representations were developed using SigmaPlot software (version 14.5, Systat Software Inc., San Jose, CA, USA).

3. Result and Discussion

Graph 1 presents the analysis of some aroma components, specifically several terpenes, β-damascenone, and methanol, extracted at various temperatures under vacuum conditions (50 mBar). The results indicated no significant difference in these parameters across the operating temperatures. This suggests that other factors likely had a greater influence, such as the pomace fermentation process, storage conditions prior to distillation, and pre-treatment.

3.1. Ethanol and Processing Time

As expected, higher temperatures accelerated the distillation process. Increasing the liquid raising the liquid's temperature also increases the kinetic energy of its molecules. This allows more molecules to overcome the intermolecular forces holding them together and transition into the gas phase, consequently increasing the vapor pressure of the liquid. This leads to drastically decrease distillation times, from 25°C (85 ± 3 minutes) to 90°C (4 ±1 minutes). Interestingly, the lower distillation temperatures of 25 and 35°C resulted in a slightly higher alcohol concentration in the distillate compared to higher temperatures of 45 and 90°C, produced slightly lower concentrations of ethanol in the final distillate. The observed dilution of the aroma extract at elevated distillation temperatures can be attributed to the principles governing vapor-liquid equilibrium (VLE) and mass transfer kinetics which shift as temperature increases. Specifically, as temperature increases, more water co-distills with ethanol, potentially diluting the aroma-rich fraction.

3.2. Terpenic Compounds

Terpenic compounds are characteristic of aromatic grape varieties such as Muscat [19,20]. Among these, linalool, α-terpineol, and citronellol are the most prominent monoterpene alcohols in grapes and grape-derived products [21]. These compounds typically exist in bound forms, linked to sugars as glycosides, and can be liberated through enzymatic or acid hydrolysis [24]. Thermal processing (e.g., distillation or aroma recovery) can facilitate the hydrolysis of glycosides, thereby releasing free monoterpenes ([25]).

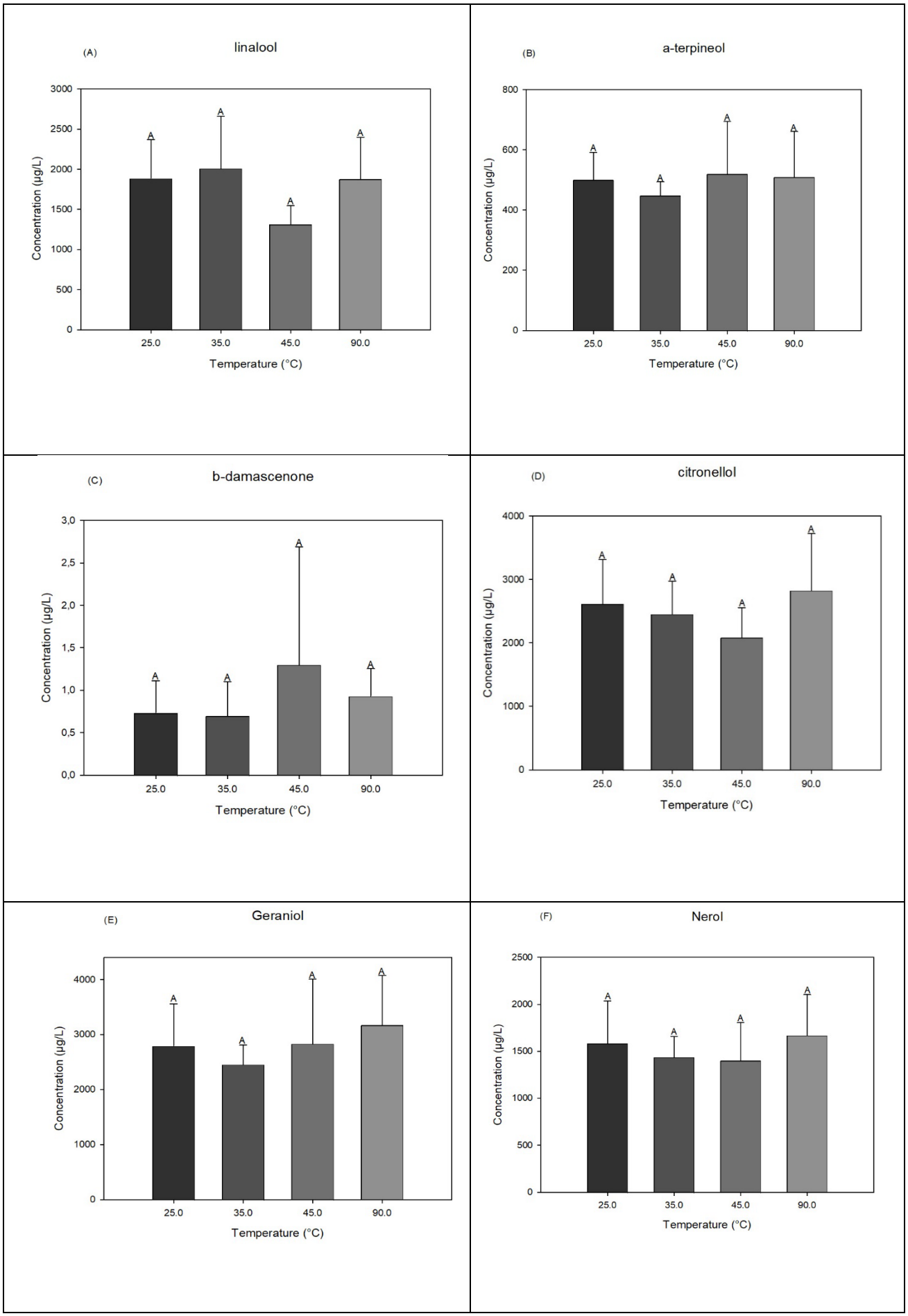

Figure 1 Bar graph showing the content (with standard deviation error bars) of selected aroma compounds in the final 100 mL aroma extract obtained using different temperature approaches. The applied pressure was consistently 50 mbar. (A) linalool; (B) α -terpineol ; (C) β -damascenone; (D) citronellol; (E) geraniol; (F) nerol. "A" indicates no statistically significant differences among samples ($p<0,05$).

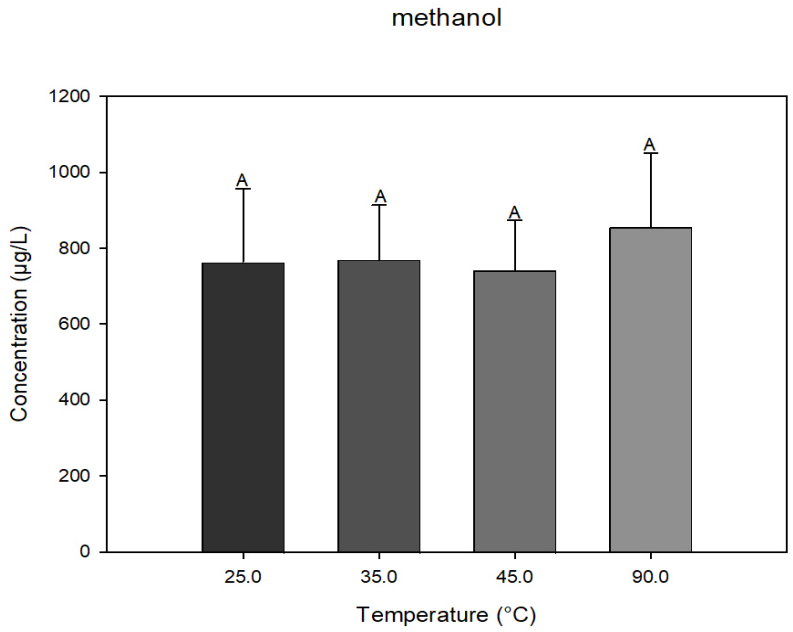

Figure 2 Bar graph showing the content (with standard deviation error bars) of methanol in the final 100 mL aroma extract obtained using different temperature approaches. "A" indicates no statistically significant differences among samples ($p<0,05$).

Table 2. The temperatures used, the duration of the distillation after a 5-minute machine warm-up, the final ethanol concentration, and the steam temperature are reported. For each temperature, the pressure used was standard, at 50 mbar.

Temperature	**Duration of the distillation**	**Ethanol**	**Steam Temperature**
(°C)	(min)	(% V/V)	(°C)
25	85 ± 3	19.13 ± 3.40	26.17 ± 0.45
35	65 ± 4	19.21 ± 4.39	33.17 ± 0.70
45	13 ± 4	16.23 ± 2.66	39.03 ± 0.21
90	4 ± 1	16.33 ± 4.81	52.13 ± 1.80

Table 3. Data for the main compounds analyzed, including boiling point, Henry's constant, odor threshold, typical concentration in pomace distillate, aroma descriptors, and references

Compound	Boiling Point (°C @760 mmHg)	Volatility (Henry's constant)	Odor Threshold (µg/L, in wine)	Typical concentration in Pomace Distillate	Aroma Descriptors	Ref.
ethanol	78.24	5×10^{-6} atm·m³·mol⁻¹ at 25°C	~150	~35–60% v/v (350–600 g/L)	Pungent, "alcoholic"	[29] [30] [31]
methanol	64.7	4.55×10^{-6} atm·m³·mol⁻¹ at 25°C	~9	up to ~3 g/L	Pungent	[29] [32]
water	99.9	— (solvent)	— (odorless)	~40–60% v/v (main solvent)	Odorless, neutral	[29] [31]
linalool	198.0	2.15×10^{-5} atm·m³·mol⁻¹ at 25°C	~50	30-60	Sweet, floral-citrus (lavender, lily-like)	[29] [30] [32] [33] [34] [35]
α-terpineol	218.0-221.0	2.8×10^{-4} atm·m³·mol⁻¹ at 25°C	~400	15	Floral-lilac, piney, fresh	[29] [31] [33] [36] [37] [38]
β-damascenone	274.0–276.0	3.2×10^{-1} atm·m³·mol⁻¹ at 25°C	~0.05	0.05	Honeyed, fruity (apple/plum), floral	[38] [39]
citronellol	223.0–224.0	2.1×10^{-5} atm·m³·mol⁻¹ at 25°C	~18	20	Sweet, fresh floral (rose, citrus)	[29] [31] [33] [36] [38]
geraniol	230.0	1.15×10^{-5} atm·m³·mol⁻¹ at 25°C	~130	120	Rosy, sweet floral (geranium-like)	[29] [31] [33] [41] [42]
nerol	224.0–225.0	3.2×10^{-4} atm·m³·mol⁻¹ at 25°C	~400	80	Sweet, fresh (rose-like, geranium-like)	[29] [31] [32] [33] [37] [38]

3.3. Linalool and α-Terpineol

Despite the potential for hydrolysis, the concentrations of linalool and α-terpineol did not differ significantly across the tested temperatures. This can be attributed to the balance between glycoside compound release and thermal degradation or transformation. Under vacuum (50 mbar), the boiling points of these compounds are sufficiently reduced to allow their extraction at relatively mild temperatures, thereby minimizing decomposition. However, the relatively short exposure times and mild acidity may have limited glycosidic hydrolysis, resulting in similar final concentrations.

This observation is supported by literature indicating that linalool and α-terpineol are both relatively thermally stable under controlled distillation conditions, although they may interconvert or degrade under more aggressive processing [22,24].

3.4. Citronellol, Geraniol, and Nerol

Similarly, citronellol, geraniol, and nerol did not differ significantly across the temperature range. These compounds have relatively high boiling points (~225–230°C) and are known to be stable under vacuum conditions. The lack of significant variation suggests that thermal hydrolysis of their glyosidic precursors was limited, and minimal thermal degradation or transformation occurred under the mild vacuum conditions applied.

Studies on Muscat pomace distillation have shown that under low-pressure conditions, citronellol, geraniol, and nerol remain stable and are consistently found in aroma fractions, confirming our findings [26].

3.5. Norisorpenoids

Norisoprenoids are potent aroma compounds derived from carotenoid degradation and can exist as free volatiles or sugar-bound precursors [27]. Among these, β-damascenone is one of the most relevant due to its extremely low odor threshold, 50 µg/L [29], and its presence significantly affects wine aroma.

In this study, β-damascenone levels showed a numerical peak at 45°C, but the variation was not statistically significant. However, the high standard deviations observed across temperatures suggest that matrix effects or precursor availability may have influenced the results more than temperature.

Previous studies confirm the importance of this compound for aroma recovery. For example, when added back via distillates, β-damascenone is almost fully recovered in dealcoholized wines, unless additional treatments (e.g., resin-based ethanol removal) are applied [22].

3.6. Methanol

The data on methanol concentration did not differ significantly across the temperature range. This suggests that once the fraction containing the majority of the methanol was collected, changing the pressure or temperature didn't change the total amount of methanol obtained. In the results, the methanol levels were similar from 25 to 90°C. While any methanol left in the pomace after the first 100 mL would likely appear in later fractions (which it was not analysed), so it is possible, that this remaining amount is relatively small. Interestingly, the study by Da Porto [28] on grape pomace distillation indicated that methanol concentrations in later fractions stayed quite consistent, whether they used vacuum or normal pressure.

Even though the extracted aroma concentrate shows high concentration of methanol, using it to add back aroma in the wine, it should not increase the level of methanol over the legal limit, due the dilution effect (1:10), into wine gives very low final levels.

4. Conclusion

The extraction yields did not differ significantly between the temperatures tested. This is probably because factors such as the pomace's fermentation state and storage conditions play a much larger role than the modest head-space volume collected (100 mL from 2.5 kg). Since we never exceeded temperatures high enough to break down target compounds, we simply recovered the most volatile components—aside from minor changes in ethanol content.

Although our methods were precise and reproducible, the study's scope was limited by the small number of compounds analysed and the narrow selection of grape varieties. Future research should examine how condenser temperature influences the aroma profile, include a broader spectrum of volatile compounds, and—most importantly—validate these results through sensory trials by blending the recovered aroma back into dealcoholized wine and assessing consumer preference.

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