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EXPLORING THE INFLUENCE OF WOOD CHIPS ON THE AROMA COMPLEXITY OF APPLE BRANDIES

– Research paper –

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Abstract: Wooden barrels are traditionally used for aging alcoholic beverages. We decided to use different types of wood chips (pear, cherry, walnut, medium roasted oak and heavily roasted oak) to improve flavor of apple brandies obtained from Rubin cultivar. Our research proved that aging brandies with wood chips could result in alcoholic beverages with an interesting flavor. Moreover, obtained results would provide guidance on adjusting the flavor of brandies during aging. Esters, particularly methyl and ethyl esters, were found to be dominant compounds in the brandies, with some (e.g. isopropyl acetate, ethyl propanoate) present in the brandies before aging and others likely developed during aging with specific wood types, such as methyl pentanoate. Brandies aged with cherry chips displayed the most diverse profile of volatile compounds, while those aged with roasted chips showed higher concentrations of certain higher alcohols and other compounds e.g. 1-tetradecanol (31.2 mg/L), 5-methyl-2-furancarboxaldehyde (100.1 mg/L) and benzeneacetaldehyde (22.1 mg/L). Additionally, some terpenes were detected exclusively in brandies aged with wood chips, e.g. citral, limonene, geraniol, β-citronellol, linalool, α-copaen-11-ol. Sensory analysis revealed that brandies aged with cherry and heavily roasted chips received the highest overall ratings, characterized by descriptors including pleasant, sweet, floral, clove, citrus, and intensely apple. The aroma profile of brandies aged with roasted oak chips was attributed to various aromas associated with roasting and maturation processes, such as woody, coconut, burnt sugar, and buttery notes.

Keywords: wood chips, apple brandies, volatile compounds, aging, aroma-active compounds

INTRODUCTION

Aging is a crucial stage in the production of alcoholic beverages, such as wine, beer, mead, and spirits. It involves storing the beverages in barrels, vats, or tanks within designated cellars, under specific conditions. The primary purpose of aging is to clarify the alcoholic beverage and facilitate gradual physical and chemical changes, thereby enhancing its flavor profile (Rum,2005).

Calvados is the most famous apple brandy, produced in Normandy. Traditionally, Calvados matures for a minimum of two years. Subsequently, the distillate is diluted with water to reduce its strength to about 40-50%. It is preferable for the aging time of Calvados to extend well beyond the required two years, as this additional time enhances the development of its characteristic aroma and appropriate color. Traditionally, oak or chestnut barrels with a capacity of no more than 600 liters are used for this process. As aging progresses, the most astringent components, which give Calvados its spicy taste, are transformed into aromas that are pleasant to the senses. Calvados acquires various chemical substances from the wood, including volatile esters, terpenes, lactones, and tannins (Institut Départemental de l'Appellation Calvados, 2024).

Natural and French oak wood (*Quercus alba, Quercus petraea*, and *Quercus robur*) are the primary materials used for producing barrels and wood chips for aging alcoholic beverages. Wood is mainly composed of cellulose, holocellulose and lignin, which account for about 90% of its structure. The remaining 10% of wood is made up of a small amount of inorganic compounds and, more importantly for the

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production of alcoholic beverages, substances extracted during maturation. These substances contribute unique aromas, flavors, and colors to the drinks. Key extractives include volatile and nonvolatile steroids, esters, alcohols, terpenes, terpenoids, lactones, and phenols (Tarko et al., 2023).

The use of wood chips has been a common practice permitted in Europe since the beginning of the 21st century. Wood chips play a crucial role in achieving various oenological objectives, such as extracting volatile compounds from the wood and enhancing the quality of beverages (Tarko et al., 2023). Furthermore, the economic benefit of using wood chips—due to their lower cost compared to traditional wooden barrels—cannot be overlooked (Petrozziello et al., 2020). By employing various types of wood chips, with different levels of burning and in varied concentrations, it is possible to shorten the aging time while achieving specific sensory characteristics in the final product. In the current study, we analyzed the profile of volatile compounds in apple brandies that were aged with the addition of wood chips derived from American and European oak, as well as other tree species. Some of the woods selected for this study have been either poorly investigated or completely unexplored in the context of aging apple brandies. The types of wood chips used in this study included pear, cherry, walnut, medium roasted oak, and heavily roasted oak to age apple brandies made from the Rubin apple cultivar, which was selected based on previously published research (Januszek et al., 2020; Satora et al., 2008).

MATERIALS AND METHODS

Fermentation and distillation

The apple cultivar 'Rubin' used in the current experiments was selected based on previous studies and was harvested from the pomological orchards of the University of Agriculture in Krakow, located in Garlica Murowana. The apples were washed, crushed, and pressed using a Zottel hydropress (Zottel Trade d.o.o., Žalec, Slovenia), and the must was then transferred into conical fermentation flasks. The musts were supplemented with $(NH_4)_2HPO_4$ (0.2 g/kg of raw fruit) and inoculated with the SafSpirit HG-1 yeast strain (Starowar, Warsaw, Poland) – 0.3 g (dry weight) per 1 L of must. This yeast strain, which had been selected in previous research, was previously known under a different commercial name - Ethanol RED (Januszek et al., 2020a; Januszek et al., 2020b). The flasks were sealed with fermentation tubes filled with glycerol, and fermentation was conducted for 28 days at 20°C.

The fermented apple musts were distilled twice. The first distillation ceased when the ethanol concentration in the collected distillate dropped below 0.5% (w/v). These distillates contained between 11.8-13.1% (v/v) ethanol and were then subjected to fractional distillation using a glass column (40 cm) filled to 60% capacity with Raschig rings. During fractional distillation, three fractions were collected: the heads (2% of the total volume of 100% (v/v) distillate), the heart fraction (83%), and the tails (15%). The final ethanol concentration in the obtained brandies (heart fraction) was approximately 60% (v/v), in the head fraction 80% (v/v), and in the tail fraction 20% (v/v). The apple brandies were diluted to 40% (v/v) before aging and were transferred into glass bottles. Then, wood chips (heavily roasted oak, medium roasted oak, cherry, pear, walnut) were added (Browin, Łódź, Poland), and the samples were aged for 3 months at 20°C.

Volatile Composition of Apple Brandies Using Solid Phase Microextraction-Gas Chromatography-Mass Spectrometry (SPME-GC-MS) and Gas Chromatography-Flame Ionisation Detector (GC-FID) Methods

Volatile compounds were analysed by gas chromatography-mass spectrometry. We suspended 0.05 mg of 4-methyl-2-pentanol/L and 0.5 µg of ethyl nonanoate, (Sigma-Aldrich, St. Louis, MO, USA) in 1 mL of saturated saline, and this solution was used as an internal standard. We transferred 1 mL of samples to a 10 mL vial. We conditioned the SPME device (Supelco Inc., Bellefonte, PA, USA, 100 µm polydimethylsiloxane fibre) in the GC injector port at 250 °C for 1 h. After inserting the fibre into the glass vials, the samples were stirred (300 rpm/40 °C/30 min) and then the fibres were transferred to the injector port of the Agilent Technologies 7890B chromatograph system (Agilent Technologies, Santa Clara, CA, USA). The system is equipped with a LECO Pegasus HT, high-throughput time-of-flight mass spectrometry (TOFMS) detector. Fibres were held in the inlet for 3 min. Fibre transfer was automated using a GERSTEL MultiPurpose Sampler (MPS). Volatile compounds were separated on the Rtx-1ms capillary column (Crossbond 100% dimethyl polysiloxane, 30 m × 0.53 mm × 0.5 µm). The injector and detector were heated to 250 °C, while the separation of compounds was initiated at 40 °C for 3 min, and then the temperature was increased at a rate of 8 °C/min to 230 °C. Finally, the samples were held at the maximum temperature for 9 min. Helium, used as carrier gas, was supplied at a constant flow rate (1.0 mL/min), while the EIMS electron energy was 70 eV. The temperature of the source and connecting parts was 250 °C. The analytes were transferred in splitless mode. The mass spectrometer detector (MSD) was set to scan from m/z = 40 to m/z = 400.

Volatile compounds were identified using mass spectral libraries and linear retention indices (calculated from a series of C_6 to C_{30} n-alkanes). Semi-quantitative analysis of the compounds was assessed by the ratio of the relative peak area of each identified component to the relative peak area of an appropriate internal standard (ethyl nonanoate for esters, anethol for terpenoids and 4-methyl-2-pentanol for other components). The results obtained were analysed in the National Institute of Standards and Technology (NIST) database.

Determination of the selected volatiles was performed by gas chromatography (Hewlett Packard 5890 Series II chromatograph system; Agilent Technologies, Santa Clara, CA, USA) with a flame ionisation detector. A HP-INNOWax capillary column (30 m × 0.53 mm ID with 1.0 µm film thickness, cross-linked polyethylene glycol stationary phase; Agilent, Santa Clara, CA, USA) was used for the separation of volatile compounds. The temperature of both the detector and the injector was set at 250 °C. The initial temperature of the column was 35 °C (5 min), then increased to 110 °C at a rate of 5 °C/min, and then to 220 °C at a rate of 40 °C/min. The final temperature was maintained for 3 min. Helium was used as the carrier gas (20.0 mL/min). Hydrogen was supplied at a flow rate of 33.0 mL/min and air at 400 mL/min. Volatiles were identified and quantified by comparing the surface area obtained with that of standards.

Aroma-active Volatile Components (HS-SPME-GC-O)

Aroma-active volatile compounds of spirits were identified by olfactometry. A 2 mL sample of spirits containing 1 g NaCl in a 10 mL vial was exposed to a 50/30 µm divinylbenzene/carboxene/ polydimethylsiloxane (DVB/CAR/PDMS) SPME fiber (Supelco/Sigma-Aldrich, Bellafonte, PA, USA) for 40 min at 40 °C. The SPME device was then inserted into the injector port of the Hewlett Packard 5890 Series II chromatograph system and held in the inlet for 3 min. The components tested were separated on an Rxi®-1ms capillary column (Crossbond 100% dimethyl polysiloxa-ne; 30 m × 0.53 mm × 0.5 m). The detector temperature was 250 °C and the column was heated using the following programme 35 °C for 4 min at a rate of 5 °C/min to 110 °C, then at a rate of 20 °C/min to 230 °C, and then at a constant temperature for 4 min. The carrier gas was helium at a constant flow rate of 1.0 mL/min. An olfactory detection port (ODP-3, Gerstel, USA) was used for this analysis. Three trained GC-O analysts were asked to describe the aromas they perceived and its intensity. The odour intensity method was used with a 4-point scale (not detected, weak, moderate and strong).

Color analysis of apple brandies (CIElab method)

Colour assessment was performed using the CIE (Lab*) system with an instrumental approach. Measurements were taken using a Konica Minolta CM-3500d spectrophotometer (Konica Minolta, Inc., Tokyo, Japan), employing the reflectance method with illuminant D65 and a 10° visual angle. The parameters measured were:

- L* (lightness): L* = 0 indicates blackness, and L* = 100 indicates whiteness
- a* (color component on the red-green axis): a* < 0 indicates green, and a* > 0 indicates red
- b* (color component on the blue-yellow axis): b* < 0 indicates blue, and b* > 0 indicates yellow
- C* (chroma): the intensity or purity of the color
- h° (hue angle): the angle on the color wheel that defines the hue
- Delta E (ΔE) represents the difference between the colour of the test sample and the control, calculated as the square root of the sum of the squared differences between the L*, a*, and b* values.

Sensory assessment

Sensory assessment of apple brandies was based on 17 sensory indicators (floral, clove, sweet, fruity, apple, citrus, grassy, smoked, woody, coconut, vanilla pod, medicinal, burnt sugar, toast, yeast, pungent, buttery) and an overall note (general acceptance of the tested brandies) rated on a 5-point hedonic scale (1 - the lowest intensity; 5 - the highest intensity). We applied the Quantitative Descriptive Analysis (QDA) test (Satora et al., 2008). Panelists were scientific staff from the Faculty of Food Technology and Human Nutrition, who had graduated from that faculty and completed an extensive course in sensory analysis. Aroma evaluation was determined using a set of standards that panelists were trained to recognize at various concentrations. Only those panelists who successfully passed these evaluations were selected for the sensory analysis. Apple brandies were diluted to 40% vol. ethanol (EtOH) and subjected to sensory assessment by a panel of 10 testers (Januszek et al., 2020a; Januszek et al., 2020b; Caldeira et al., 2002). Brandies were coded and presented to panelists in 50 mL glass bottles.

Statistical analysis

All experiments were conducted with at least five replicates, and results are presented as arithmetic means ± standard deviation. Statistical analysis was performed using R version 3.5.0 (Vienna, Austria). ANOVA was conducted using the linear model (lm) function, and Tukey's Honest Significant Difference (HSD) test was applied using the 'agricolae' package. Sensory analysis results were subjected to one-way analysis of variance (ANOVA), followed by the Pearson correlation test for each descriptor.

RESULTS AND DISCUSSION

Volatile composition of apple brandies aged with chips

Aroma compounds can be divided into three categories: primary aromas, which are compounds present in the fruits; secondary aromas, which are volatiles generated during the fermentation process; and tertiary aromas, which are compounds formed during aging. Tertiary aromas include the aroma of wood, toasty, vanilla, caramelized, and spicy notes that enhance the flavor (Espitia-López et al., 2017). Many compounds are released from wood into alcoholic beverages during aging, including terpenoids, lactones, coumarins, polysaccharides, hydrocarbons, fatty acids, norisoprenoids, steroids, carotenoids, furan compounds, and esters (De Rosso et al., 2009). The maturation of brandies in wooden barrels or with wood chips leads to changes in the profile of volatile compounds. This is partly due to the concentration of ethanol decreasing as a result of its preferential evaporation through the wood staves (Călugăr et al., 2020). Beverages aged with the wood chips used in this study demonstrated a varied composition of volatile compounds. Among these, esters, which contribute to the fruity character of the brandies, were the dominant compounds. An exception was found with benzoates, among which only ethyl benzoate was detected. The highest concentration of this compound was observed in brandies aged with oak chips. Karathanos et al. (2008) discussed the evolution of certain major representatives of benzoate derivatives and their hydroxycinnamate analogues during the aging of wine in French oak barrels.

Methyl and ethyl esters were the predominant compounds in aged apple brandies, with 22 compounds identified. Among these, all of them were detected in brandies aged with medium roasted oak chips, and 19 compounds in those aged with cherry wood chips. Some of these methyl and ethyl esters, such as isopropyl acetate and ethyl propanoate, were present only in spirits, while others appeared to have transferred from specific types of wood chips during aging or as a result of transesterification.

For example, methyl pentanoate, characterized by its sweet, ripe, fruity aroma with dairy and cheesy nuances (Burdock, 2010), was found in brandies aged with cherry wood chips. Additional esters that appeared in spirits as a result of aging with wood chips include ethyl 2-methylbutanoate, ethyl 3-methylbutanoate, ethyl hexanoate, and ethyl 3-hexenoate (Table 1). Similar findings by Balcerek et al. indicated that concentrations of ethyl esters (ethyl hexanoate, ethyl octanoate, and ethyl decanoate) in plum brandies aged with wood chips gradually increased over storage. The migration of compounds from wood chips to brandies is evidenced by the significant increase in the concentration of ethyl butanoate (with a fruity, pineapple aroma) (Burdock, 2010) in brandies aged with these types of chips. Some esters originate from precursors extracted from wood during aging, leading to significant variations in their concentrations in alcoholic beverages. An illustrative example is ethyl furoate, whose content was markedly higher in samples aged with medium roasted and heavily roasted chips (200.9 mg/L and 254.4 mg/L, respectively) compared to the control sample (133.4 mg/L). The concentration of ethyl furoate in brandies significantly depends on the degree of wood toasting and increases with longer maturation times (Tarko et al., 2023).

Ethyl acetate was found to be one of the predominant esters in the apple brandies analysed. The concentration of ethyl acetate increased the most with ageing. Valcárcel-Muñoz et al. (2021) showed that wood releases acetic acid into the distillate during ageing, which subsequently increases the amount of ethyl acetate in the brandy.

Apple brandies aged with wood chips contained a variety of other esters such as pentyl propanoate, isopentyl hexanoate and 2-phenylethyl propanoate (Table 1).

Table 1. Aroma composition of apple brandies aged with different types of wood chips [mg/L 100°]

	LRI*	Apple brandies aged without the addition of wood chips	Apple brandies aged with pear wood chips	Apple brandies aged with cherry wood chips	Apple brandies aged with walnut wood chips	Apple brandies aged with medium roasted oak wood chips	Apple brandies aged with heavily roasted oak wood chips	Significance	Method
Methyl and ethyl esters									
Isopropyl acetate	662	0.5b	4.0a	0.4b	0.8b	1.8b	0.5b	***	GC-MS
n-Ethyl propanoate	678	36.3b	8.3cd	73.0a	9.2cd	26.6b	16.8c	**	GC-MS
Ethyl butanoate	789	15.4c	14.2c	26.7bc	18.9c	40.2b	88.1a	***	GC-MS
Methyl pentanoate	823	0.0b	0.0b	4.1a	0.0b	0.0b	0.0b	***	GC-FID
Ethyl 2-methylbutanoate	854	0.0b	0.0b	3.8a	0.0b	3.0a	4.6a	***	GC-MS
Ethyl 3-methylbutanoate	857	0.0b	0.0b	4.0a	0.0b	2.9a	2.9a	***	GC-MS
Ethyl hexanoate	986	0.0c	21.6a	10.2b	12.2b	25.0a	25.2a	***	GC-FID
Ethyl decanoate	902	8.1bc	40.0a	42.2a	5.3c	13.2b	19.2b	**	GC-FID
Ethyl 3-hexenoate	997	0.0c	5.0a	1.4b	0.0c	5.6a	5.8a	***	GC-MS
Ethyl furoate	1029	133.4bc	177.7b	219.6a	155.8b	200.9a	254.4a	***	GC-MS
Ethyl 2-hydroxy-4-methylpentanoate	1060	1.9b	2.0b	4.9a	9.7a	1.8b	0.0c	**	GC-MS
Diethyl butanedioate	1149	0.0c	0.0c	0.0c	0.0c	5.3a	2.3b	***	GC-FID
Ethyl octanoate	1180	64.3a	76.0a	18.8c	10.1c	53.1b	63.9a	**	GC-FID
Ethyl 9-decenoate	1382	2.8b	3.3b	2.4bc	6.9a	3.2b	5.9a	***	GC-MS
Ethyl 3-methylbutyl butanoate	1421	2.0a	0.6b	2.4a	3.0a	2.0a	2.9a	***	GC-MS
Methyl tetradecanoate	1707	2.4a	0.0b	0.0b	0.0b	1.7a	0.0b	***	GC-MS
Ethyl tetradecanoate	1790	14.5b	19.2a	11.6b	18.9a	23.5a	13.5b	***	GC-MS
Ethyl dodecanoate	1803	3.2a	0.0b	0.0b	0.0b	1.9ab	4.3a	***	GC-FID
Ethyl pentadecanoate	1880	2.1ab	1.9b	2.5a	4.9a	2.4a	0.7b	***	GC-MS
Methyl hexadecanoate	1927	3.4a	0.0c	0.4c	1.7b	3.2a	0.0c	***	GC-MS
Ethyl E-11-Hexadecanoate	1974	1.0b	1.0b	1.2b	2.5a	0.9b	0.0c	***	GC-MS
Ethyl hexadecanoate	1990	31.8a	41.2a	30.4a	27.8b	37.5a	35.1a	***	GC-MS
Acetate esters									
Ethyl acetate	614	116.9a	23.5bc	34.1b	46.5b	54.7b	111.0a	**	GC-FID
Isobutyl acetate	763	292.7a	18.7b	6.8b	23.2b	10.0b	13.4b	*	GC-MS
Butyl acetate	799	0.0b	4.3ab	7.6ab	3.6ab	5.5ab	11.0a	***	GC-MS
Isoamyl acetate	876	37.1ab	47.1ab	69.1a	67.1a	70.2a	68.0a	***	GC-FID
3-Hexen-1-ol acetate	999	0.0b	155.9a	100.2a	88.0ab	207.3a	356.8a	**	GC-MS
Hexyl acetate	1006	33.7c	1083.1ab	694.7b	533.7b	1328.0ab	2858.2a	**	GC-MS
Phenylmethyl acetate	1131	0.0c	11.4a	3.9b	5.7a	2.1b	0.0c	***	GC-MS
2-phenylethyl acetate	1228	56.1b	14.1c	75.1b	115.2a	101.1a	147.1a	**	GC-FID
Butoxyethoxyethyl acetate	1354	0.0c	0.0c	5.1a	6.9a	3.4a	1.8b	***	GC-MS
Verdyl acetate	1406	5.6b	8.2ab	6.9ab	7.1ab	10.6a	11.5a	***	GC-MS
Benzoates									
Ethyl benzoate	1142	11.4b	27.6b	12.9b	12.2b	197.2a	113.2a	**	GC-MS

Other esters									
Pentyl propanoate	958	0.0c	1.7c	3.0b	2.7b	3.8b	11.4a	***	GC-MS
Isopentyl hexanoate	1238	1.9c	2.7bc	1.4c	2.4bc	5.9b	11.4a	***	GC-MS
2-phenylethyl propanoate	1328	0.0c	9.0b	6.4bc	6.2bc	9.1b	14.1a	***	GC-MS
Isobutyl octanoate	1351	2.0a	2.5a	0.6b	0.5b	1.8a	3.3a	***	GC-MS
2-phenylethyl-2-methyl propanoate	1385	3.5b	11.9a	6.0b	5.7b	10.4a	19.1a	***	GC-MS
Propyl nonanoate	1388	3.7a	3.0a	0.8b	0.5b	3.3a	6.4a	***	GC-MS
á-Phenylethyl butanoate	1431	7.0a	6.2a	4.5b	3.5b	7.3a	10.8a	***	GC-MS
3-methylbutyl octanoate	1445	28.4	48.5a	5.1b	24.7a	13.7b	38.8a	**	GC-MS
2-methylbutyl octanoate	1449	4.5	5.6a	1.4bc	2.5b	4.0a	6.1a	***	GC-MS
2-Phenylethyl 2-methylbutanoate	1466	2.5a	2.9a	1.3b	1.1b	3.1a	3.0a	***	GC-MS
3-methylbutyl nonanoate	1533	2.8c	16.1a	4.5c	8.5b	20.1a	7.4b	***	GC-MS
Isobutyl decanoate	1546	2.0ab	0.0c	0.8b	0.9b	2.1ab	3.4a	***	GC-MS
2-phenylethyl hexanoate	1611	89.7ab	86.1ab	48.9b	55.2b	110.2a	155.1a	***	GC-MS
1-methylethyl dodecanoate	1617	15.2b	20.1ab	11.0bc	17.9b	46.9a	37.2a	**	GC-MS
3-methylbutyl pentanoate	1641	16.4b	46.9a	16.9b	13.2b	42.7a	41.6a	***	GC-MS
2-methylbutyl pentanoate	1650	0.0c	2.5b	3.0ab	2.1b	3.0ab	10.6a	***	GC-MS
2-phenylethyl octanoate	1820	5.3a	2.2b	3.4ab	0.5b	0.0c	6.4a	***	GC-MS
Isopentyl dodecanoate	1844	1.0ab	1.1ab	0.7b	2.3a	2.3a	1.2ab	***	GC-MS
Alcohols									
Methanol	381	2596.0a	2069.0ab	2917.0a	2136.0ab	2013.0ab	2000.0b	***	GC-FID
Isobutanol	617	162.0a	178.0a	129.0b	182.0a	176.0a	147.0ab	***	GC-FID
4-methyl-1-pentanol	838	9.9b	13.4b	18.1b	32.5a	15.9b	16.1b	***	GC-MS
3-methyl-1-pentanol	850	99.3b	129.0b	185.0b	319.6a	154.7b	149.1b	**	GC-MS
3-Hexen-1-ol	863	753.7b	862.9b	1354.5a	832.1b	1025.9a	995.4b	***	GC-MS
Hexanol	865	13.6ab	18.6a	19.2a	16.6ab	15.3ab	18.4a	***	GC-FID
2-Heptanol	900	5.5b	5.8b	9.8ab	12.2a	11.5a	14.7a	***	GC-MS
1-Heptanol	960	5.7ab	1.9b	6.0a	7.5a	2.2b	8.4a	***	GC-MS
2-ethyl-1-hexanol	1017	43.2ab	54.9ab	42.7ab	35.6b	73.6a	78.0a	***	GC-MS
1-Octanol	1070	38.1ab	32.0ab	36.4ab	32.1ab	54.1a	55.9a	***	GC-MS
Phenylethanol	1102	640.4b	332.2c	1346.3ab	2406.9a	768.9b	1277.6ab	**	GC-MS
1-Decanol	1272	6.8a	8.3a	0.0c	4.2b	2.4b	9.0a	***	GC-MS
1-Undecanol	1364	42.3b	74.3a	4.7c	2.4c	2.0c	44.4b	***	GC-MS
1-Dodecanol	1460	10.9b	18.5a	13.3ab	12.1ab	18.6a	21.3a	***	GC-MS
1-Tetradecanol	1664	0.0c	0.0c	23.4a	0.8b	0.8b	31.2a	**	GC-MS
Aldehydes and ketones									
Furfural	815	151.1c	110.3c	465.6b	1203.8a	166.5c	137.0c	**	GC-MS
Benzaldehyde	924	159.8b	231.9a	273.8a	102.0b	338.1a	289.4a	***	GC-MS
5-methyl-2-Furancarboxaldehyde	942	0.0b	0.0b	46.0ab	0.0b	0.0b	100.1a	**	GC-MS
Benzeneacetaldehyde	1015	0.0c	13.3a	17.8a	7.0b	12.8a	22.1a	**	GC-MS

Acetophenone	1044	43.5bc	51.5b	37.1c	64.1b	371.0a	69.5b	**	GC-MS
Nonanal	1083	12.2ab	6.6b	19.5a	33.2a	32.1a	27.5a	***	GC-MS
Decanal	1182	5.5a	5.2a	2.9ab	2.4	6.5a	3.5ab	***	GC-MS
Vanillin	1378	0.0b	0.0b	0.1b	0.0b	0.3a	0.1b	***	GC-MS
Dodecanal	1401	3.1a	2.9b	2.1ab	2.2ab	3.1a	2.2ab	***	GC-MS
Terpenoids									
3-Carene	1011	2.0b	5.8a	3.9ab	1.4b	3.8ab	9.4a	***	GC-MS
o-Cymene	1014	5.2b	15.1ab	9.1b	27.1a	13.1ab	22.5a	***	GC-MS
Limonene	1020	0.0b	0.4a	0.3ab	0.4a	0.6a	0.6a	***	GC-FID
cis-Linaloloxide	1075	9.1b	10.2ab	14.3a	18.9a	14.4a	14.2a	***	GC-MS
Trans-Linalool oxide	1078	1.1ab	2.0a	2.2a	2.0a	0.5b	2.3a	***	GC-FID
Linalool	1094	0.0b	0.6a	0.2ab	0.0b	0.3a	0.4a	***	GC-FID
Guaiacol	1095	0.0b	0.0b	0.3a	0.0b	0.0b	0.4a	***	GC-FID
α-Terpineol	1171	18.0bc	62.3b	57.3b	142.1a	68.5b	9.9c	**	GC-MS
Terpinen-4-ol	1181	0.1ab	0.2a	0.1a	0.1ab	0.2a	0.2a	***	GC-FID
β-Cyclocitral	1194	3.9b	4.7a	2.8b	2.5b	6.0a	7.7a	***	GC-MS
α-Ionene	1215	2.0ab	3.2a	1.9ab	1.5b	2.3a	5.1a	***	GC-MS
ß-citronellol	1229	0.0b	0.1b	0.1b	0.4a	0.1b	0.1b	***	GC-FID
Geraniol	1258	0.0b	0.0b	0.0b	0.2a	0.0b	0.1a	***	GC-FID
Citral	1240	0.0c	0.2a	0.3a	0.1b	0.1b	0.1b	***	GC-FID
trans-Edulan	1309	2.8b	3.8b	2.1bc	1.3c	3.8b	5.8a	***	GC-MS
Eugenol	1326	0.0c	0.9a	0.0c	0.0c	1.5a	0.4b	***	GC-FID
β-Damascenone	1357	3.3c	7.2b	10.3a	9.6a	7.2b	8.1a	***	GC-MS
Methyleugenol	1408	0.8a	1.3a	1.2a	0.1b	1.4a	1.3a	***	GC-FID
trans-Geranylacetone	1443	1.8c	6.6b	29.4a	49.0a	33.8a	28.9a	**	GC-MS
β-Famesene	1458	10.6a	10.6a	5.0b	4.4b	6.1b	7.0b	***	GC-MS
β-Farnesene	1480	7.0a	2.3b	1.6b	1.9b	1.9b	2.3b	***	GC-MS
β-Ionone	1490	1.1b	0.1c	4.3a	0.0c	2.8a	0.0c	***	GC-FID
ŕ-Farnesene	1494	3.4a	2.8a	2.4a	1.6ab	2.6a	0.0c	***	GC-MS
Nerolidol	1552	29.7b	45.1a	29.8b	16.7c	38.0b	47.3a	***	GC-MS
α-Copaen-11-ol	1572	0.0c	0.4ab	0.6ab	0.0c	0.5ab	1.7a	***	GC-MS
2,3-Dihydrofarnesol	1696	7.8a	9.9a	7.0b	3.9b	7.8a	8.0a	***	GC-MS
Farnesol	1702	31.4a	12.0b	14.3b	7.9bc	14.9b	12.3b	***	GC-MS
Farnesal	1730	1.1a	0.8a	0.0b	0.0b	0.0b	0.0b	***	GC-MS
Other compounds									
1,1-diethoxy-ethane	720	0.0c	208.6a	91.6b	30.5b	106.6b	313.7a	*	GC-MS
1,1-diethoxy-butane	872	6.4bc	20.1ab	21.6ab	12.9b	17.9b	71.6a	**	GC-MS
1,1-diethoxy-3-methyl-butane	952	2.0b	3.5b	4.2b	4.1b	3.5b	24.4a	**	GC-MS
1-(1-ethoxyethoxy)-pentane	966	22.2bc	206.7s	57.0b	50.3b	78.9b	231.6s	**	GC-MS
2-pentyl-furan	981	21.2a	14.5a	5.9b	8.5b	6.0b	18.9a	***	GC-MS
1-(1-ethoxyethoxy)-hexane	1104	0.0c	6.0b	0.0c	2.9b	4.7b	11.1a	***	GC-MS
n-Decanoic acid	1360	7.0c	0.0c	70.1b	14.9c	186.3a	0.5c	**	GC-MS
1,1'-oxybis-octane	1657	33.2a	1.6c	6.3b	0.5c	2.7b	0.0d	**	GC-MS

*, **, *** Display the significance at 5, 1 and 0.1 % by least significant difference at $p < 0.05$, $n = 5$, LRI*—linear retention index; the amount of components was determined semi-quantitatively by measuring the relative peak area of each identified compound, according to the NIST (National Institute of Standards and Technology) database, in relation to that of the internal standard.

It is generally recognized that the intensity of fruity and floral aromas diminishes as maturation progresses, due to the chemical modifications that decrease the concentration of esters. These esters gradually hydrolyse into alcohols and acids until a chemical equilibrium is reached. However, we observed a tendency for the addition of wood to increase the concentration of esters (compared to samples aged without chips). This phenomenon may be related to the increased concentration of polyphenols, such as ellagitannins, which are antioxidants. These substances may protect the esters from oxidative degradation (Kyraleou et al., 2016). As a result, the flavour of brandies is enhanced, as esters contribute to fruity, buttery or creamy flavours, while also contributing to the sensation of roundness in the mouth (Stegăruș at al., 2021). Among the 28 detected higher alcohols, aldehydes, and ketones, 25 were present in all analyzed apple brandies. Only 1-tetradecanol, 5-methyl-2-furancarboxaldehyde, and benzen-eacetaldehyde could have originate from wood chips, especially those that are roasted. The compound 1-tetradecanol may contribute a slight aroma of cooked beef (Burdock, 2010). 5-Methyl-2-furancarboxaldehyde, which offers almond, burnt sugar, cream, and caramel notes, was found in brandies aged with roasted chips. This compound is typically formed under thermal conditions. During the roasting process, three chemical reactions play crucial roles in aroma compound formation: Maillard reactions, lipid degradation, and sugar caramelization. The Maillard reaction often yields nitrogen heterocyclic compounds, such as pyrazines, furans, and aliphatic aldehydes (Stegăruș et al., 2021). Furthermore, caramelization of sugars produces low molecular weight, open-chain oxygen-containing products and heterocyclic oxygen-containing compounds, including furan derivatives (Stegăruș et al., 2021). These compounds impart caramel, vanilla, buttery, burnt sugar, smoky, and medicinal aromas (Burdock, 2010). As indicated in Table 1, brandies aged with roasted chips exhibited higher concentrations of most higher alcohols. The thermal degradation of lipids can produce oxygen-containing compounds, including alcohols, ketones, and aldehydes (Stegăruș et al., 2021). Furfural is one of the most significant compounds originating from wood, responsible for imparting dried fruit and roasted almond aromas. The highest concentration of this compound was observed in brandies aged with walnut chips. Bautista-Ortín et al. have demonstrated that the use of oak wood during aging increases the concentration of furfural. This increase is sustained for up to 6 months of aging, after which it begins to decrease, that could be associated with the biological conversion of furfural into corresponding alcohols (Stegăruș et al., 2021).

Methanol is the most abundant major volatile compound in apple brandy. As a typical component of many fruit distillates, its presence is considered a marker of authenticity. Apples, being a significant source of pectin (ranging from 11.6 to 32.6 g/kg fresh mass), can lead to higher concentrations of methanol in apple brandies (Coldea et al., 2020). The maximum permitted level of methanol in apple brandy is set at 12 g/L of 100° ethanol. However, our experiments showed methanol concentrations ranging from 2000 mg/L to 2917 mg/L of 100° ethanol, which is well below the limit. Similar results have been reported in other studies. For example, Croitoru et al. (2013) found methanol concentrations of about 0.5% in apple brandy and about 1% in apple-plum brandy, and Coldea et al. (2020) reported concentrations of up to 10,000 mg/L in apple brandy. As methanol has a boiling point of 64.7°C, a significant amount of this compound is distilled off in the head fraction during production.

The last significant group of compounds identified in the brandies were terpenoids. We observed that over 80% of terpenes were present in samples aged with roasted wood chips. Guo et al. (2019) showed that the concentration of terpenoids increases with higher roasting temperatures and longer roasting times. It is widely recognised that under conditions of elevated temperature and prolonged time, volatile compounds are formed by the Maillard reaction. Selecting the correct roasting temperature is therefore critical, as too low a temperature will not produce volatile compounds, while too high a temperature may destroy them. During the toasting process, compounds derived from lignin, such as guaiacol and eugenol, increase in concentration with temperature. Călugăr et al. (2020) reported a significant increase in the levels of these compounds in Muscat Ottonel wine after ageing, noting a significantly higher concentration in samples aged with wood chips compared to those aged in barrels.

Some terpenes, such as citral, limonene, geraniol, β-citronellol, linalool and α-copaene-11-ol, were found exclusively in wood-chip aged brandies. Many of these compounds either originate directly from the wood chips or are transformed from one form to another. For example, citral can be converted to geraniol and linalool or further metabolized to citronellol by yeast enzymes (Guo et al., 2019; Esmaeili and Tavassoli, 2010; Pardo et al., 2015). In addition, linalool can be converted to geraniol in the presence of water, a reversible process. Geraniol serves as a precursor for the production of a variety of volatile compounds, including citronellol, linalool, nerol, citronellyl acetate and geranyl acetate (Esmaeili and Tavassoli, 2010).

Sensory evaluation and aroma-active compounds in apple brandies

Brandies aged with heavily and medium roasted chips scored high (4.5 points, see Figures 1-2), possibly due to the presence of a variety of aromas associated with roasting and maturation processes, such as woody, coconut, burnt sugar and buttery notes. These aromas effectively masked the pungent and medicinal aromas found in non-chip aged brandies, which were negatively correlated with overall sensory quality. These quality-reducing aromas were mainly found in spirits aged with the highest concentration of heavily roasted wood chips. The highest ratings were given to spirits aged with wood chips added at the highest concentrations, i.e., 8 g/L and 12 g/L. Toasting wood leads to the formation of new aromatic compounds, such as vanillin, eugenol, 4-ethylguaiacol, 4-ethylphenol, 4-vinylguaiacol, and acetovanillin, which give spirits their characteristic vanilla, spicy, smoky, and caramel notes (Tarko et al., 2023). These compounds are formed as a result of the decomposition of lignin, hemicellulose, and other wood components during thermal processing (Tarko et al., 2023). The results presented in Table 2 confirm the aromatic activity of eugenol and vanillin in samples aged with the addition of toasted wood chips. The level of roasting affects the proportions of individual compounds – a higher degree of roasting promotes the formation of more intense vanilla and smoky aromas, while a lower degree promotes subtler, woody, and fruity aromas (Tarko et al., 2023). The distinctive burnt sugar aroma identified in brandies aged with roasted oak chips can be attributed to the formation of furan and pyran derivatives during the wood burning process involved in barrel making. These compounds, such as 5-methyl-2-furancarboxaldehyde (see Table 1), are known for their toasty caramel aroma (De Rosso et al., 2009). In addition, the pungent aroma observed in some brandies could be related to the relatively high presence of fusel alcohols in the main distillation fraction, alongside significant concentrations of other compounds such as ethyl acetate and eugenol. Eugenol in particular imparts a clove aroma which can contribute to a pungent and burning flavour (Burdock, 2010). Spirits aged with walnut chips also scored above 4 points (Figure 3), especially those aged with chips at a concentration of 12g/L. The spirits were characterized by a sweet, apple aroma with scents of woody, smoked and citrus. The citrus aroma could be related to the high level of a-terpineol detected in this sample. In turn, the high concentration of phenylethanol could have given the sample a pleasant, sweet aroma.

Brandies aged with fruit chips also were highly rated for their overall sensory quality, with scores 4.5 for those aged with cherry chips and 4 for those aged with pear chips (see Figures 4-5). The aroma profile of these brandies was generally described as pleasant, sweet, floral, with notes of cloves, citrus and a strong apple character. This high sensory rating is probably due to the diverse volatile compound profiles of the cherry and pear chips aged brandies, with terpenoids in particular contributing significantly to these results. For example, guaiacol and eugenol, which were identified exclusively in the cherry and toasted oak chips aged brandies, imparted a distinct clove aroma, as confirmed by the sensory analysis. However, olfactometric analysis revealed that only eugenol showed significant aroma activity in samples aged with roasted chips (Table 2). Weixi et al. (2023) suggested that eugenol may be an important aroma-active compound contributing to the smoky aroma. Literature, including studies by Ribéreau-Gayon et al. (2006), reported a higher concentration of eugenol and guaiacol in wines aged in heavily roasted barrels compared to those aged in medium or lightly roasted barrels, suggesting that these volatiles can be transferred from oak to alcoholic beverages during barrel aging. The composition reflects the lignin structure of the oak and the heating temperature applied. Guaiacol is also associated with smoky, phenolic and medicinal notes, in addition to its clove aroma, which correlates with the high degree of oak roasting. The significant presence of eugenol in samples aged with roasted chips can be attributed to the thermohydrolysis of lignins and the formation of their precursors. Limonene probably contributed to the citrus aroma, which was more pronounced in samples aged with wood chips than in control samples without wood. This observation was supported by olfactometric analysis, where the aromatic activity of limonene was only detected in samples aged with wood chips (Table 2). Research by Ndao and Adjallé (2023) confirms that limonene can be found in wood residues and citrus fruits. Research by Guerrero-Chanivet et al. (2020) also confirmed the presence of limonene in wood chips from American oak, Spanish oak, French oak, chestnut and cherry. In comparison, brandies aged without wood chips had less pronounced aromas and a significantly lower total concentration of volatile compounds than those aged with wood chips.

Table 2. Heatmap of aroma-active compounds intensities detected by GC-O in apple brandies aged with different types of wood chips.

	Apple brandies aged without the addition of wood chips	Apple brandies aged with pear wood chips	Apple brandies aged with cherry wood chips	Apple brandies aged with walnut wood chips	Apple brandies aged with medium roasted oak wood chips	Apple brandies aged with heavily roasted oak wood chips	GC-O descriptors*
Ethyl acetate	2,5	0,5	1,3	1	2,5	3	floral, solvent
Ethyl 3-methylbutanoate	0	0	0,5	0	0	0	Apples
Ethyl propanoate	1,5	1	1	1	1,8	1	fruity, pineapple
Isobutyl acetate	2	1	1	1	1	1	fruity, apple, banana
1-Butanol, 3-methyl-, acetate	0	0	0	0	0	0,5	fruity, apple
1-Butanol, 2-methyl-, acetate	0	0	0	0	0	0,5	fruity, banana, pear, sweet
Ethyl butanoate	1	1	1	1	1,8	2	pineapple, sweet, fruity
Ethyl hexanoate	0	1,3	1,8	1	1	1	fruity, red apple
2-phenylethyl acetate	0,5	0,5	0	1	0	1	rose, honey
Ethyl octanoate	0	0,5	0	0,5	0	0	sweet, fruity, winey
Diethyl butanedioate	0	0	0	0	1,8	2	fruity, waxy, floral, slightly musty
Phenylethyl acetate	1	0,5	1	1,5	1,5	2	floral, rose, honey, raspberry
Isopentyl hexanoate	0,5	0,5	0,5	0,5	0	1	fruity
Phenethyl hexanoate	1	1	1	1	1,5	1,5	fruity, pineapple
Limonene	0	1	1	1	1	1,3	lemon, citrus
Trans- linalool oxide	1	1,5	1,5	1,5	1	1	sweet, earthy, floral, lavender, herbal
α-terpineol	1	1,8	1,5	2	1,5	1	lilac, peach
Eugenol	0	0	0	0	1,5	1,5	clove
Vanillin	0	0	0,5	0	0,5	0,5	sweet, vanilla
Acetal	0	0	0,5	0	0,5	0	green, woody
1-Butanol, 3-methyl-	0	0	0,5	0,5	0,5	0,5	bready, alcoholic, and fruity
2-methyl-1-propanol	0	0,5	0,5	0,5	0,5	0	ethereal
Hexanol	1	1	1	1	1	1	herbal, grassy, sweet
Octanol	0,5	0	0	0	0	0	orange, rose
Phenyethanol	1	1	1,5	2	1	1,5	rose, sweet, peach
Decanol	0	0	0	0	1	1	fruity
Nonanal	0	0	0	1,5	0	0	orange, rose
Benzaldehyde	0,5	1	1	0,5	1,3	1,3	almond
Acetophenone	0	0	0,5	0	0	0	sweet, medicinal

The lowest intensity of aroma in column is in the darkest red and the highest is in the darkest green. SD < 5%, *Burdock (2010).

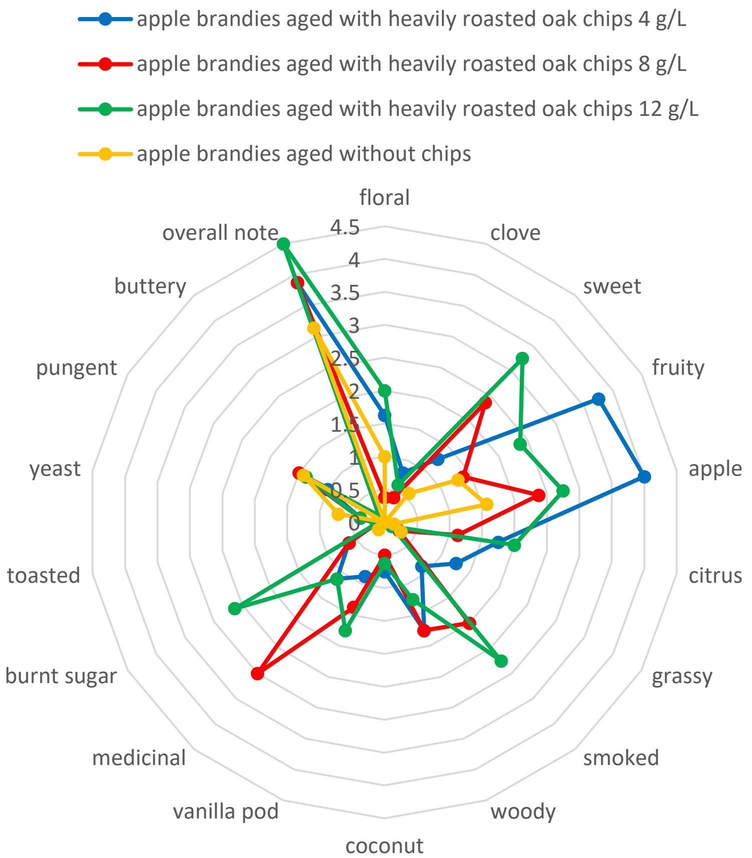

Figure 1. Characteristic aroma traits of apple brandies aged with heavily roasted oak wood chips, n = 5, STD < 5

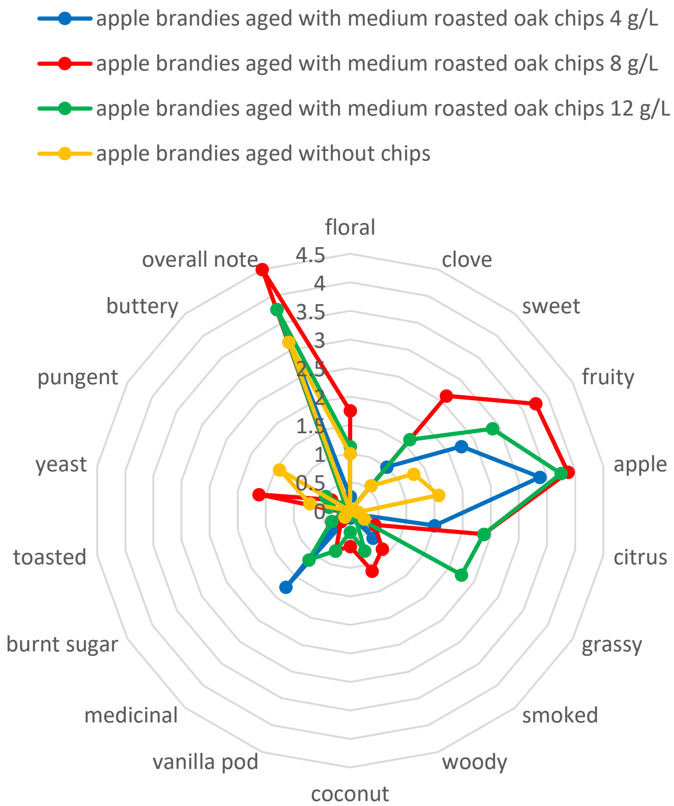

Figure 2. Characteristic aroma traits of apple brandies aged with medium roasted oak wood chips, n = 5, STD< 5

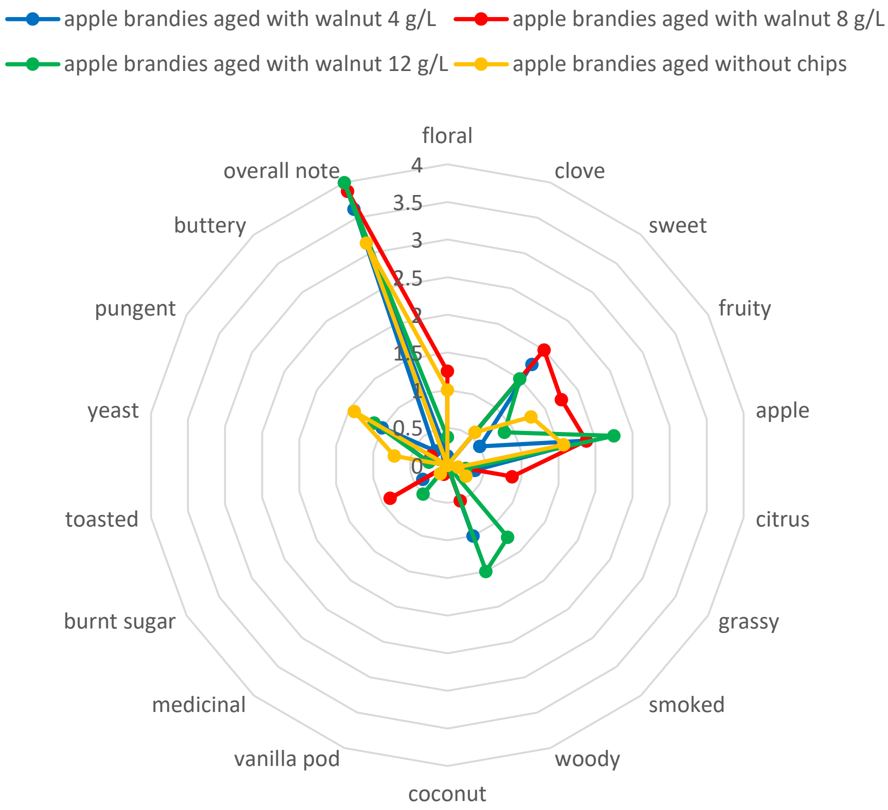

Figure 3. Characteristic aroma traits of apple brandies aged with walnut wood chips, n = 5, STD < 5

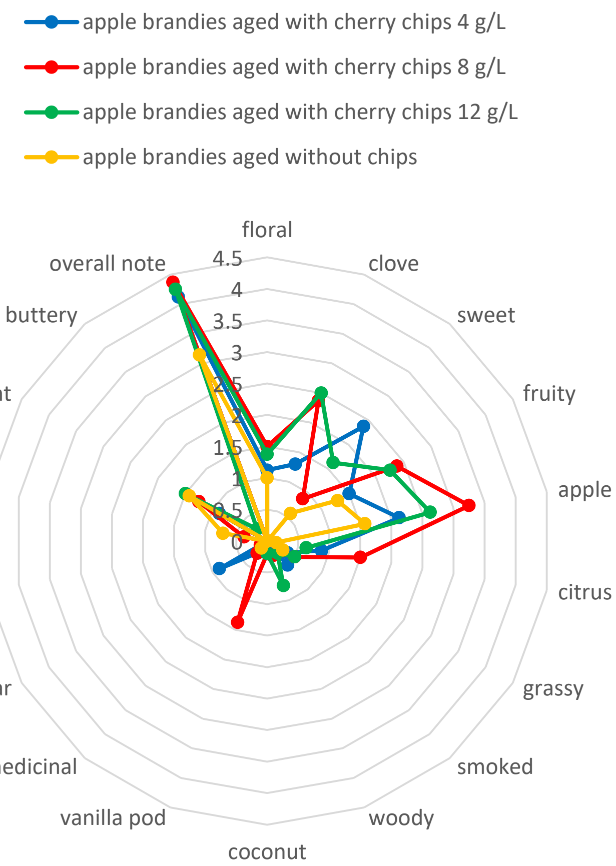

Figure 4. Characteristic aroma traits of apple brandies aged with cherry wood chips, n = 5, STD < 5

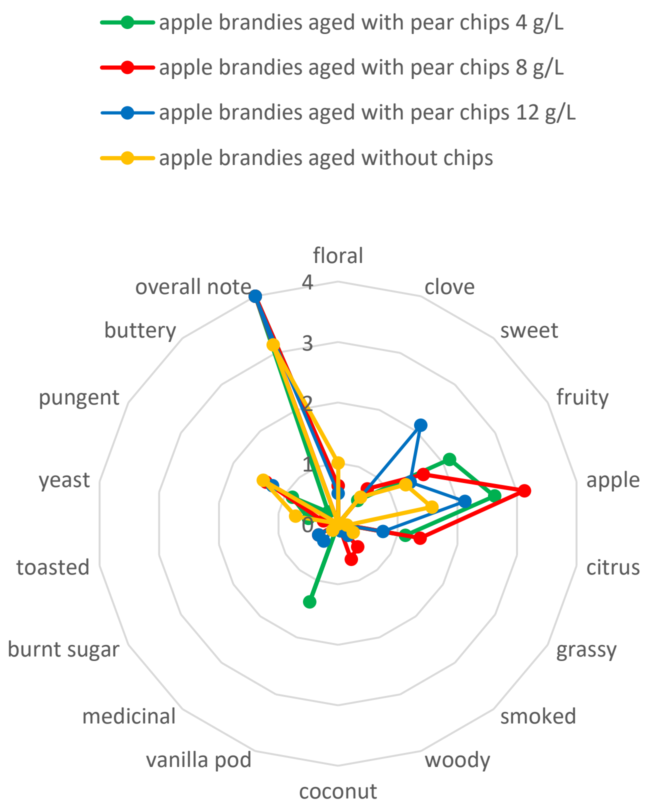

Figure 5. Characteristic aroma traits of apple brandies aged with pear wood chips, n = 5, STD < 5

Olfactometric analysis also revealed fewer active aroma compounds in samples aged without wood chips. Esters such as 2-phenylethyl acetate, responsible for light floral aromas, tend to decompose or undergo interesterification during ageing, resulting in reduced concentrations (Tarko et al., 2023). However, in our study a higher aromatic activity of 2-phenylethyl acetate and phenylethanol (rose aroma) was observed in samples aged with roasted wood chips, confirmed by chromatographic analysis and resulting in higher floral scent ratings by panelists (Table 1 and 2).

Statistical analysis using Pearson's test revealed positive correlations between the overall sensory rating of the brandies and a number of aromatic descriptors including floral, clove, sweet, fruity, apple and citrus. Conversely, negative correlations were found between overall sensory quality and certain descriptors such as pungent, medicinal, yeasty and buttery aromas.

The results presented indicate that the concentration of added wood chips is also important. The addition of wood chips did not mask the aromas derived from the raw material, as apple and fruit aromas were rated highest in all samples (see Figures 1- 5).

All the samples received the maximum scores for clarity. The colour of the brandies ranged from straw to brown (see Figure 6 and Table 3). The exception was the control sample which was transparent (Figure 6). Brandies aged with walnut chips and heavily roasted oak chips were found to have the most appealing colour, suggesting a potential consumer preference for drinks with a more intense colour. In contrast, colourless control samples received the lowest ratings.

Table 3. Colour of apple brandies aged with different types of wood chips

	CIE L	CIE a*	CIE b*	Sample color
Apple brandies aged with heavily roasted oak wood chips	34,8	1,5	0,9	Mortar Grey
Apple brandies aged with medium roasted oak wood chips	29,2	3,1	22,8	Irish Coffee
Apple brandies aged with cherry wood chips	25,4	18,8	51,8	Baker's Chocolate
Apple brandies aged with pear wood chips	25,1	7,1	26,5	Brown Bramble
Apple brandies aged with walnut wood chips	26,4	12,2	24,2	Baker's Chocolate

Figure 6. The color of apple brandies aged with different types of wood chips. a) Apple brandies aged without wood chips; b) Apple brandies aged with heavily roasted oak wood chips; c) Apple brandies aged with medium roasted oak wood chips; d) Apple brandies aged with cherry wood chips; e) Apple brandies aged with pear wood chips; f) Apple brandies aged with walnut wood chips

Lightness (L*) values were highest for brandies aged with heavily roasted oak chips (Table 3), followed by those aged with moderately roasted oak chips, and were similar for the remaining samples. The highest values of the a* parameter were observed in brandies aged with cherry and walnut chips. The positive value of this parameter is associated with reddish tones, while a negative value indicates green tones. The oxidation of anthocyanins during ageing in oak barrels or with wood chips, in combination with oak tannins, increases colour stability and imparts red-purple tones (Śliwińska et al., 2016; Jordão et al., 2019). Interestingly, the values of a* and b* parameters were lowest in samples aged with heavily roasted oak chips. Aging with oak wood also plays a crucial role in the formation of 'new' pigments that could enhance and maintain colour intensity for longer periods. For example, the formation of several oligomeric and polymeric pigments results from reactions between malvidin-3-monoglucoside and (+)-catechin mediated by oak-derived compounds such as furfural, methylfurfural, vanillin, ellagic acid and ellagitannins (Jordão et al., 2019). While the majority of publications on the effect of wood on the colour of alcoholic beverages primarily discuss the use of oak wood species, less attention has been paid to other non-oak wood species such as cherry, pear and walnut. However, research by Jordão et al. (2019) has considered cherry wood chips as a potential source of wood for ageing.

CONCLUSIONS

Our research has shown that the use of wood chips to age spirits can effectively replace the traditional barrel aging process, resulting in beverages with interesting flavours. By using different types of wood chips, we were able to produce significantly different spirits in terms of their volatile profiles and sensory characteristics, allowing the flavours of the brandies to be customised during maturation. Of the five types of chips tested, brandies aged with cherry and heavily roasted oak chips showed the most varied volatile compound profiles.

The analysis identified 24 compounds in the methyl and ethyl esters, of which 23 were found in brandies aged with medium roasted oak chips and 21 in those aged with cherry chips. Certain compounds were found in all the brandies (e.g. isopropyl acetate, ethyl propanoate), while others, such as methyl pentanoate, appeared to result from ageing with specific types of wood. Brandies aged with roasted chips showed higher concentrations of higher alcohols such as 1-tetradecanol, 5-methyl-2-furancarboxaldehyde and ben-zene-acetaldehyde, probably due to thermal degradation of lipids producing aldehydes, ketones and alcohols. In addition, 80% of all identified terpenes were most concentrated in samples aged with roasted wood chips. The temperature during chip roasting is crucial - too low a temperature prevents the formation of volatile compounds, while too high a temperature can destroy flavour compounds. The sensory analysis gave the highest overall scores to the cherry and heavily roasted oak chip aged brandies. The aroma of cherry-aged brandies was described as pleasant, sweet, floral, clove, citrus and intense apple. In contrast, the aroma of the roasted oak chip casks was associated with roasting and maturation processes, including woody, coconut, burnt sugar and buttery notes. The Pearson test again showed positive correlations between overall scores and descriptors such as floral, clove, sweet, fruity, apple and citrus. However, negative correlations were also observed between overall ratings and pungent, medicinal, yeast and buttery aromas.

Cherry wood chips, known for their high porosity and oxygen permeability, are typically used for short ageing periods. They produced the most varied volatile profile, with over 90% of the compounds analysed present. Samples aged with roasted wood chips contained the most aroma active compounds and received the highest sensory ratings. We plan to extend our research to compare apple brandies aged in barrels with those aged using wood chips and to further investigate the volatile compounds present in the wood chips.

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Conflicts of Interest: The authors declare no conflict of interest

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