# Sensory-directed approach to explore cider typicity: the case of ciders from the Canary Islands (Spain)

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## Abstract

**Purpose** – The main aim of this study is to characterise and identify specific chemo-sensory profiles of ciders from the Canary Islands (Spain).

**Design/methodology/approach** – Commercial samples of Canary ciders were compared to ciders from the Basque Country and Asturias. In total, 18 samples were studied, six for each region. The analysis comprised their sensory profiling and chemical characterisation of their polyphenolic profile, volatile composition, conventional chemical parameters and CIELAB colour coordinates. In parallel, the sensory profile of the samples from the Canary Islands was first compared with their Basque and Asturian counterparts by labelled

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sorting task. Then, their specific aroma profile was characterised by flash profile. Further quantification of sensory-active compounds was performed by GC–MS and GC-FID to identify the volatile compounds involved in their aroma profile.

**Findings** – Results show that Canary ciders present a specific chemical profile characterised by higher levels of ethanol, and hydroxycinnamic acids, mainly t-ferulic, t-coumaric and neochologenic acids, and lower levels of volatile and total acidity than their Asturian and Basque counterparts. They also present a specific aroma profile characterised by fruity aroma, mainly fruit in syrup and confectionary, and sweet flavours related to their highest levels of vinylphenols formed by transformation of hydroxycinnamic acids.

**Originality/value** – An integrated strategy to explore the typicity of the currently existing Canary ciders in the market was developed. The results are important in that they will help other regions to identify specific typical chemo-sensory profiles and to promote the creation of certifications supporting regional typicity.

**Keywords** Typicality, Hydroxycinnamic acids, Vinylphenols, Aroma, Apple

**Paper type** Research paper

## 1. Introduction

Apples (*malus domestica*) and their derivatives, in particular cider, have a substantial role on global fruit cultivation (Fabien-Ouellet and Conner, 2018). Cider is a traditional alcoholic beverage fermented from apples, with consumption and production rapidly increasing worldwide (Sousa *et al.*, 2020). Cider is an original and traditional European Atlantic beverage, the main producer being the United Kingdom followed by France and Spain, with the largest non-European producing country being the United States (Merwin *et al.*, 2008). Spaniards consume 2.5 litres of cider per year, compared to 14 litres in the UK, while in Spain, cider accounts for 3% of the alcoholic beverage market, growing by 3.5% between 2014 and 2019, compared to 0.2% growth for beer in the same period (Calleja, 2021). The definition of cider includes both flavoured products mixing apple with other fruits such as lime or berries as well as artisanal products resulting exclusively from the fermentation of apples, bounded to specific geographic areas and manufactured according to traditional production methods. In the European Union, traditional ciders are protected under the umbrella of Protected Geographical Indications (PGI) and Protected Designations of Origin (PDO). This qualification prevents fraud and guarantees consumers the veracity of producers' claims about their foodstuffs. The demonstration of the typicity of PDO and PGI products often requires the existence of both cultural and biochemical factors that ensure their differential character against other similar products (Cayot, 2007).

In Spain, only the traditional ciders of Asturias and the Basque Country have achieved official PDO recognition in 2005 and 2016 respectively. However, the Spanish cider-making tradition is not unique to these areas. This paper addresses the rather unexplored topic of cider production in the Canary Islands, an archipelago located nearby the Saharan coast in the Atlantic Ocean. Apple cultivation started immediately during the Spanish conquest of the islands in the sixteenth century, and cidermaking is documented already in the late nineteenth century (Viera and Clavijo, 2004). This traditional craft cider did not achieve commercial status until the 1980s, when the first commercial ciders appeared in the market. By 2020, the archipelago boasts 323 hectares of apple trees and nine professional cider producers with an estimated total production of 20,000 litres per year, mainly located in the islands of La Palma, Tenerife, La Gomera, El Hierro and Gran Canaria. Many other producers make cider for self-consumption or are considering becoming official registered cider-makers. The archipelago is considered a hotspot of apple germplasm diversity, presenting at least 55 endemic apple varieties identified through simple sequence repeats or SSR, showing also clear correlations with apple varieties from Galicia (Pereira-Lorenzo *et al.*, 2018). Despite this variety, the main cultivar is the Reinette apple, whose cultivation was promoted by public institutions throughout the twentieth century.

It is well known that the chemical and sensory profile of ciders varies due to different edaphoclimatic and geographical characteristics, sun exposure hours, fermentation microbiology, apple varieties employed, ripening stage, ageing on lees, storage and production methods (Rosend *et al.*, 2019; Villière *et al.*, 2015). Differences between Basque

and Asturian ciders are well established in the literature (Picinelli-Lobo *et al.*, 2016; Antón *et al.*, 2014), and in-depth studies have characterised the ciders from the island of Madeira, located nearby the Canary Islands (Medina *et al.*, 2020; Sousa *et al.*, 2020). Similar studies have established differences between Normandy and Brittany (Haider *et al.*, 2014) or French ciders (Le Quéré *et al.*, 2006). Most studies have focused on the volatile profile of ciders for their differentiation (Perestrelo *et al.*, 2019; Rosend *et al.*, 2019; Picchi *et al.*, 2023), while others focus on phenolic and elemental composition together with their chemical profile (Alonso-Salces *et al.*, 2004; Nicolini *et al.*, 2018; Way *et al.*, 2020; Rodríguez Madrera *et al.*, 2006). More recently, the sensory profile of Virginia ciders has been established and related to consumer preference based on their flavour (Cole *et al.*, 2023) and extrinsic factors (i.e. considering packaging and labels) (Kessinger *et al.*, 2021). These studies have allowed differentiating not only the origin of ciders but also to discriminate them based on harvest conditions (Girschik *et al.*, 2017; Keller *et al.*, 2004). However, no studies have addressed such issues in the Canary Islands yet.

Currently, the Canary Islands lack any trademark or protected designation to differentiate their ciders, although there is a current debate on the topic among both producers and policymakers. This paper aims to identify specific sensory and chemical features characteristic of Canary cider, thus contributing to fill a research gap given the lack of previous research on the topic. It starts from the hypothesis that Canary ciders present unique characteristics differentiating them from their Asturian and Basque counterparts, due to the subtropical climate, volcanic soils and unique combination of Reinette and local varieties. The outcomes of this study will offer novel opportunities to enhance knowledge about Canary ciders and ensure the production of high-quality products, improving the market opportunities of cider-makers and facilitating the market entry of new producers. Moreover, results will be of outstanding importance to promote the certification of Canary ciders under a PDI or PDO scheme and to provide robust data to be used as authenticity markers of the product and create an official tasting panel. To achieve these objectives, this paper sets out to compare Canary ciders with their Basque and Asturian counterparts, including both natural and sparkling ciders. The analysis includes their sensory profiling and chemical characterisation of their polyphenolic profile, volatile composition, conventional chemical parameters and CIELAB coordinates.

## 2. Materials and methods

### *2.1 Cider samples*

Eighteen commercial young ciders produced in different Spanish regions (6 from Canary Island, 6 from Basque Country, 6 from Asturias) were studied (see Table 1). For each region, three natural (N) and three natural sparkling (NS) ciders were selected. The cider selection was based on the presence of six legally operating cider houses in the Canary Islands in total, three of which exclusively produce sparkling ciders. Therefore, ciders with similar production methods and vintages to their Canary counterparts were sought to reduce bias in the comparison. In the case of Asturias and the Basque Country, ciders with Protected Designation of Origin (PDO) were chosen to ensure their origin. All non-sparkling natural ciders from Canary Islands followed the same mechanised production process, starting with apple harvesting, transportation, crushing and pressing, followed by obtaining the must and fermentation in stainless steel barrels and tanks. In the case of Asturian and Canary Island ciders, the cider remains in stainless steel tanks until it is bottled. In the Basque Country, the cider is aged in *kupelas* chestnut casks of different dimensions, for between four to five months before bottling. All sparkling ciders in the sample were produced using the traditional method (i.e. Champenoise method) by carrying out the secondary fermentation in bottle. All the samples had less than 7 g/L of reducing sugars.

**Table 1.**
Conventional chemical parameters of the 18 ciders used for the study from three different Spanish regions: Asturias (A), Basque Country (B) and Canary Islands (C) and two different methods of elaboration: natural (N) and natural sparkling (NS)

| Origin | Code | Elaboration method | Alcohol content | VA | MA | RS | Free $SO_2$ | Total $SO_2$ | pH | TA | a* | b* | L* | C* | h* | CI | Turbidity | TPI | HCA |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Asturias | A_NS_1 | NS | 5.62 | 1.84 | <0.1 | 6.6 | 29 | 126 | 3.71 | 5.0 | 0.0 | 3.9 | 98.3 | 3.9 | 90.5 | 0.485 | 0.37 | 17.28 | 8.20 |
| | A_NS_2 | NS | 6.83 | 1.28 | <0.1 | 1.1 | 8 | 20 | 3.67 | 5.2 | 0.1 | 1.7 | 99.0 | 1.7 | 87.0 | 0.245 | 1.05 | 9.30 | 5.58 |
| | A_NS_3 | NS | 5.98 | 1 | <0.1 | 1.1 | 7 | 62 | 3.81 | 3.9 | −0.3 | 3.4 | 98.9 | 3.4 | 94.9 | 0.365 | 19.02 | 17.58 | 13.45 |
| | A_N_1 | N | 5.68 | 1.2 | <0.1 | 1 | 6 | 20 | 3.8 | 4.9 | −0.2 | 3.4 | 98.4 | 3.4 | 93.6 | 0.440 | 97.45 | 11.28 | 6.38 |
| | A_N_2 | N | 7.52 | 1.56 | <0.1 | 6.2 | 15 | 86 | 3.64 | 5.9 | −0.1 | 2.5 | 98.9 | 2.5 | 93.1 | 0.315 | 0.09 | 11.45 | 5.83 |
| | A_N_3 | N | 5.61 | 1.9 | <0.1 | 1.1 | 7 | 42 | 3.84 | 4.5 | −0.2 | 2.5 | 98.9 | 2.5 | 93.4 | 0.310 | 101.93 | 8.13 | 4.58 |
| Basque | B_NS_1 | NS | 6.85 | 1.24 | <0.1 | 1 | 6 | 26 | 3.72 | 4.3 | −0.2 | 1.7 | 99.5 | 1.7 | 96.1 | 0.180 | 20.61 | 9.20 | 3.53 |
| Country | B_NS_2 | NS | 7.21 | 1.04 | <0.1 | 6.4 | 6 | 36 | 3.72 | 4.3 | −0.2 | 1.8 | 99.4 | 1.8 | 95.2 | 0.190 | 19.65 | 8.70 | 4.40 |
| | B_NS_3 | NS | 8.42 | 1.34 | 0.18 | 1 | 5 | 37 | 3.83 | 5.0 | −0.1 | 1.3 | 99.5 | 1.3 | 95.1 | 0.140 | 3.97 | 8.75 | 4.90 |
| | B_N_1 | N | 6.84 | 2.64 | <0.1 | 1 | 6 | 20 | 3.83 | 5.5 | −0.4 | 3.8 | 98.9 | 3.8 | 96.1 | 0.400 | 94.89 | 9.33 | 5.85 |
| | B_N_2 | N | 6.43 | 2.96 | <0.1 | 1 | 6 | 20 | 3.71 | 6.8 | −0.3 | 2.9 | 99.0 | 2.9 | 94.9 | 0.315 | 69.55 | 7.68 | 3.70 |
| | B_N_3 | N | 6.79 | 2.92 | <0.1 | 1 | 6 | 20 | 3.8 | 6.0 | −0.2 | 3.0 | 98.9 | 3.0 | 93.4 | 0.330 | 86.83 | 9.05 | 4.08 |
| Canary Island | C_NS_1 | NS | 7.37 | 1.6 | <0.1 | 1.1 | 6 | 20 | 3.88 | 5.3 | −0.7 | 6.0 | 97.2 | 6.0 | 96.2 | 0.795 | 8.35 | 14.28 | 6.88 |
| | C_NS_2 | NS | 8.95 | 0.5 | <0.1 | 1.2 | 8 | 62 | 3.92 | 3.4 | 0.0 | 1.7 | 99.2 | 1.7 | 90.1 | 0.220 | 3.31 | 26.38 | 21.73 |
| | C_NS_3 | NS | 8.55 | 0.89 | <0.1 | 2.5 | 8 | 108 | 3.95 | 4.5 | −0.4 | 3.9 | 98.1 | 3.9 | 95.1 | 0.515 | 21.07 | 10.63 | 5.93 |
| | C_N_1 | N | 8.46 | 0.81 | <0.1 | 2.8 | 6 | 55 | 3.91 | 4.0 | −0.3 | 2.5 | 99.1 | 2.5 | 96.4 | 0.290 | 5.92 | 9.08 | 7.95 |
| | C_N_2 | N | 7.03 | 0.27 | <0.1 | 1.2 | 7 | 20 | 3.9 | 3.6 | −0.3 | 4.4 | 98.1 | 4.4 | 93.2 | 0.545 | 84.33 | 22.68 | 22.45 |
| | C_N_3 | N | 7.40 | 0.62 | 0.18 | 2.2 | 10 | 162 | 3.62 | 3.8 | −0.2 | 2.3 | 98.9 | 2.3 | 95.7 | 0.300 | 0.72 | 9.73 | 6.78 |

**Note(s):** Alcohol content (% v/v); VA: volatile acidity (g/L of acetic acid); MA: malic acid (g/L); RS: reducing sugars (g/L); free $SO_2$ (mg/L); total $SO_2$ (mg/L); TA: titratable acidity (expressed as g/L of malic acid); CI: colour intensity (au); Turbidity (NTU); TPI: total polyphenol index (au); HCA: hydroxycinnamic acids (au)
**Source(s):** Authors work

*2.2 Conventional parameters and CIELAB coordinates*

Conventional parameters were analysed by accredited procedures at *Estación Enológica de Haro* (La Rioja, Spain) following the UNE-EN ISO/IEC 17025 standards. Alcohol content was determined by Near Infrared (NIR), volatile acidity (VA), reducing sugars (RS), free and total $SO_2$ by an autoanalyser, pH and total acidity (TA) by potentiometry, and malic acid (MA) following the method proposed by the OIV (OIV-MA-AS313-10).

The samples were homogenised before opening the bottle, except for the sparkling ciders. Then, 50 mL of cider were degasified with ultrasound in order to measure the turbidity. The turbidity was measured with the portable turbidimeter Hanna HI 93703-11 (Hanna Instruments SL, Eibar, Spain).

Further 50 mL of samples were centrifuged at 10,000×*g* for 20 min (Zuriarrain *et al.*, 2015) and filtered at 0.45 µm using a sterile syringe. Absorbance at 280 nm (estimation of the total polyphenol content-TPI) and 320 nm (total hydroxycinnamic acid-HCA content) of diluted samples (1:50) were measured using Shimadzu UV-1800 spectrophotometer (Shimadzu Corporation, Kyoto, Japan) and quartz cuvettes of 10 mm path-length.

Colour of ciders was determined by calculating the CIELAB coordinates. The parameters calculated corresponded to the degree of cider lightness ($L_{10}$*) and the degree of red (when $a_{10}$* > 0), green (when $a_{10}$* < 0), yellow (when $b_{10}$* > 0) and blue colour (when $b_{10}$* < 0); and by its derived magnitude chroma (C*) that defines the saturation of de colour or "colourfulness", and hue (h*), which is the angle on the chromaticity axe. Therefore, the spectra (recorded every 1 nm between 380 and 780 nm) of centrifuged and filtered samples (0.45 µm) was acquired with a UV-1800 SHIMADZU spectrophotometer using 0.2-cm path-length quartz cuvettes. From the spectra, the colour coordinates were calculated using the CIE method with the CIE 1964 10° standard observer and the illuminant D65, according to the OIV.

*2.3 Polyphenol analysis*

Analysis of phenolic compounds was carried out at the Instrumental Analysis service of the ICVV according to Royo *et al.* (2021). The liquid chromatography (LC) analyses were carried out on a Shimadzu Nexera liquid chromatograph (Shimadzu Corporation, Japan), coupled to a hybrid triple quadrupole/linear ion trap AB Sciex 3200QTRAP® mass spectrometer (Sciex, USA). The analytical column used was a Waters ACQUITY UPLC BEH C18 (100 mm × 2.1 mm i.d., 1.7 µm) equipped with a Waters VanGuardTM pre-Column (5 mm × 2.1 mm i.d., 1.7 µm) containing the same stationary phase (Waters, Milford, MA, USA).

The mobile phase was 0.1% formic acid in water (eluent A), and 0.1% formic acid in acetonitrile (eluent B). The elution gradient was: 0–0.5 min, 1% B isocratic; 0.5–1.5 min, 1–8% B; 1.5–4 min, 8% B isocratic; 4–5 min, 8–12% B; 5–5.5 min, 12% B isocratic; 5.5–6 min, 12–14% B; 6–7 min, 14% B isocratic; 7–9 min, 14–22% B; 9–12 min, 22–30% B; 12–13.5 min, 30–90% B; 13.5–14.5 min, 90% B isocratic; 14.5–15 min, 90–1% B; 15–18 min, 1% B isocratic.

The flow rate was 0.45 mL/min and 2.5 µL of all samples analysed were injected. The autosampler and oven temperatures were respectively 8 °C and 40 °C.

Tandem MS analyses were carried on with a mass spectrometer (AB Sciex, USA) equipped with an electrospray ionisation source (ESI Turbo V™ Source). For further information about MS conditions refer to Appendix A.

Data acquisition and quantification were carried out with the Analyst ® 1.6.2 and MultiQuant ® 3.0.2 softwares (AB Sciex, USA). Sixty phenolic compounds were quantified, belonging to 6 chemical classes: 25 hydroxycinnamic acids and derivatives, 12 hydroxybenzoic acids and other acids, 3 volatile phenols, 6 flavonols, 11 flavanols and 3 dihydrochalcones (Table S1 of Appendix A).

*2.4 Sensory analysis*

Two tasks were carried out. The first was a labelled sorting task with the 18 samples, and aimed at comparing the sensory profile of the Canary ciders to typical ciders from Asturias and Basque Country based on overall flavour (aroma, flavour, taste and mouthfeel). The second consisted in a flash profile focused on the aroma description of the cider sample set of the Canary Islands.

*2.4.1 Sorting task.* 2.4.1.1 Panel. Sixteen participants (12 males and four females; ranging from 24 to 57 years old, average = 37) were recruited from staff members of the sensory panel of the *Instituto de Ciencias de la Vid y del Vino* (ICVV). All of them presented long experience in descriptive tasks of complex beverages such as wine, beer and cider and their derivatives and thus they are considered experts according to Parr *et al.* (2002). All participants were volunteers and before participating in the study, they signed an informed consent form defining the type of research and voluntary participation. All data were collected anonymously.

2.4.1.2 Procedure. Each panellist participated individually in one session. Twenty mL of samples (12 °C) were poured in dark glasses labelled with random three-digit codes and covered by plastic Petri dishes to allow the volatiles to equilibrate in the headspace. Panellists were presented with 20 ciders (18 samples together with two replicates) simultaneously (random and different order for each participant) and they were asked to taste samples once in the proposed order, and then as many times as they wanted and in any order. The panellists were asked to form groups of samples based on their sensory similarity. They could form as many groups as they wanted, and to put as many samples as they wished in each group (groups of single samples were permitted). After the completion of the task, the panellists were asked to provide a maximum of three words to describe each of the groups they had formed. The tasters were provided with water and pectin (concentration of 1 g/L) as rinsing agents.

*2.4.2 Flash profile.* 2.4.2.1 Panel. Eleven (five men, and six women; 24–52 years, average = 36 years old) semi-trained panellists carried out this task. They attended four 20-min training sessions, where they were presented with aroma and taste/mouthfeel references and they had to correctly identify each standard. All the panellists were members of the sensory panel of *Laboratorio de Análisis de Aroma y Enología* (LAAE) from the University of Zaragoza and had extended experience (minimum of 3 years and maximum of 30 years, average of 10 years) in sensory characterisation of different alcoholic products.

2.4.2.2 Procedure. Ten samples (six Canary ciders and two replicates together with one Asturian and one Basque cider selected as references based on the results of the sorting task) were presented simultaneously to each participant. Twenty-mL samples were poured in dark glasses labelled with random three-digit codes covered with a Petri dish. The task consisted in two phases. Firstly, participants had to orthonasally smell all the samples in the proposed order (different for each judge) and identify the attributes that made them different. Then, for each of the attributes identified in the first stage, they had to rank the 10 samples on a 10-cm scale anchored with low and high intensity on the left and right part of the scale, respectively.

*2.5 Volatile compounds*

*2.5.1 Reagents and standards.* Internal standards 2-butanol (≥99%), 4-methyl-2-pentanol (99%), 4-hydroxy-4-methyl-2-pentanone (99%), ethyl heptanoate (99%) and heptanoic acid (99%) were used for major volatile compounds, and 2-octanol (99.5%), 3-octanone (99%) and 3,4-dimethylphenol (99%) for minor volatile compounds. Dichloromethane (DCM) and methanol (≥99%) were used. All the chemical standards used in this study (>98%) were purchased from Merck (Darmstadt, Germany).

*2.5.2 Quantification of major volatile compounds.* Major volatile compounds include higher alcohols, acetates, ethyl esters and volatile fatty acids. They are present in the concentration range of 10–200 mg/L. To quantify all of them, the procedure described by Ortega *et al.* (2001) was used, but with some adaptations to the cider matrix. Therefore, the cider volatile fraction was obtained carrying out a liquid–liquid micro-extraction. The eluted fraction was then analysed by gas-chromatography with a flame ionisation detector (FID, temperature = 250 °C) using a DB-20 column (50 m × 0.32 mm, 0.5 mm of film thickness) from J&W Scientific (Folsom, CA, USA), with an uncoated pre-column (2 m × 0.53 mm). The injection was performed at 250 °C in split mode (split flow: 30 mL/min, split ratio: 1/20). The temperature program was as follows: 40 °C for 5 min, raised at 4 °C/min up to 120 °C, at 2 °C/min up to 112 °C, at 3 °C/min up to 125 °C, hold for 5 min, raised at 3 °C/min up to 160 °C and at 6 °C/min up to 200 °C and hold for 30 min. Carrier gas was $H_2$ at 2.2 mL/min.

*2.5.3 Quantification of minor volatile compounds.* Minor volatile compounds include acetate and ethyl esters, volatile phenols, terpenes, norisoprenoids and lactones. Their concentration is between 0.1 and 1,000 μg/L and were quantified as described in Lopez *et al.* (2002) with some modifications to adapt the method to the cider matrix. Therefore, cider volatiles were firstly concentrated by solid phase extraction (SPE) using pre-conditioned 65 mg LiChrolut SPE cartridges. The extract was then injected in a QP2010 gas chromatograph using a DB-WAXetr column (30 m × 0.25 mm, 0.5 μm film thickness) from Agilent (USA), preceded by a 2 m × 0.25 mm medium-polar uncoated pre-column and equipped with a Shimadzu (Japan) quadrupole mass spectrometer detector. Two millilitres of the extract were injected at 250 °C in splitless mode (flow: 4.5 mL/min), being the carrier gas He at 1.26 mL/min. The chromatographic program was as follows: 40 °C for 5 min, it was then raised at 1 °C/min up to 65 °C, at 2 °C up to 220 °C and hold for 50 min. Ion source and interface temperature were set at 220 °C and 230 °C. MS was set in single ion monitoring mode (SIM).

*2.6 Statistical analysis*

*2.6.1 Chemical parameters.* In order to evaluate the effect of cider origin and elaboration method on chemical composition, two-way ANOVAs for conventional oenological parameters, colour coordinates, volatile compounds and polyphenolic composition were calculated considering origin, method of elaboration and their interaction as fixed factors. Significance was considered at $\alpha \leq 0.05$. Analyses were carried out using XLSTAT (version 2022.2.1).

*2.6.2 Sorting task.* The results of each subject were firstly encoded in an individual similarity matrix (ciders × ciders). In individual matrices, 1 indicates two ciders were placed together in the same group and 0 when they do not share the group. Individual matrices were summed resulting in a co-occurrence matrix (ciders × ciders) that represents a global similarity matrix where larger numbers show greater similarity between two given samples. The hypothesis behind this method is that the samples grouped together are more similar than samples sorted into different groups. To obtain a spatial representation of samples, the co-occurrence matrix was submitted to an MDS analysis. A non-parametric scaling algorithm was used (alternating least-square scaling). Finally, the coordinates of samples in the retained MDS configuration were submitted to a hierarchical cluster analysis (HCA) with the Ward criteria. All the analyses were performed with the software XLSTAT (version 2022.2.1).

Regarding the analysis of the description of the groups, firstly, all the attributes were extracted, and firstly lemmatised (i.e. attributes with the same root were substituted by the same attribute; e.g. sourness and sour were replaced by sour). The list of lemmatised attributes was then provided to three experimenters and they were asked to form groups of attributes that shared meaning, and for those grouped together they had to select a representative one (e.g. red fruit, cherry, strawberry and red fruit was the representative). By

consensus, the final list of attributes was defined and for each panellist, the attributes given for a group of samples were associated to each cider of the group. The underlying assumption within this approach is that all the samples belonging to the same group were described by the terms in the same way. Thus, the frequency of quotation of each term was calculated and only terms cited at least by 15% of the panel were considered. The attributes mostly cited for a given cluster of samples derived from MDS coordinates were the ones characterising each cluster.

*2.6.3 Relationship between aroma properties and volatile compounds.* The individual rank data for aroma description following flash profile were firstly collected in a matrix built for each participant (ciders in rows and terms in columns). The global data matrix formed by the individual matrices generated by the 11 assessors was submitted to Generalised Procrustes Analysis (GPA), further HCA was calculated with the coordinates derived from the GPA. Descriptors mentioned by at least 15% of the panel were used to visualise the relationships between samples and attributes. Analyses were carried out with XLSTAT software (version 2022.2.1).

For each volatile compound, first the Odour Activity Value (OAV) was calculated as the ratio of the concentration of each molecule and its sensory odour threshold (ODT). For compounds at concentrations below their limit of detection (LOD), the ODT was replaced by its own LOD. Subsequently, aroma vectors were built by summing the OAVs (summed-OAV) of compounds sharing chemical and odour properties based on the suggestions proposed by Ferreira *et al.* (2021). Some compounds with specific sensory characteristics have been considered individually as vectors. These compounds are acetic acid, ethyl acetate, β-phenylethyl acetate, β-damascenone, diacetyl, acetaldehyde and acetoin (see Table S2 of Appendix A for detailed vector composition). The summed OAVs or individual OAVs (in the case of aroma vector composed of an individual compound) below 0.2 were arbitrarily not considered, as they are assumed to have no sensory impact (Ferreira *et al.*, 2021).

A one-way ANOVA was calculated considering the clusters derived from the flash profile as fixed factors to identify the aroma vectors that could induce significant sensory differences among the ciders.

## 3. Results and discussion

### *3.1 Effect of origin on conventional parameters, colour coordinates and polyphenolic composition*

Table 1 shows the conventional parameters evaluated for the 18 ciders studied. The alcohol content (range of 5.61–8.95, average = 7.09 ± 1.03) complied with the Spanish definition of "Cider" (≥5% v/v for natural ciders and ≥5.5% v/v for natural sparkling ciders). The alcohol and pH range values of ciders from Asturias (ethanol = 5.61–7.52; pH = 3.64–3.84) and Basque Country (ethanol = 6.23–8.42; pH = 3.71–3.83) are overall comparable with previous published results from both regions: Asturias (ethanol = 5.57–7.43; pH = 3.60–3.93) and Basque country (ethanol = 6.23–7.08; pH = 3.55–3.81) (Picinelli-Lobo *et al.*, 2016). The range of volatile acidity, which is between 0.27 and 2.96 g/L, exceed the legal limit (2.20 g/L) in three Basque natural ciders, which is in line with the results reported by Picinelli-Lobo *et al.* (2016). The authors showed levels of VA as high as 2.90 and 2.20 g/L for ciders from Asturias and Basque Country, respectively. The titratable acidity, which ranges between 3.4 and 6.8 g/L (average = 4.77 ± 0.91), is overall higher than that reported for Polish ciders (2.28–4.47 g/L, average = 3.58 ± 0.74) (Tarko *et al.*, 2021) but comparable with Spanish ciders (maximum average of 3.99 ± 0.47) (Picinelli-Lobo *et al.*, 2016).

In Basque ciders, the measured total polyphenol content (TPI) (average = 8.78 au) and the total content in hydroxycinnamic acids (HCA) (average = 4.41 au) are comparable with the

results reported for Basque ciders produced with the Mantonni apple variety (Zuriarrain *et al.*, 2015).

To evaluate the effect of the origin on the studied parameters, two-way ANOVA (Table 2) was calculated. Results showed that there is a significant simple effect of origin on the alcohol content, being significantly higher for Canary ciders (average = 8.0 ± 0.8; range: 7.0–9.0% v,v) than for those from Asturias (average = 6.2 ± 0.8; range: 5.6–7.5% v,v). Basque ciders present intermediate alcohol contents (average = 7.1 ± 0.7; range: 6.4–8.4% v,v). The warmer climate of the Canary Islands can cause a greater accumulation of sugars subsequently transformed into alcohol, in comparison with the other two northern regions.

Significant effects for the interaction origin*method were observed for volatile and total acidity, luminosity (L*) and colour intensity (Table 2). This suggests that the effect of origin observed depends on the method of production employed (natural or natural sparkling). Figure 1 shows that ciders from Canary Island (CI) show the lowest levels of volatile and total acidity regardless the method of production. The lower levels in total acidity could be attributed to apple variety, and thus genetics, and/or climate conditions. Acids concentration in apples is lower at higher temperatures, solar radiation and lower precipitation, which is the case of the apples farmed in Canary Island in comparison with those raised in the North of Spain such as Asturias and Basque Country (Mignard *et al.*, 2022). Moreover, many varieties aside from Reinette are employed in cider making, including autochthonous ones such as Bomba, Camusa, Pajarita or Pana, and foreign, such as Golden, Fuji or Starking Delicious. However, there is a lack of studies on the composition of apples in the Canary Islands, which does not allow us to compare their acidity to the rather acidic apples employed for cider making in Asturias and the Basque Country (Del Campo *et al.*, 2008; Picinelli *et al.*, 2000).

In relation to the parameters L * and colour intensity, ciders from the Basque Country and the Canary Islands present differences depending on the method of elaboration (natural and natural sparkling), being the ciders of the islands the ones that present greater difference between them. The greatest weight in the colour intensity is the absorbance at 420 nm, thus natural ciders from the Islands are the ones with the highest intensity of yellow colour, which implies lower levels of luminosity (L *). The specific procedure in the elaboration of natural

| | Origin | | Method | | origin*method | |
|---|---|---|---|---|---|---|
| | F | *P* | F | *P* | F | *P* |
| Alcoholic content | 9.366 | ** | 5.497 | * | 0.062 | ns |
| VA | 16.918 | *** | 10.745 | ** | 9.841 | ** |
| RS | 0.571 | ns | 5.007 | * | 1.067 | ns |
| Free $SO_2$ | 3.053 | ns | 3.211 | ns | 2.703 | ns |
| Total $SO_2$ | 2.028 | ns | 2.709 | ns | 0.241 | ns |
| pH | 3.542 | ns | 5.078 | * | 0.819 | ns |
| TA | 7.101 | ** | 2.519 | ns | 7.101 | ** |
| MA | 0.500 | ns | 2.000 | ns | 0.500 | ns |
| a* | 2.675 | ns | 9.418 | * | 0.370 | ns |
| b* | 3.052 | ns | 19.920 | ** | 3.336 | ns |
| L* | 10.203 | ** | 18.456 | ** | 6.911 | * |
| C* | 3.025 | ns | 19.834 | ** | 3.313 | ns |
| h* | 3.463 | ns | 1.651 | ns | 1.714 | ns |
| CI | 6.464 | * | 18.992 | ** | 4.977 | * |
| Turbidity | 1.812 | ns | 22.687 | *** | 0.959 | ns |
| TPI | 2.296 | ns | 0.001 | ns | 0.020 | ns |
| HCA | 2.874 | ns | 0.036 | ns | 0.052 | ns |

**Note(s):** Ns: not significant; significant *p*-values: *$p < 0.05$; **$p < 0.01$; *** $p < 0.001$
**Source(s):** Authors work

**Table 2.** Two-way ANOVA calculated with conventional chemical parameters considering origin, the elaboration method and their interaction as fixed factors

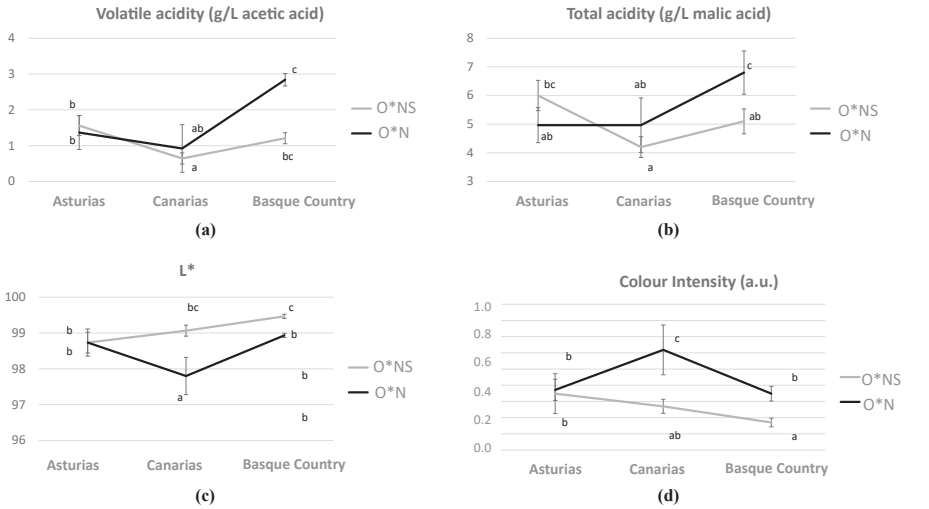



**Figure 1.**
Average of parameters with significant ($p < 0.05$) effect for the interaction origin (O) by method of production (NS or N)

**Note(s):** Natural sparkling-NS or Natural-N; a) volatile acidity, b) titratable acidity, c) luminosity (L*) Cielab coordinate, and d) colour intensity. Error bars are the standard deviation

**Source(s):** Authors work

Canary ciders makes them significantly different from those of Asturias and the Basque Country. However, the sparkling operation minimises these differences in both parameters resulting in no significant differences among the three regions for the natural sparking ciders.

It is interesting to remark that when relaxing the significance level at 10%, the overall content in hydroxycinnamic acids (HCA) is higher in the CI ciders, regardless the method of production. HCA and derivatives are the main polyphenols present in ciders (He *et al.*, 2022). In order to identify the specific compounds that are in higher concentrations in the ciders of Canary Islands, a complete analysis of individual phenolic compounds belonging to different families was carried out including: hydroxycinnamic acids and derivatives, benzoic acids, other acids, volatile phenols, flavonols, flavan-3-ols and dihydrochalcones (see Appendix A Tables S3 and S4 quantitative data for each of the 18 ciders).

Table 3 shows the results of the ANOVA calculated for individual phenolic compounds. It is confirmed that the hydroxycinnamic acids and derivatives including *t*-ferulic, *t*-coumaric and neochlorogenic acids are significantly higher in the Canary ciders (up to 8, 4 and 6 times higher in CI ciders, respectively). These compounds are among the individual compounds with the highest discrimination capacity (MAX/MIN) among the three regions studied. Regarding neochlorogenic acid (or 3-*O*-caffeoylquinic acid), the isomer of the chlorogenic acid and a major acid in apples and ciders (Alonso-Salces *et al.*, 2004; Castillo-Fraire *et al.*, 2019), was found to be at concentrations up to six times higher (2.39 mg/l) in the ciders from the Canary Islands than in those from Asturias and Basque Country (0.52 and 0.41, respectively). The production of hydroxycinnamic acids is aimed at defending the plant from ultraviolet radiation (Manach *et al.*, 2004). This fact could explain the higher levels of this class of polyphenols in the Canary Islands, given the overall higher temperature and sun exposure in the islands in comparison with northern Spain (namely Asturias and Basque Country). However, it cannot be ruled out that these differences in HA concentration could be related to the apple variety (He *et al.*, 2022).

| Phenolic compound | Canary Island | Asturias | Basque country | MAX/MIN |
|---|---|---|---|---|
| Gallic acid | 0.41 ± 0.72 b | 3.06 ± 2.71 a | 0.17 ± 0.10 b | 18.33 |
| Trans-ferulic acid | *0.16 ± 0.09 a* | 0.02 ± 0.01 b | 0.01 ± 0.01 b | 15.75 |
| Trans-coumaric acid | *0.43 ± 0.28 a* | 0.10 ± 0.09 b | 0.05 ± 0.04 b | 8.76 |
| Catechol | 0.13 ± 0.19 c | 1.10 ± 0.21 a | 0.63 ± 0.37 b | 8.47 |
| Neochlorogenic acid | *2.38 ± 1.99 a* | 0.52 ± 0.12 b | 0.41 ± 0.18 b | 5.83 |
| Hydrocaffeic | 3.47 ± 8.24 b | 15.55 ± 3.70 a | 18.36 ± 3.63 a | 5.29 |
| Protocatechuic acid | 0.69 ± 0.48 a | 0.71 ± 0.31 a | 0.17 ± 0.12 b | 4.10 |
| 2,3-dihydroxy-benzoic acid | 0.19 ± 0.06 b | 0.60 ± 0.20 a | 0.45 ± 0.11 a | 3.19 |
| Trans-aconitic acid | 25.34 ± 13.74 a | 17.89 ± 6.43 ab | 9.36 ± 8.10 b | 2.71 |
| Hydrocoumarin acid | 0.98 ± 0.47 b | 1.90 ± 0.52 ab | 2.53 ± 1.21 a | 2.59 |
| 3-O-coumaroylquinic acid | 1.24 ± 0.47 b | 2.74 ± 0.83 a | 1.82 ± 0.35 ab | 2.22 |
| Vanillic acid | 0.30 ± 0.12 ab | 0.45 ± 0.18 a | 0.21 ± 0.16 b | 2.12 |
| Syringic acid | 0.06 ± 0.02 ab | 0.09 ± 0.03 a | 0.04 ± 0.03 b | 2.00 |
| Vanillic acid HexB | 0.08 ± 0.00 b | 0.15 ± 0.07 a | 0.08 ± 0.00 b | 1.82 |
| Caffeic acid HexA | 0.04 ± 0.01 b | 0.07 ± 0.01 a | 0.05 ± 0.02 ab | 1.65 |
| Gallic acid Gluc | 0.01 ± 0.01 b | 0.02 ± 0.01 a | 0.02 ± 0.01 ab | 1.60 |
| Gallocatechin | 0.09 ± 0.03 b | 0.12 ± 0.03 ab | 0.14 ± 0.04 a | 1.57 |

**Note(s):** For a given compound, different letters indicate significant differences according to Fischer post hoc test, $p < 0.05$. In italic, polyphenols from Canary Island with highest concentrations among the three regions. For a compound, the rate between the maximum and the minimum concentration (MAX/MIN) shows the discrimination capacity for each compound among the three regions

**Source(s):** Authors work

**Table 3.** Average (± standard deviation) concentration (expressed as mg/L) of phenolic compounds significantly ($p < 0.05$) differing among the three regions

### 3.2 Sensory analysis

*3.2.1 Sorting task.* In order to sensory characterise the Canary ciders (6 samples), they were compared with their counterparts from Basque Country (6 samples) and Asturias (6 samples) following a labelled sorting task based on overall flavour (taste, mouthfeel and aroma). This approach evidences the most salient differences among the studied samples. Figure 2 shows the dendrogram derived from the HCA calculated with the MDS dimensions (see Table S5 of Appendix B for the contingency table). The results showed that the replicated samples were clustered close together (A_NS_1-A_NS_1R and A_NS_2-A_NS_2R), which suggests that the panel was globally reproducible and confirms the validity of the results.

Three main clusters of ciders were identified. Cluster 1 was mainly characterised by reduction, animal aromas and sweet taste and includes exclusively natural sparkling ciders from Asturias (A_NS_1 and A_NS_2) and from the Canary Islands (C_NS_1 and C_NS_2). Cluster 2 is mainly made up of Canary ciders (C_N_2, C_NS_3, C_N_3) and one natural sparkling cider from the Basque Country (B_NS_1). This cluster is mainly described with fruity aromas and sweet taste. In both clusters, this sweet taste seems to be the result of cognitive interactions because the levels of sugars are not significantly higher than in the other cluster. As described Sáenz-Navajas *et al.* (2010), sweet-related aromas can increase sweet taste perception. The third cluster is the most numerous comprising ten ciders: five out of six from Basque Country (B_NS_2, B_NS_3, B_N_1, B_N_2, B_N_3), four from Asturias (the three natural ciders: A_N_1, A_N_2, and A_N_3 and one natural sparkling cider: A_NS_3) and one natural cider from Canary Islands (C_N_1). This cluster is mainly described with fresh fruit aroma, with low aroma intensity and acidic taste. As in the case of the sweet taste of the previous cluster, this group of samples does not present significantly higher levels of total acidity. Thus, the acidic taste could be explained in terms of aroma interactions at cognitive levels related to the possible congruent sensations between acid taste and fresh fruit aroma (such as citrus).

Interestingly, the ciders from Canary Islands are represented in the three sensory profiles. Two natural sparkling (NS) Canary samples share the profile with the natural sparkling

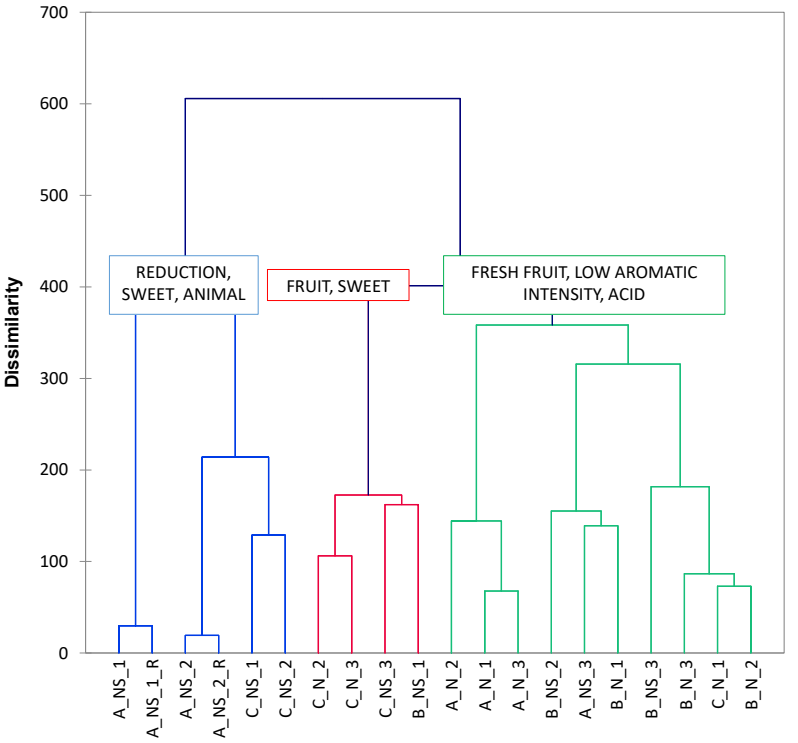
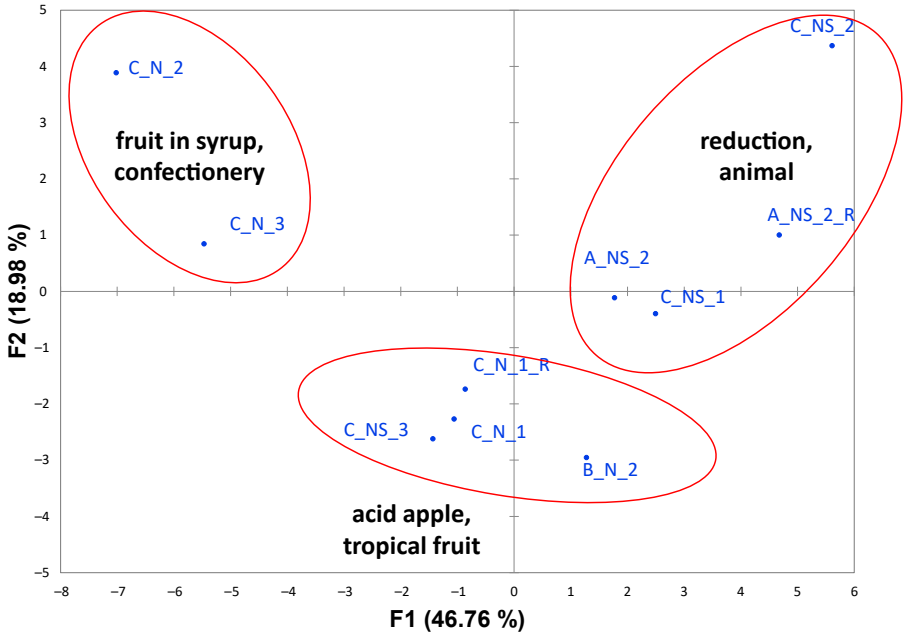



**Figure 2.**
Tree diagram derived from the Hierarchical Cluster Analysis (HCA) calculated on all the dimensions derived from the multidimensional scaling (MDS) obtained from the sorting task performed with 18 ciders and 16 panellists

**Note(s):** The descriptors are those with higher citation frequency for each cluster. The first letter of the codes represents the origin: Canary Islands (C), Asturias (A) or Basque Country (B), and the second the method of production: natural (N) or natural sparkling (NS). Replicates samples are represented by an R
**Source(s):** Authors work

ciders from Asturias (cluster 1), another natural cider (C_N_1) shares sensory profile with the Basque ones, and with natural Asturian ciders (cluster 3), while three Canary ciders (C_N_2, C_NS_3, C_N_3) present a specific sensory profile (cluster 2). In order to further deepen the specific aroma profile of Canary ciders, they were characterised exclusively in aroma following a flash profile and considering two ciders representative for the other two regions: B_N_2 for Basque country and A_NS_ 2 from Asturias, and thus as control samples. Their sensory-active volatile compounds were also quantified.

*3.2.2 Flash profile.* The GPA plot obtained from the Flash Profile carried out with the six Canary ciders together with two replicates and two ciders representative of the other two regions studied is shown in Figure 3 (see Appendix C for original data). The first two dimensions accounted for 65.74% of the original variance (46.76% and 18.98%, respectively for first and second dimensions). Interestingly, the two replicates (A_NS_2-A_NS_2R and C_N_1- C_N_1R) are plotted together, which confirms the validity of results. The HCA calculated on the GPA dimensions yielded three main specific aroma profiles. The first cluster comprising three natural sparkling ciders (upper right side of the plot: C_NS_1, C_NS_2, A_NS_2) is described with terms such as reduction and animal aromas. This result is well in accordance with the sorting task where these samples were grouped together and described




**Note(s):** The three clusters are derived from the HCA calculated on the two dimensions. The descriptors are those with higher citation frequency for each cluster
**Source(s):** Authors work

**Figure 3.**
Projection on the first to dimensions of the Generalised Procrustes Analysis (GPA) calculated with the data derived from the flash profile performed with 10 cider samples and 11 trained panellists

with terms such as reduction, sweet, and animal. A more accurate description of samples: C_N_1, B_N_2, C_NS_3 (plotted on the bottom-middle) could be specified by the flash profile, which are characterised with terms such as acid apple and tropical fruity aroma. The third group (plotted on the top-left) is formed exclusively by two Canary natural ciders (C_N_2, C_N_3) with descriptors including fruit in syrup and confectionery aromas.

Interestingly, the animal aroma profile of the first cluster can be explained in terms of ethylphenol concentration (see Table S6 of Appendix B for quantitative data for the eight ciders submitted to flash profile). Table 4 shows that this aroma vector presents the highest discrimination ability (ratio between the maximal and minimal concentration among the three clusters) among the studied samples. This vector is composed by 4-ethylphenol and 4-ethylguaiacol, which has been extensively attributed with the expression of an animal, leather-like aroma, most likely resulting from contamination with *Brettanomyces* yeasts and/or certain lactic acid bacteria (Nunes De Lima *et al.*, 2021). The relationship between the animal character and quality is not clear. While wine experts associate this aroma with clear defective and low-quality wines, non-experts have reported to appreciate this aroma at certain levels (Sáenz-Navajas *et al.*, 2015). Its role on cider perception should be further clarified given the lack of studies in this regard. Moreover, the existence of sulphur-like aroma compounds not quantified in this study such as $H_2S$ or methanethiol, responsible for reductive aroma notes, cannot be ruled out (Franco-Luesma *et al.*, 2016).

The vinylphenol vector, composed of 4-vinylphenol and 4-vinylguaiacol, shows a medicinal sweet-like aroma and presents the second highest discrimination ability among the three clusters. This could explain the fruit in syrup and confectionary aroma profile of the third cluster. The formation of both ethylphenols (animal and leather aromas) and

| Aroma vector | Cluster 1 (Fruit in syrup, bakery) | Cluster 2 (Sour apple, tropical fruit) | Cluster 3 (Animal, reduction) | *P* |
|---|---|---|---|---|
| Ethylphenols | *1.43 ± 0.41b* | *0.15 ± 16.02b* | *117.50 ± 42.79a* | *<0.05* |
| Vinylphenols | *169.94 ± 56.02a* | *7.05 ± 1.02b* | *7.87 ± 7.64b* | *<0.01* |
| Acetaldehyde | 5.66 ± 1.46 | 7.05 ± 5.86 | 4.90 ± 2.15 | ns |
| Cinnamates | 14.74 ± 5.07 | 16.05 ± 17.95 | 6.36 ± 4.41 | ns |
| Methoxyphenols | 15.11 ± 2.74 | 51.28 ± 22.85 | 14.83 ± 3.74 | ns |
| Acetates | 38.22 ± 46.52 | 11.84 ± 5.76 | 17.79 ± 13.78 | ns |
| Acetic acid | 1.81 ± 1.77 | 4.38 ± 2.33 | 2.37 ± 1.02 | ns |
| y-lactones | *0.13 ± 0.01b* | *0.66 ± 0.13a* | *0.22 ± 0.10b* | *<0.01* |
| Branched acid | 42.68 ± 16.68 | 58.99 ± 40.40 | 37.82 ± 9.19 | ns |
| b-damascenone | 184.92 ± 56.23 | 111.95 ± 34.67 | 84.37 ± 51.39 | ns |
| Phenols | 36.42 ± 5.61 | 85.08 ± 29.06 | 98.21 ± 37.45 | ns |
| Ethyl acetate | 6.42 ± 4.55 | 6.71 ± 2.56 | 5.01 ± 1.87 | ns |
| Ethyl esters | 17.61 ± 8.28 | 22.67 ± 8.01 | 18.65 ± 4.13 | ns |
| Linear fatty acids | 27.68 ± 1.79 | 21.26 ± 3.96 | 24.44 ± 10.63 | ns |
| Higher alcohols | 10.53 ± 3.16 | 12.02 ± 3.93 | 10.84 ± 2.23 | ns |
| Terpenes | 0.64 ± 0.08 | 0.63 ± 0.14 | 0.64 ± 0.09 | ns |
| Ionones | 1.78 ± 0.07 | 1.58 ± 0.26 | 1.56 ± 0.22 | ns |

**Note(s):** P: significance according to ANOVA considering cluster as fix factor. For a given aroma vector, different letters indicate significant differences among the three clusters according to Fischer post hoc test, $p < 0.05$. In italic, vectors with significant effects
**Source(s):** Authors work

**Table 4.**
Mean values and their standard deviation of the aroma vectors for the clusters of ciders derived from flash profile

vinylphenols (medicinal and sweet aroma) has its origin in the transformation of hydroxycinnamic acids *by Brettanomyces dekkera* yeasts and/or *Lactobacillus collinoides* lactic acid bacteria strains (Buron *et al.*, 2012). This formation depends on the genus and sexual form (teleomorph) of the yeast (Nunes De Lima *et al.*, 2021; Chatonnet, 1997). Two sequential enzymatic reactions are reported to occur: 1) the hydroxycinnamic acid is decarboxylated in 4-vinylphenol (from *p*-coumaric acid) or 4-vinylguaiacol (from ferulic acid) and then 2) a reductase converts the vinylphenol in 4-ethylphenol or 4-ethylguaiacol (Nunes De Lima *et al.*, 2021). The fact that the Canary Islands' ciders present the highest levels of hydroxycinnamic acids could explain their genuine fruit in syrup and confectionery aroma attributed to their highest vinylphenol levels. In turn, the acid apple or tropical fruit character of the second cluster could be attributed to the presence of polyfunctional mercaptans, not measured in the present study, with characteristic tropical and fresh-fruit aroma (Mateo-Vivaracho *et al.*, 2010).

## 4. Conclusions

For the first time, this exploratory study offers new knowledge regarding the chemical and sensory characteristics of the currently existing Canary ciders in the market. Ciders from this archipelago show a differential sensory and chemical profile, characterised by higher levels of ethanol and of hydroxycinnamic acids, mainly *t*-ferulic, *t*-coumaric and neochlorogenic acids, and lower total and volatile acidity. All those can be interpreted as potential markers of the typicity of Canary ciders. Likewise, while Canary ciders present different aroma profiles, a genuine fruit in syrup and confectionary aroma profile has been identified by sensory analysis. This aroma is related to their highest levels of vinylphenols formed by transformation of hydroxycinnamic acids. All this information could be useful to obtain a typical profile of Canary cider and to promote the creation of a certification of ciders from the Canary Islands under a Designation of Origin scheme.

A possible limitation of the present work is the reduced number of Canary ciders available in the market. This hinders the generalisation of the concept of typicity of Canary ciders. Future research could address a more detailed description of samples by conventional descriptive analysis in order to provide more detailed profiles of the ciders as reported by Cole *et al.* (2023).

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(The Appendix follows overleaf)

## Appendix

The supplementary/Appendix material for this article can be found online.

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