

## CHEMICAL CHARACTERIZATION OF BOTTLED SWEET WINES FROM THE CANARY ISLANDS (SPAIN)

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Samples of sweet wines from the Canary Islands belonging to the Denominations of Origin of La Palma and Lanzarote islands were analysed in relation to chemical parameters. The main chemical parameters analysed demonstrated that these wines fulfil all the legal requirements, since the content of all components tested falls below the maximum concentration admissible. Applying techniques of multivariate analysis (principal component, discriminant and cluster analysis), a complete differentiation could be achieved between the wines according to the island of production using only alcohol degree and isobutanol, which are chemical parameters related to the elaboration process.

**Keywords:** Canarian sweet wines, chemical analysis, quality control, multivariate analysis

The Canary Islands are a group of seven islands located in the Atlantic Ocean, near the West African coast, between 27° 37' and 29° 23' north latitude, the same latitude as the Sahara desert. However, due to the very humid Atlantic winds, the Canarian cold stream, the orography and the circulating masses of air, the Canary Islands have many different microclimates and they are an excellent region to grow grapevines between 200 m and 1500 m, according to the zone. The soils are volcanic, formed over different periods of time and are thus in different states of evolution. Generally, the soils on which vines are grown are light, permeable, rich in nutrients and have slightly acidic pH. Temperatures are moderate with little variation, changing with altitude and proximity to the sea (LÓPEZ ARIAS et al., 1993).

Grapes have been grown in the Canary Islands (Spain) since their introduction by the Spanish conquerors at the beginning of the XVth century. Most of them do not grow anywhere else in the world because of the epidemics of phyloxera that razed European crops, but did not affect the Canaries, where this pathogen has never been detected. Wines constituted the main sources of income of the islands for almost three centuries. The main variety was Malmsey (Malvasía) and the wines produced were sweet. Canary Islands' wines became known by the generic name "Canary", a sort of Denomination of Origin (RODRÍGUEZ RODRÍGUEZ, 1973). Although the traditional sweet wines have been maintained, production was limited to the islands of Lanzarote and La Palma, both

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being a Denomination of Origin. Wines are elaborated mainly with the Malmsey grape variety and in many vineyards the vines are more than 100 years old. The systems of cultivation and elaboration processes in both these islands are totally different. In Lanzarote the vines are either planted in trenches or in holes about two metres in diameter and one metre deep, protected by semi-circular stone walls. In La Palma, the plants are also grown close to the ground, in the manner of a creeper. The majority of plants, in both islands, are grown in black volcanic soil, with a high moisture-retaining capacity. Besides, there are substantial differences in the elaboration processes for these wines between both islands. In La Palma the wines are natural sweet wines elaborated with overmatured grapes, without addition of alcohol or sugar, while in Lanzarote the grapes are not overmatured and wines were fortified with the addition of alcohol.

The sweet wines from La Palma, particularly Malmsey, are much more expensive than the wines from Lanzarote and it is therefore necessary to typify them in order to avoid fraud.

No reports have been published to date on the bottled sweet wines of the Canary Islands. The aim of the present work is to characterize for the first time the sweet bottled wines from the Canary Islands, according to the island of production (Lanzarote or La Palma). To improve the quality control of such wines, the main chemical parameters were evaluated, establishing accurate differentiation criteria by employing multivariate data analysis techniques.

## 1. Materials and methods

*Samples.* The samples used for the present study represent all the sweet wines bottled, produced during two consecutive harvests, carrying the Denominations of Origin (DOs) of the islands of Lanzarote and La Palma (Canary Islands). The distribution of the wines was as follows: nine wines from the first harvest and eight wines from the second harvest, eight from the DO Lanzarote from four wine cellars, and nine from the DO La Palma from five wine cellars. All the samples were provided by the Certification Denomination of Origin Council to ensure the geographic origin of the wines. The main variety used in the production was Malmsey, both in Lanzarote and La Palma, although some samples of La Palma were elaborated with Sabro (two samples) and Verdello (one sample) varieties. All the wines of La Palma were elaborated as natural sweet. The wines of Lanzarote were elaborated in different ways: one sample was elaborated until total fermentation and then concentrated must was added; two wines were elaborated without fermentation, with addition of alcohol only (mistelas); and five wines were fermented partially and then alcohol was added.

*Analytical determinations.* Samples were analysed to determine the following parameters: pH, total acidity, malic acid, volatile acidity, density, alcoholic strength, ashes, alkalinity of ashes, free sulfur dioxide, total sulfur dioxide, fructose, glucose, colour and major volatiles (acetaldehyde, ethyl acetate, methanol, 1-propanol, isobutanol, amylic alcohols) according to Official Methods (OIV, 1990), dry extract by

mathematical approaches, tartaric acid according to the modified Rebelein method (VIDAL & BLOUIN, 1978), reducing sugars according to the Rebelein method, tannins by the Lowenthal method, total polyphenol index by the Masquelier method and catechins by the Pompei and Peri method (GARCÍA BARCELÓ, 1990). Determinations were performed in duplicate.

*Multivariate analysis.* Pattern recognition analysis was performed by means of the statistical software package STATGRAPHICS Plus for Windows 4.0 from Statistical Graphics Corporation, on a Pentium 100 using a HP 690 C as graphic output.

*Data analysis.* Data were autoscaled to zero mean and unit standard deviation. Principal Component Analysis (PCA) was used to achieve a reduction of dimensionality. Stepwise Linear Discriminant Analysis (LDA) was used to select the most discriminant variables. Cluster Analysis (CA) was used to group wines in terms of similarity (OTTO, 1998).

## 2. Results and discussion

Table 1 shows the mean value and standard deviation of the different chemical parameters corresponding to the sweet wines from the islands of Lanzarote and La Palma, grouped in five blocks: acidic; alcoholic, glucidic and extract; sulphur dioxide and mineral; phenolic and volatile compounds.

With respect to the acidic composition, the sole parameters whose mean values showed significant differences between the wines of the two islands were total and volatile acidity. The mean pH values of the wines from La Palma and Lanzarote were similar. The mean total acidity of the La Palma wines was greater than those of Lanzarote, both presenting similar variability. Some wines from Lanzarote have a total acidity slightly below the minimum limit ( $4.5 \text{ g l}^{-1}$ ) stipulated by the legislation for wines with a Denomination of Origin. A wine from La Palma elaborated with the Verdello grape variety presented a total acidity ( $6.9 \text{ g l}^{-1}$ ) greater than those of the remainder, characteristic of the variety. The tartaric acid/malic acid ratio is lower than 1.0 in both DOs (La Palma 0.9 and Lanzarote 0.8). This characteristic is uncommon in wines elaborated with grapes of the species *Vitis vinifera* L. In the majority of the cultivars studied in different countries, the content in tartaric acid is invariably greater than that of malic acid, although in Italy the white Prosecco variety also exhibits levels of malic acid higher than those of tartaric acid (COSTACURTA & TOMASI, 1994). This difference in acidity may be due to the variety of grape employed, since the varieties do not have the same ability to accumulate and degrade malic acid (CHAMPAGNOL, 1984). Thus, in the mature state, the varieties Chenin, Pinot Noir and Cariñena are richer in malic acid than the Chasselas, Sultanina or Cabernet Sauvignon varieties (KLEWER et al., 1967). Likewise, the degradation of malic acid in grape is correlated with temperature (HALE & BUTTROSE, 1974) and with light intensity (KLEWER & LIDER, 1970).

Table 1. Average value and standard deviation of chemical analysis of sweet wines from the Canary Islands, according to the island

Composition <sup>†</sup>	Island	
	Lanzarote	La Palma
<i>Acidic parameters</i>		
pH	3.59 ± 0.33	3.62 ± 0.23
Total acidity (g l <sup>-1</sup> tartaric acid)**	4.32 ± 0.71	5.42 ± 0.72
Volatile acidity (g l <sup>-1</sup> acetic acid)*	0.50 ± 0.33	0.81 ± 0.19
Tartaric acid (g l <sup>-1</sup> )	1.48 ± 0.34	1.29 ± 0.43
Malic acid (g l <sup>-1</sup> )	1.64 ± 0.92	1.55 ± 0.34
<i>Alcohol, glucidic and extract parameters</i>		
Density (g l <sup>-1</sup> ; 20 °C)	1040.3 ± 14.78	1029.9 ± 12.40
Alcohol degree (% vol, 20 °C)	13.4 ± 2.13	14.51 ± 1.65
Total dry extract (g l <sup>-1</sup> )	150.5 ± 42.6	131.7 ± 39.86
Reducing sugars (g l <sup>-1</sup> )	145.8 ± 52.7	108.4 ± 45.60
Glucose (g l <sup>-1</sup> )**	61.84 ± 25.67	27.97 ± 15.76
Fructose (g l <sup>-1</sup> )	74.0 ± 23.77	76.41 ± 18.40
Glucose/Fructose***	0.85 ± 0.24	0.37 ± 0.21
<i>Sulphur dioxide and mineral</i>		
SO <sub>2</sub> total (mg l <sup>-1</sup> )*	97.0 ± 34.6	139 ± 39.5
SO <sub>2</sub> free (mg l <sup>-1</sup> )	11.72 ± 7.65	20.64 ± 17.95
Ash (g l <sup>-1</sup> )	3.09 ± 0.96	3.42 ± 1.0
Ash alkalinity (meq l <sup>-1</sup> )	26.7 ± 7.29	26.92 ± 5.78
<i>Phenolic parameters</i>		
O.D. 420 nm	0.27 ± 0.17	0.31 ± 0.06
Tannins (g l <sup>-1</sup> )	1.61 ± 0.91	1.83 ± 0.91
Total polyphenol index	23.79 ± 13.17	26.2 ± 12.9
Catechins (mg l <sup>-1</sup> )	15 ± 0.05	17 ± 0.05
<i>Volatile compounds</i>		
Acetaldehyde (mg l <sup>-1</sup> )	125.56 ± 46.49	166.76 ± 92.23
Ethyl acetate (mg l <sup>-1</sup> )***	57.66 ± 30.08	132.1 ± 29.8
Methanol (mg l <sup>-1</sup> )	112.9 ± 27.3	131.5 ± 31.9
1-Propanol (mg l <sup>-1</sup> )*	14.0 ± 13.5	27.29 ± 5.37
Isobutanol (mg l <sup>-1</sup> )***	19.47 ± 15.46	51.61 ± 8.68
Amylic alcohols (mg l <sup>-1</sup> )***	85.27 ± 69.18	206.93 ± 44.77
Total higher alcohols (mg l <sup>-1</sup> )***	116.0 ± 95.4	285.8 ± 60.83

\*, \*\* and \*\*\* indicate significance at P<0.05, 0.01 and 0.001, respectively.

†: Mean ± standard deviation.

The mean volatile acidity is significantly greater in wines of La Palma (0.81 g l<sup>-1</sup>) than those of Lanzarote (0.50 g l<sup>-1</sup>), probably attributable to differences in the development of the alcoholic fermentation (AF) (PEYNAUD, 2000). In the La Palma wines, the AF is developed until the levels of ethanol are sufficiently high to inhibit yeast development (FUGELSANG, 1997), while in those from Lanzarote, the wines are fortified before AF finalises, and in some cases (mistelas) it is not even performed. All samples were below 1.0 g l<sup>-1</sup>, which may be considered correct.

No significant differences were observed in mean density content, total dry extract, reducing sugars and alcohol grade between the wines of both islands. All the wines analysed fulfil the minimum legal requirement of a sugar content of 45 g l<sup>-1</sup>. Four of the wines analysed exceed 15% vol, and should therefore be classified as liqueur wines. Likewise, the Lanzarote wines (between 31.9 and 96.55 g l<sup>-1</sup>) exhibited a significantly greater mean content of glucose than the wines of La Palma (between 7.6 and 61.0 g l<sup>-1</sup>), as well as a mean glucose/fructose ratio (between 0.39 and 1.20) significantly higher than those of La Palma (between 0.14 and 0.73). During fermentation this ratio varies between 0.95 at the beginning and 0.25 at the end (PEYNAUD, 2000). The shorter fermentation process of Lanzarote wines accounts for their high glucose/fructose ratio.

The mean content in total SO<sub>2</sub> was significantly greater in the wines of La Palma, and that of free SO<sub>2</sub> in those of Lanzarote. The highest value of total SO<sub>2</sub> may be accounted for by the longer fermentation process of the La Palma wines, generating a larger amount of acetaldehyde (BURROUGHS & SPARKS, 1973), or a greater sulfur content at the onset of fermentation (BOULTON et al., 1996). All the wines present levels of SO<sub>2</sub> below the maximum value permitted by the legislation (200 mg l<sup>-1</sup>). The mean content of ash and that of alkalinity do not present significant differences between the two islands.

No significant differences in phenolic components were observed between the wines of both islands. Only slightly higher values of the total polyphenols index, and tannin and catechins content were found in the wines of La Palma.

The greatest differences detected between the wines of both islands were those observed in the volatile compounds. Thus, the wines of La Palma presented a mean content in all the volatile components greater than those of Lanzarote, which can be attributed to differences in the processes of elaboration. These observed differences in contents were significant in the cases of ethyl acetate, propanol, isobutanol, amyl alcohols and the sum of higher alcohols.

The mean ethyl acetate content of the wines of La Palma were more than twice than that of the Lanzarote wines. Ethyl acetate may be produced by the yeasts from the beginning of alcoholic fermentation, and it develops as a linear function of the increase in alcoholic graduation (USSEGLIO-TOMASSET, 1998) and because of the acetic bacteria. Moreover, its production may be related to oxygen levels in the wine (DUPUY & MAUGENET, 1962; DRYSDALE & FLETE, 1988). In any case, all the samples presented a content lower than 200 mg l<sup>-1</sup>, above which value ethyl acetate is considered to cause a negative effect on aroma.

The levels of methanol were only slightly greater in the wines of La Palma. This is due to the fact that methanol is not generated during the alcoholic fermentation but is caused by demethylation of the pectins by pectin methylesterase, which action can be reinforced by pectic enzymes added to the must before fermentation (OUGH & CROWELL, 1979). The mean values of the wines of both DOs present a content lower than 150 mg l<sup>-1</sup>, which is in agreement with the recommendations of the OIV and well below the legislated maximum limit of 500 mg l<sup>-1</sup>.

Higher alcohols, in general, presented values lower than those reported in the literature. The differences in higher alcohol content may be due to the variety of grape employed (RANKINE, 1967; CABRERA et al., 1988) or to differences, that are more qualitative than quantitative, in the composition of the must (SINTON et al., 1978; OUGH & BELL, 1980; HERRAIZ et al., 1989; RAPP & VERSINI, 1991). In our case, the wines of La Palma (228 a 368 mg l<sup>-1</sup>) presented higher alcohol values than those of Lanzarote (0 to 280 mg l<sup>-1</sup>), which is probably attributed by us to the elaboration process. Indeed, even two samples of Lanzarote wines do not possess higher alcohols due to the fact that no AF process was performed and therefore these wines are “mistelas”, that is, musts to which alcohol has been directly added without initiating fermentation. A concentration of higher alcohols that exceeds 400 mg l<sup>-1</sup> is a factor conducive to lowering the quality of a wine (RAPP & VERSINI, 1991), a situation that was not observed in any of the samples analysed in this work.

Although certain parameters displayed significant differences in content between the islands of La Palma and Lanzarote, it is difficult to establish the differentiation according to the island of production on the basis of the univariate analysis. It is more reliable to use multivariate techniques of data analysis (NOGUEIRA & NASCIMENTO, 1999).

In order to perform an overall study of the different chemical variables of the wines, a principal component analysis was carried out to reduce the number of variables that could account for the variance and to detect the internal structure between variables and samples. Initially, an explained variance of 53% was obtained. However, after removing those variables that possessed a percentage smaller than 70% in the explanation of principal components (PC), 72.8% of total variance was obtained with 11 of the 29 initial variables (Table 2). The PC1 with a great weight of the parameters of volatiles (in the negative part of the axis) and those related to the sugars (positive part of the axis) represents the alcoholic fermentation process and differentiates between the samples (La Palma to the left and Lanzarote to the right of the figure) as a function of the degree to which the alcoholic fermentation was allowed to develop (Fig. 1). Thus, the wine sample of Lanzarote that is presented together with the wines of La Palma corresponds to the sole sample of Lanzarote to undergo total alcoholic fermentation. The wines from La Palma presented a smaller dispersion than those of Lanzarote, accounted for by the greater homogeneity in the elaboration (all as natural sweet wines).

A stepwise discriminant analysis is presented below in order to determine which chemical variables differentiate best the wines according to the island of production. It is observed that the sweet wines of the two islands could be separated with only two variables, isobutanol and alcoholic degree (Fig. 2). If the correlations between variables are taken into account, differentiation between the wines from the two islands was also achieved with the following pairs of variables: isobutanol/fructose, isobutanol/density, isobutanol/dry extract, amylic alcohols/dry extract, amylic alcohols/fructose, higher alcohols/grade and higher alcohols/fructose. It can also be observed here that the samples from La Palma present a lesser dispersion than those of Lanzarote, which may be accounted for by a more homogeneous winemaking practice among the different vineyards of the island (elaboration as naturally sweet wines).

Table 2. Factor loadings for the first two PCs of a test set of chemical data from the 17 samples of Canarian sweet wines

Chemical parameters	Factor loadings	
	PC 1	PC 2
pH	0.235	0.851
Density	0.914	-0.247
Total dry extract	0.869	-0.137
Total sugar	0.968	-0.057
Fructose	0.745	-0.316
SO <sub>2</sub> total	-0.825	-0.015
Ash	-0.011	0.895
1-Propanol	-0.846	-0.063
Isobutanol	-0.853	0.005
Amylic alcohols	-0.907	0.002
Total higher alcohols	-0.916	-0.006

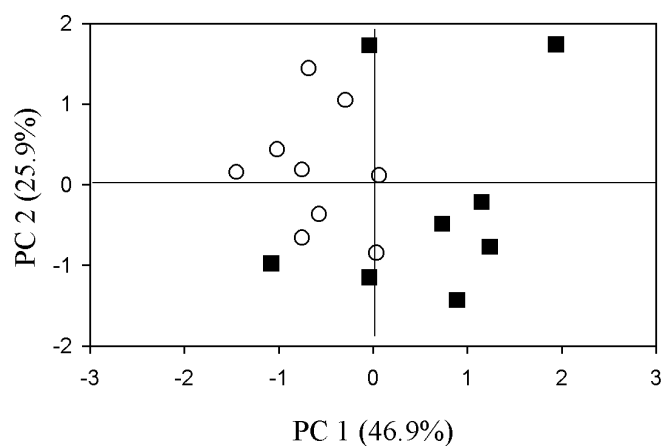


Fig. 1. Principal component analysis of eleven chemical variables for seventeen samples of Canary Islands sweet wines according to island. ■: Lanzarote; ○: La Palma

Taking the isobutanol and alcoholic degree variables, a cluster analysis was carried out using the Ward's method and making use of the Manhattan distance, Fig. 3, and it was confirmed that not only can the wines of the two islands be separated, but it is possible to differentiate the individual vineyards of the island of Lanzarote.

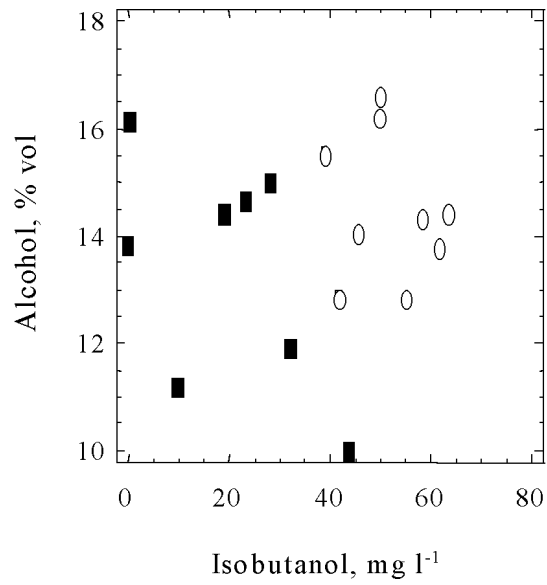


Fig. 2. Plot of isobutanol vs alcoholic degree ■: Lanzarote; ○: La Palma

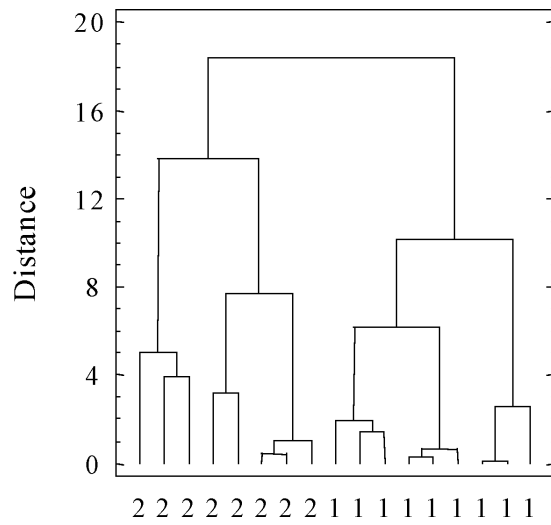


Fig. 3. Dendrogram using the variables isobutanol and alcoholic degree (Ward's method and Manhattan distance). 1: La Palma; 2: Lanzarote



### 3. Conclusions

Four wines of Lanzarote have a total acidity slightly below the legally established limit for wines with this Denomination of Origin. The wines of Lanzarote present a significantly higher content than those of La Palma in glucose and glucose/fructose ratio, and the wines of La Palma present a significantly greater content than those of Lanzarote in total acidity, volatile acidity, total SO<sub>2</sub>, ethyl acetate, 1-propanol, isobutanol, amylic alcohols and higher alcohols. The tartaric acid/malic acid ratio is lower than 1.0, which is highly unusual in wines elaborated with the *Vitis vinifera* grape variety. The principal components analysis indicates that with 11 variables 73% of the variance can be explained, a differentiation being observed in the wines in accordance with the intensity of the development of the alcoholic fermentation, marked by the levels of higher alcohols and the sugar content. The stepwise discriminant analysis showed that with chemical parameters, isobutanol and alcoholic degree, a total differentiation could be achieved between the wines of La Palma and Lanzarote. This result was confirmed by way of a cluster analysis using these two variables.

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