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Multivariate analysis of composition and sensory quality criteria of white vinegars

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RÉSUMÉ

Analyse multivariée de paramètres de composition et d'évaluation sensorielle de vinaigres blancs.

Les consommateurs sont de plus en plus intéressés aux vinaigres naturels comme ceux de vin ou de pomme ; en conséquence, l'offre de ces produits s'est accrue. Il est donc nécessaire de mettre en valeur les produits de qualité et de distinguer les paramètres qui les caractérisent. A cet effet, 71 vinaigres, achetés en Italie et à l'extérieur, ont été analysés et les résultats ont été interprétés par analyse statistique multidimensionnelle. L'analyse de nombreux échantillons a permis d'identifier des paramètres chimiques pour classer des produits d'acidité et de source différentes. La comparaison entre évaluations sensorielles et résultats analytiques a permis d'ajuster un modèle linéaire de la qualité sensorielle en fonction de la composition chimique. En particulier, on a observé que les dégustateurs, parmi les produits de même acidité, ont préféré ceux de plus forte vinosité et de plus grand extrait sec.

Mots clés : *vinaigre de vin, analyse discriminante linéaire, analyse sensorielle, analyse en composantes principales, régression linéaire multiple.*

SUMMARY

Natural vinegars such as those produced from wine or apples have aroused increasing interest in consumers causing an increase in the range of products available. It has therefore become necessary to underline the value of quality products by pinpointing the characteristics of the product. For this, multivariate statistical analysis was used to analyse the test results of 71 white vinegars found on French, Swiss, Spanish and Italian markets. Such analysis gave rise to specific analytical parameters that may be used to distinguish products made from the various raw materials with different acidity levels. A comparison between the results of sensory and analytical testing gave rise to a simple model in which the sensory qualities of a given vinegar are a function of its chemical composition. In

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particular with products of identical acidity, the testing panel preferred vinegars with a higher wine flavour and dry extract content.

Key-words : *wine vinegar, linear discriminant analysis, sensory analysis, principal component analysis, principal component regression.*

1 - INTRODUCTION

Europe currently produces over five million hectoliters of fermented vinegar. These vinegars are both directly consumed and used as an ingredient by the food industry. Italy and Spain together produce over 20% of Europe's vinegar but this production results almost entirely from the acetic bioxidation of wine. As European prospects pave the way towards an easier and further-reaching circulation of food products between different countries, the time has come to characterise not only wine vinegars but also alcohol and apple vinegars.

In Europe the majority of work on vinegar has been performed by the Spanish school at Llaguno. With the exception of research on balsamic vinegars, most of the Italian work in the field has been carried out by the Mecca's group, in charge of the Vinegar Control Office in Milan (CARNACINI and GERBI, 1992). Nevertheless, there has been much less published about wine vinegar than about other food products and most research in the field has been concentrated on studying analytical methods to reveal adulteration.

As a result, countries with ancient vinegar-making traditions, such as Italy and Spain, have started research into finding precise methods for characterising quality products. The research is not so much concerned with providing criteria for characterising the authenticity of the product since these criteria are already available, (Ministero dell'Agricoltura e delle Foreste, 1986; MECCA and VICARIO, 1971; SAKATA *et al.*, 1991), as with finding indicators which characterize the quality, typicallity and value of vinegars on the basis of physico-chemical and sensory parameters.

The aim of this work is to provide a contribution to singling out these parameters by performing chemical analysis of commercial vinegars and then determining the relationship between these results and the sensory evaluation of quality.

2 - MATERIALS AND METHODS

Seventy-one white vinegar samples acquired on Italian, French, Spanish and Swiss markets were analysed. The samples were divided into ten categories according to raw material, declared acidity and country of production *(table 1)*.

Samples were selected from practically all commercial brands on the market. While this resulted in a good representation of the available market, it gave rise to an imbalance in the number of vinegars per category since certain vinegars (alcohol, apple, malt) are commercialised only by a few companies or by a single manufacturer.

Table 1

Vinegar categories analysed with their identification codes

Vinegar categories	Identification code	Samples
Italian – Wine – White – 6 % of acidity	IWW6	23
Italian – Wine – White – 7 % of acidity	IWW7	14
Italian – Wine – Decolored	IWDE	2
French – Wine – White	FWW	5
Spanish – Wine – White	EWW	6
Swiss – Wine – White	CHWW	2
Alcohol	AL	4
Apples	AP	13
Malt	MA	1
Honey	HO	1

The main analytical parameters (density, total acidity, volatile acidity, fixed acidity, dry matter, ash, ash alkalinity, pH) were determined according to official italian methods (Ministero dell'Agricoltura e delle Foreste, 1986). Ethanol was determined by packed gas chromatography after the sample was neutralised with Na_2CO_3 (ANTONELLI, 1994). Tartaric, malic, lactic, citric and succinic acids and glycerol were determined by HPLC using an ion exchange column (GERBI and TORTIA, 1991). Total polyphenols were determined by spectrophotometry (SINGLETON and ROSSI, 1965) while fractionation of tannic and non tannic phenols was carried out according to PERI and POMPEI (1980). Procyanidins and catechins were determined according to MARGHERI and FALCIERI (1972) whilst metals were determined by atomic absorption spectrophotometry. Prolin was determined, after purification (ADAMS, 1974), according to PIRINI (1992).

Absorption spectra of vinegars were measured with a spectrophotometer using cells of 1 cm path length. Data were collected at 625, 550, 495 and 445 nm in order to calculate the X, Y and Z tristimulus values. Ligthtness, saturation and chroma were calculated by X, Y and Z tristimulus values according to the OIV method (OIV, 1990). The color was determined by measuring the absorbance (A) at 420 nm (10 mm cell) using a spectrophotometer.

Sensory analysis was carried out by a panel of 57 assessors (47 women and 10 men). The average age category was 31-50 years. Since preliminary methods used to limit the taste and smell aggressiveness of the samples (*e.g.* dilution with hot or cold water, neutralisation with alkali), were found to distort the aroma and attenuate the differences between samples, the vinegars were tasted in their natural state. Samples were served in normal tasting glasses and the taste was evaluated using a glass rod or a stainless steel teaspoon to limit the quantity of vinegar ingested. The panel expressed a "degree of liking" for the vinegars with a score between 0 (extreme dislike) and 100 (extreme liking). During tasting sessions the assessors were only told the acidity of the product, without being given any information on the manufacturer's name or the source (wine, apple, etc.) of the vinegars.

Results of physico-chemical and sensory analysis were processed using SPSS ver. 6.0 software (SPSS Inc., Illinois, USA).

2 - RESULTS

2.1 Chemical analysis

Table 2 presents mean values and standard deviations of the physico-chemical parameters examined. Copper, zinc, manganese and lead are not reported because they were not detected in any of the analysed vinegars.

Decolored vinegar is a vinegar present only on the Italian market and obtained by the intense decoloration of wine vinegars. The vinous origin of this product is underlined above all by a high content of tartaric acid and prolin, although decoloration also affects polyphenolic and mineral content. Furthermore, the iron content of such products is, on an average, about three times that of wine vinegars, and is only interpretable by transfer of iron from the decolorizing carbons used.

Alcohol vinegars are characterised by an almost total absence of most of the examined components, with the exception of acidity, which is exclusively volatile. As they are generally coloured with caramel (Mecca *et al.*, 1979), this justifies the values found for colour. Mineral substances are also absent, except for those used as integrators during acetic bioxidation.

Honey vinegar represents an infinitesimal fraction of the Italian and European vinegar market, but it has a definite placing on the ever increasing market for "organic" products. The sample examined, the only brand on the Italian market, revealed an acidity higher than that of apple vinegars and had a particularly high extract value, which was probably due to the presence of a sugary residue. For honey vinegar, as for apple vinegars, acidity is given only by acetic acid, while organic fixed acids are absent or nearly absent, with the exception of citric acid. The presence of citric acid may be due to subsequent addition to correct the acidity of the vinegar or due to the activity of gen. *Kloeckera* yeasts (CANTARELLI, 1965) during alcoholic fermentation. The quantity of mineral substances, ash, and polyphenolic substances is low and thus the colour is particularly pale (see absorbance at 420 nm).

A comparison between Italian wine vinegars and cider vinegars indicates a high number of variables that are statistically different between products *(table 3)*.

No statistically significant differences were revealed by a comparison between the cations, except for sodium and potassium. In particular, sodium levels were low in cider vinegars and high in wine vinegars possibly due to additions of NaCl

Table 2

Mean values and standard deviations of analytical parameters for vinegar categories. (For interpretation of the categories see table 1)

		IWW6		IWW7		IWDE		FWW		EWW		CHWW		AL		AP		MA	HO
		X	s	X	s	X	s	X	s	X	s	X	s	X	s	X	s		
Density		1.013	0.0008	1.0157	0.0016	1.0136	0.0006	1.0152	0.0026	1.0157	0.003	1.0103	0.0011	1.011	0.0014	1.0136	0.002	1.0124	1.0177
Alcohol	mLL^{-1}	2.8	8.9	6.1	4.4	1.0	1.1	5.8	8.1	4.2	3.2	3.1	0.4	4.4	4.6	2.1	0.4	0.6	9.6
Total acidity	gL^{-1}	61.3	3.6	74.4	2.2	63.0	0.1	65.4	6.3	70.1	6.2	49.8	5.9	73.5	10.8	54.0	0.7	61.2	64.8
Volatile acidity	gL^{-1}	60.2	4.8	71.2	4.5	62.1	1.2	51.4	5.5	60.1	9.1	41.4	4.2	67.9	18.5	47.8	5.4	54.9	58.2
Fixed acidity	gL^{-1}	1.9	0.9	3.5	3.2	1.9	0.6	13.9	5.8	9.9	7.2	8.4	1.7	9.4	17.2	8.5	5.3	10.5	8.2
Extract	gL^{-1}	13.0	4.1	15.1	2.3	8.5	5.1	16.0	6.8	15.7	6.1	9.3	0.8	3.6	4.0	16.4	4.3	9.5	25.0
Ash	gL^{-1}	2.00	0.52	1.98	0.38	1.49	0.25	2.44	1.22	2.97	1.52	1.69	0.15	0.33	0.20	2.25	0.42	1.20	0.96
Ash alkalinity	$meqL^{-1}$	17.5	4.9	16.7	3.8	13.0	0.8	20.8	12.1	20.7	13.3	12.5	0.7	2.6	2.3	24.8	5.5	8.4	9.6
Glycerol	gL^{-1}	3.2	1.3	4.3	0.6	1.7	1.6	1.8	0.2	2.6	1.3	2.0	0.1	0	0	1.7	0.9	1.6	3.4
Proline	mgL^{-1}	270	119	349	189	287	13	245	126	297	108	171	10	8	10	12	10	2	2
pH		2.89	0.10	2.78	0.08	2.81	0.04	2.91	0.22	2.86	0.17	2.79	0.04	2.36	0.05	3.02	0.09	2.78	2.72
Tartaric acid	gL^{-1}	1.33	0.58	1.86	0.26	0.74	0.79	1.22	0.15	1.38	0.45	0.57	0.04	0	0	0.02	0.04	0	0
Malic acid	gL^{-1}	0.27	0.15	0.49	0.32	0.17	0.03	0.51	0.31	0.47	0.16	0.11	0.04	0	0	0.88	0.99	0.57	0.18
Lactic acid	gL^{-1}	0.48	0.34	0.55	0.44	0.35	0.22	0.56	0.27	0.11	0.03	0.28	0.11	0	0	0.72	0.30	0.43	0.68
Citric acide	gL^{-1}	0.22	0.18	0.24	0.08	0.10	0.03	0.16	0.06	0.23	0.06	0.10	0.03	0.40	1.14	0.24	0.28	0	0.20
Succinic acid	gL^{-1}	0.58	0.18	0.61	0.18	0.54	0.19	0.33	0.07	0.40	0.11	0.36	0.01	0	0	0.53	0.22	0.35	0.41
Total phenols	mgL^{-1}	157	53	208	70	105	38	289	201	376	231	127	44	8	11	551	633	239	133
Tannic phenols	mgL^{-1}	75	50	96	50	15	4	159	246	163	162	28	23	8	11	143	85	32	49
Non tannic phenols	mgL^{-1}	84	59	118	71	90	34	163	72	213	104	100	21	0	0	408	608	207	84
Catechins	mgL^{-1}	9	5	14	19	5	2	11	4	13	6	18	6	3	2	44	57	7	13
Procyanidins	mgL^{-1}	51	39	67	40	4	2	51	46	71	97	59	57	9	7	203	173	21	134
OD 420 nm		0.10	0.07	0.32	0.35	0.00	0.00	1.28	1.46	1.28	1.01	0.09	0.06	0.21	0.27	0.35	0.15	0.39	0.17
Lightness	%	0.87	0.05	0.81	0.11	0.87	0.00	0.79	0.04	0.35	0.06	0.86	0.02	0.74	0.20	0.77	0.08	0.70	0.80
Saturation	%	7.3	3.4	16.6	13.2	2.3	0.0	15.5	3.1	80.1	4.3	7.2	4.0	12.8	13.1	24.8	9.4	34.4	15.5
Chroma	nm	580	12	577	1	580	0	576	1	583	2	576	0	580	2	576	1	575	575
Iron	mgL^{-1}	4	4	4	4	11	15	7	1	31	44	2	0	1	0	3	1	0	4
Sodium	mgL^{-1}	76	40	81	50	29	1	39	12	71	55	25	18	18	9	38	33	15	26
Calcium	mgL^{-1}	99	68	88	39	86	28	238	59	314	135	174	25	98	49	112	50	26	49
Potassium	mgL^{-1}	649	199	688	144	492	47	757	481	819	539	552	121	45	41	901	247	365	483
Magnésium	mgL^{-1}	64	21	56	18	48	18	45	18	45	14	28	7	21	10	47	16	52	27

(X : mean value . s : standard deviation)

Table 3

Variance analysis and Duncan test for Italian wine vinegars with 6% and 7% acidity and apple vinegars. Same letter indicates vinegar categories that are similar at $p \leq 0.05$

		Ducan Test		
	F value	IWW6	IWW7	AP
Density	4.94**	a	b	ab
Alcohol	19.32**	a	b	a
Total acidity	199.51**	b	c	a
Volatile acidity	105.93**	b	c	a
Fixed acidity	18.66**	a	a	b
Extract	9.49**	a	b	b
Ash	1.71			
Ash alcalinity	12.84**	a	a	b
Glycerol	35.93**	b	c	a
Proline	24.76**	b	b	a
pH	17.28**	b	c	a
Tartaric acid	85.77**	b	c	a
Malic acid	5.78**	a	ab	b
Lactic acid	1.78			
Citric acid	0.11			
Succinic acid	0.48			
Total phenols	7.14**	a	a	b
Tannic phenols	5.53**	a	a	b
Not tannic phenols	4.69*	a	a	b
Catechins	6.11**	a	a	b
Procyanidins	13.42**	a	a	b
OD 420 nm	8.52**	a	b	b
Lightness	6.95**	b	a	a
Saturation	19.29**	a	b	c
Chroma	0.97			
Iron	0.39			
Sodium	4.42*	b	b	a
Calcium	0.61			
Potassium	7.52**	a	a	b
Magnesium	2.84			

during the manufacturing process, whereas potassium levels were higher in cider vinegars.

A comparison between wine and cider vinegars also underlines differences in the levels of phenolic substances, malic acid, extract, tartaric acid, prolin and glycerine, which are all attributable to the different raw materials used.

Maceration during the first phase of cider production justifies the high levels of extract, phenolic substances and leucoanthocyanes in apple vinegars. The high level of procyanidins justify the easy oxidability which is demonstrated by the high optical density of 420 nm.

The acidity of apple vinegars, mainly due to acetic acid, is low when compared with that of wine vinegars. Tartaric acid, of course, is absent whilst malic and lactic acids are present. The high malic acid content of apples is commonly reduced in ciders by malolactic fermentation but the high amount of lactic acid formed is partially oxidised by acetic bacteria. The absence of tartaric acid in cider vinegars as well as in all categories of non-wine vinegars confirms the possible use of this parameter as an origin indicator for vinegars (ANTONELLI *et al.*, 1993).

The results confirm our former data regarding valorization of vinegar products (ANTONELLI *et al.*, 1993). However, the use of multivariate statistical procedures such as linear discriminant analysis (LDA) has been demonstrated to enable a better characterisation of the different categories in situations involving a high number of analytical parameters such as vinegar analysis (see, *e.g.* MORRISON, 1978; POWERS and WARE, 1986).

Since the purpose of the present work was to select a few analytical parameters for characterizing wine vinegars, linear discriminant analysis was applied only to three groups of products: wine vinegars with 6% acidity, wine vinegars with 7% acidity and apple vinegars. About 50% of the samples of each of the three categories under study were used to make up the discriminant model, whilst the rest were used to evaluate the classifying ability of the model. The samples were randomly placed in one of the data groups (NORUSIS, 1985).

All analytical parameters presented in table 2 were used to set up the discriminant model, with the exception of total, volatile and fixed acidity since these parameters are exclusively influenced by technological choices (*e.g.* dilution of concentrated vinegar) and not by raw material. Five principal analytical parameters were found to be discriminating according to the Wilks procedure of LDA (NORUSIS, 1985). These were: tartaric acid, alcohol, proline, saturation and chroma *(table 4)*.

Table 4

Standardized coefficients of discriminant functions between white wine vinegars and apple vinegars

	Function 1	Function 2
Tartaric acid	1.029	0.071
Alcohol	– 0308	0.867
Proline	1.041	0.081
Saturation	– 0.212	0.797
Chroma	0.831	– 0.198

Apple vinegars make up a uniform vinegar category, well discriminated from the rest, while there is a partial overlap between the two categories of wine vinegars since some samples were wrongly classified *(fig. 1)*.

The discriminant model set up with wine and apple vinegars was tested by using the "unknown" vinegars from the other categories (alcohol, malt, French, etc.) which had been excluded from the model. Non-italian wine vinegars were all correctly attributed to the two categories of wine vinegar except for a Swiss vinegar that was wrongly classified as an apple vinegar *(table 5)*. The model also correctly classified decolored vinegars as wine vinegars at 6% from which they are in fact made using the decoloration process. Lastly, alcohol vinegars and vinegars produced from honey and malt, not present in the model, were partly classified as wine vinegars at 6% and partly as apple vinegars but never as wine vinegars at 7%, confirming the peculiarity of these vinegars.

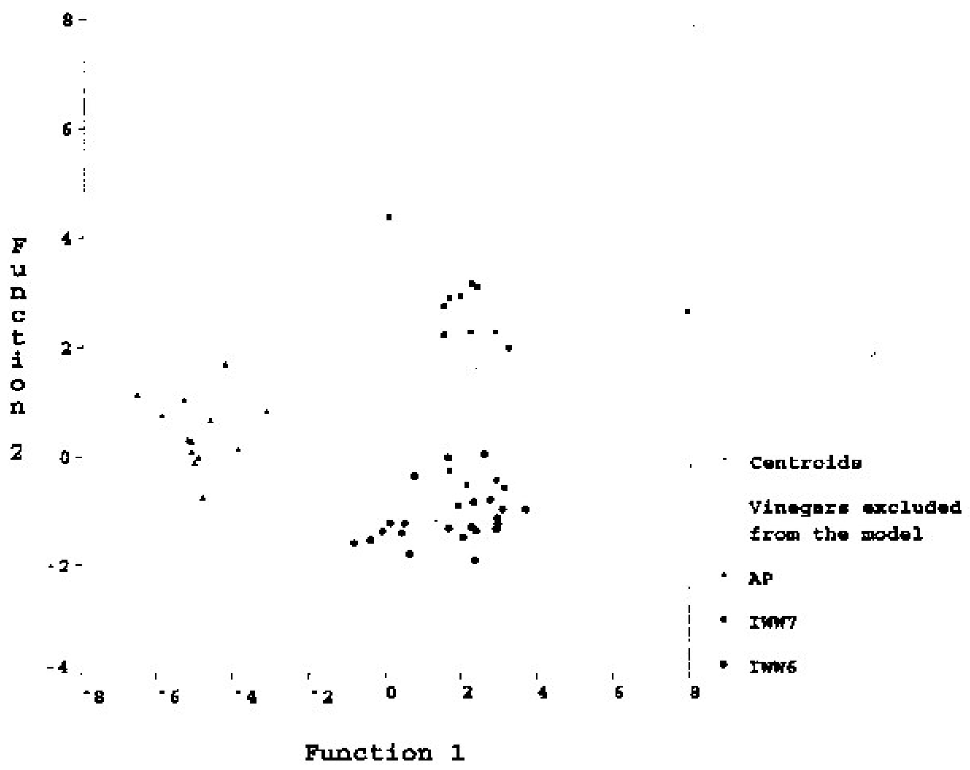

Figure 1

Distribution in the plane characterised by two discriminant functions of wine and apple vinegars (AP = apple vinegar - IWW = Italian white wine vinegar - 6 or 7 = acidity)

Table 5

Classification of vinegar samples from the categories excluded from discriminant analysis

Actual group	N° of samples	Predicted group IWW6	IWW7	AP
IWDE	2	2	0	0
FWW	5	3	2	0
EWW	6	1	5	0
CHWW	2	1	0	1
AL	4	1	0	3
MA	1	0	0	1
HO	1	0	0	1

2.2 Sensory analysis

Sensory evaluations of the 71 vinegars revealed a notable difference among the various product categories, giving rise to three groups, on the basis of the scores attributed by the tasters: the first made up of alcohol, decolored and Spanish vinegars, the second of French and Italian vinegars and the third of the remaining categories.

Wine vinegars were found to be most preferred, while alcohol vinegars that were classified together with discolored vinegars by the tasters were the least liked by the panel. Spanish vinegars, produced in the Jerez area, were also not well liked.

Principal component regression with overall taste evaluation considered as a dependent variable was used to examine the existing relationships between the overall taste evaluation of vinegars and their chemical characteristics. Spanish wine vinegars were excluded for their peculiar characteristics and production techniques. Principal component analysis (PCA) was carried out only on the analytical values. Nine factors were characterized with an eigenvalue higher than 1, which were capable of interpreting 91.7% of the total variance *(table 6)*. By calculating multivariate linear regression to the score values for the principal components characterised, a linear function was obtained ($R^2 = 0.89$; $p \leq 0.05$) *(table 7)*.

Table 6

Loading matrix for wine vinegars.
For each factor only coefficients higher than 0.5 are reported

	Factor 1	Factor 2	Factor 3	Factor 4	Factor 5	Factor 6	Factor 7	Factor 8	Factor 9
Ash alcalinity	0.907								
Potassium	0.907								
Ash	0.896								
Not tannic phenols	0.848								
Procyanidins	0.804								
Total phenols	0.792								
pH	0.667	− 0.534							
Extract	0.652	0.516							
OD 420 nm	0.622								
Glycerol		0.941							
Tartaric acid		0.919							
Total acidity		0.758							
Citric acid		0.718							
Alcohol		0.718				− 0,541			
Density	0.536	0.641							
Copper			0.887						
Lightness			− 0.822						
Saturation			0.815						
Zinc			0.806						
Succinic acid				0.907					
Volatile acidity		0.589		0.719					
Fixed acidity				− 0.701					
Manganese					0.886				
Iron					0.791				
Tannic phenols					0.674				
Sodium					0.675				
Lead						0.822			
Lactic acid						− 0.661			
Chroma						0.501			
Magnesium							0.788		
Calcium							0.747		
Proline								− 0.886	
Catechins								0.726	
Malic acid									0.841

Results show that wine vinegars are more appreciated, if they have lower pH values and higher levels of acidity (fixed, volatile and total), alcohol residue, fixed acids, glycerine, extract, density and phenolic substances. Alcohol vinegars, although possessing high acidity levels, are not well liked because they are very low in ethanol, glycerine, fixed acids, extract and polyphenolic substances.

Table 7

Coefficients and respective statistics of the straight line of multivariate linear regression of the principal components characterised for wine vinegars on overall evaluation

	B	t
Factor 9	− 0.0122	− 0.975
Factor 7	− 0.0162	− 1.685
Factor 8	0.0007	0.081
Factor 5	− 0.0094	− 1.004
Factor 6	0.0339	1.691
Factor 1	0.0538	3.038*
Factor 2	0.0811	4.804**
Factor 4	0.0569	5.125**
Factor 3	0.1151	2.554*
(Constant)	0.7266	38.477**

* $p < 0.05$; ** $p < 0.01$

3 - CONCLUSIONS

An examination of 71 vinegars using linear discriminant analysis, revealed some analytical indicators for characterising wine and apple vinegars.

A larger sampling may reveal new discriminant functions with high levels of classification potential even for those product categories (alcohol, malt, etc.) that were not included here because of the very small number of samples studied.

A comparison between overall sensory evaluation and the analytical results enabled the characterisation of a linear model that links the sensory quality of a vinegar to its chemical composition.

In particular it was shown that, if acidity is constant, assessors preferred products with higher levels of vinosity and extract. Alcohol vinegars, generally considered as being of lower quality compared to wine vinegars were found less appealing.

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