

Comparison of electro dialysis and cold treatment on an industrial scale for tartrate stabilization of sherry wines

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Abstract

Tartrate stabilization on an industrial scale of three sherry wines (“Fino”, “Medium” and “Cream”) has been carried out by means of cold treatment and electro dialysis with the objective of checking the efficacy of these techniques. This was determined by analysing the compounds involved in the treatments and by conductivity techniques for rapid tartaric stability control (saturation temperature and minicontact test). It has been proven that both cold treatment and electro dialysis imparted tartaric stabilization to all the considered wines, with the latter requiring deionisation rates of 26% in Fino wine and less than 20% in Medium and Cream wines. It has been shown that the minicontact test is a valid method to control the efficacy of the treatments, differentiating between stable and unstable wines.

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1. Introduction

One frequent cause of loss of stability of a wine is the formation of crystalline salts of potassium bitartrate (KHT) that appears mainly at low temperatures as a consequence of a large decrease in its solubility (Berg & Keefer, 1958; Ratsimba, Laguerie, Biscans, & Gaillard, 1989).

For the stabilisation of wines the addition of metatartaric acid (Celotti, Bornia, & Zoccolan, 1999; Goertges & Stock, 2000; Ribereau-Gayon, Peynaud, Ribereau-Gayon, & Sudraud, 1977) and proton exchange are sometimes used (Mourgues, 1993). Cold treatment can be applied in different ways (Blouin, 1982; Blouin & De Senne, 1983; Maujean, Vallée, & Sausy, 1986), but the most widespread is holding for one week at a temperature near to the freezing point of the wine. This treatment produces a stabilising effect in dry white wines, while this is not so clear in red and natural sweet

wines (Guitard, 1983; Serrano, Sudraud, & Ribereau-Gayon, 1983). But such cold treatment has high costs and it is time consuming. Electro dialysis is based on the separation of differently charged ions, by the use of selective permeable membranes, under the action of an electric field (Moutounet, Escudier, & Saint-Pierre, 1994; Ribereau-Gayon, Glories, Maujean, & Dubordieu, 1998). These membranes consist of a polymeric matrix, on which ionised functional groups are covalently fixed (Gavach, 1992; Guerif, 1993). The first application of electro dialysis to the tartrate stabilization of wines was carried out by Wucherpfennig (1976), who verified its adaptability to each type of wine. Since then, different studies have demonstrated that electro dialysis treatment stabilises appropriately white, rosé and red wines (Moutounet, Saint-Pierre, Batlle, & Escudier, 1997; Uitslag, Minh-Nguyen, & Skurray, 1996). The effect of electro dialysis treatment depends on the deionisation rate (DR) used which varies between 24% and 26% for white and rosé wines, and from 8% to 13% for red wines (Escudier, Moutounet, & Saint Pierre, 1993; Moutounet et al., 1997). It has also been shown that it does not have any appreciable effects on other

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Nomenclature

CT	cold treatment	Mc	minicontact test
DR	deionisation rate	Ts	saturation temperature in potassium bitartrate
ED	electrodialysis		
KHT	potassium bitartrate		

characteristics of the wine (Biau & Siodlak, 1997; Cottureau, 1993; Riponi, Nauleau, Amati, Arfelli, & Castellari, 1992).

The simplest method to verify the efficacy of a stabilization treatment is to observe the stability of a sample stored at low temperature (Brugirard & Rochard, 1992). But this system is slow, difficult to reproduce and subjective. Tartrate stability can also be determined by means of two tests based on rapid response conductivity techniques. The first one is the saturation temperature (Ts) for KHT of the wine, which globally represents the wine saturation level of this salt, and is measured by the polithermal method (Gaillard, Ratsimba, & Favarel, 1987; García Ruiz, Alcántara, & Martín, 1991). The second method is the minicontact test (Mc) which measures the decrease in the conductivity of a wine kept at low temperature in contact with KHT (Angele, 1992).

Sherry wines are liquor wines elaborated in the south of Spain (C.E., 1988; C.E., 1999), whose manufacture has been broadly described in the bibliography (González Gordon, 1972; Martínez, Pérez Rodríguez, & Benítez, 1997; Pérez Rodríguez, 1991). One of their main problems arises from the use of natural sweet wines, because their high colloids content inhibits KHT crystallisation, during their later tartrate stabilization.

The objective of this work has been to study the application of electro dialysis for tartrate stabilization of Sherry wines to compare its results with cold treatment and to verify its effectiveness for different types of wine.

2. Materials and methods

Trials of application of electro dialysis were carried out on various Sherry wines. For each one of the following types of wine: “Fino” (<2 g/L sugars), “Medium” (40 g/L of sugars) and “Cream” (100 g/L of sugars), different deionisation rates (DR) are applied, the magnitude of which represents the decrease in % of wine conductivity during the treatment. Also, a cold treatment is applied to these wines, which allows one to compare the results of these two techniques. The work is carried out in a commercial cellar of the Denominations of Origin Jerez-Xérèz-Sherry y Manzanilla de Sanlúcar de Barrameda (Spain).

2.1. Description of the industrial plants

For the electro dialysis trials an equipment with a total surface of 25 m² and a treatment capacity of 25 hL/h was used. It had a discontinuous operation regime, circulating the wine between the membrane reactor and a tank, until the wine reaches the desired conductivity, upon which a new cycle begins.

For the cold treatment trials a standard system where the wine is refrigerated to a temperature near its freezing point and kept at this temperature for one week was used. The installation had a refrigeration capacity of 85,000 frigories (one frigorie is equivalent to 1000 calories extracted per hour) and the insulated storage tanks had a volume of 23,000 L.

2.2. Determination of saturation temperature and minicontact test

The determination of Ts and the Mc test uses fully automated equipment. For the Ts determination ramps of temperature between 5 and 30 °C and a KHT dose of 4 g/L were used. In each ramp the conductivity and the temperature were measured, the Ts being determined from the crossing point of the two graphs. For the minicontact test a temperature of 0 °C ± 0.1, a KHT dose of 4 g/L, and a duration between 10 and 18 min, depending on the stability of the sample, were used.

2.3. Analytical determinations

For the determination of the alcohol content, pH, total acidity, volatile acidity free and total SO₂, and sulphates, the European Official Methods of Analysis (Ministerio de Agricultura, Pesca y Alimentación, 1993) were used. Absorbances at 420 and 520 nm were measured by a UV–VIS spectrophotometer with a cell of 10 mm pathlength. Sodium and potassium were determined by flame emission photometry, and calcium and magnesium by atomic absorption spectrophotometry. Tartaric, malic, lactic and acetic acids were analysed by HPLC (Frayne, 1986), with a UV detector at 214 nm. All the determinations were carried out twice, and the arithmetic means of the results are presented.

2.4. Sensorial analysis

Sensory analyses were carried out by five expert tasters of the cellar where the trials were done, using the bilateral test technique in coupled samples (Peynaud, 1987). All the samples treated by electrodiálisis were compared with the corresponding cold treated sample. Each taster indicated their preference in each couple of samples, and carried out general observations on the effects of the considered treatments on the sensory characteristics of the wines.

3. Results

3.1. Effects of the treatments on most common enological parameters

The influence of electrodiálisis treatment on the alcohol content, colour intensity ($A_{420} + A_{520}$), pH, volatile acidity and free and total SO_2 was small or negligible,

while the effect on the titratable acidity was small and in direct relationship with the DR (Table 1a–b), as shown by Moutounet et al. (1997). The cold treatment produced a pH decrease of 0.2 units in the Fino wines, while the maximum decrease produced by electrodiálisis in these wines was only 0.05 units. This small electrodiálisis effect on the pH was due to the opposing actions created by cationic and anionic membranes, because the effect on the pH of the elimination of protons in the cationic membrane was compensated by that of the anions eliminated in the anionic membranes. The cold treatment also reduced the colour intensity to a greater degree, especially in the Medium and Cream wines because of a partial insolubilisation of its colouring matter at low temperature.

3.2. Effects of the treatments on the main cation concentrations

With regard to potassium, the electrodiálisis produced an appreciable reduction in its concentration for

Table 1
Variations in most common enological parameters and tartrate stability during electrodiálisis and cold treatments

Treatments Deionisation rate (%)	Fino							
	Control ^a	ED ^b						CT ^c
		19.6	26.7	30.4	32.3	34.2	40.4	
<i>(a) Fino wine</i>								
Alcohol content (% vol.)	15.1	15.0	15.0	14.9	14.9	15.0	15.0	15.1
A ₄₂₀	0.014	0.013	0.015	0.024	0.019	0.020	0.015	0.015
A ₅₂₀	0.006	0.004	0.004	0.012	0.008	0.009	0.005	0.004
PH	3.18	3.16	3.17	3.13	3.16	3.16	3.14	2.98
Titratable acidity (g/L)	4.23	4.01	3.90	3.90	3.92	3.90	3.83	3.84
Volatile acidity (g/L)	0.16	0.14	0.14	0.13	0.08	0.10	0.15	0.13
Free/total SO ₂ (mg/L)	3/27	1/25	1/27	2/20	1/19	3/20	1/18	3/24
Ts (°C) ^d	23.9	19.7	18.4	16.6	16.3	16.0	13.7	13.0
Mc (μS/cm) ^e	85	61	9	6	1	6	3	1
Tartrate stability ^f	+++	++	–	–	–	–	–	–
		Medium			Cream			
		Control ^a	ED ^b		CT ^c	Control ^a	ED ^b	CT ^c
			20.8	25.2	30.1		18.3	
<i>(b) Medium and Cream wines</i>								
Alcohol content (% vol.)	15.3	15.2	15.2	15.2	15.3	15.52	15.31	15.5
A ₄₂₀	0.055	0.057	0.057	0.058	0.044	0.074	0.069	0.054
A ₅₂₀	0.022	0.023	0.023	0.021	0.020	0.030	0.026	0.017
pH	3.54	3.50	3.52	3.51	3.45	3.43	3.37	3.43
Titratable acidity (g/L)	4.12	4.11	4.07	4.03	4.02	4.09	3.94	3.88
Volatile acidity (g/L)	0.60	0.62	0.72	0.69	0.68	0.65	0.63	0.67
Free/total SO ₂ (mg/L)	3/13	3/11	3/11	1/12	3/12	1/14	1/13	3/12
Ts (°C) ^d	24.4	18.8	18.4	16.6	19.5	21.0	16.6	19.1
Mc (μS/cm) ^e	67	9	5	5	9	9	5	8
Tartrate stability ^f	++	–	–	–	–	–	–	–

^a Control, wine before treatment.

^b ED, electrodiálisis.

^c CT, cold treatment.

^d Ts, saturation temperature.

^e Mc, minicontact test.

^f Tartrate stability: (+++) big sized sediment; (++) medium sized sediment; (–) without sediment.

all tested wines, its amount being directly related to the applied DR (Table 2a–b). Conversely, the effect of the cold treatment on this cation varied in each of the different types of wines, being considerable in the Fino and Medium wines and of significantly reduced magnitude in the Cream wine, owing to the high degree of inhibition of precipitation of KHT in this type of wine. The electro dialysis also produced an appreciable decrease in the concentrations of the Ca^{+2} and Mg^{+2} ions, while during the cold treatment an increase in their concentrations was frequently produced due to the diatomaceous earths used in the filtration after cold treatment. In any case, it should be pointed out that no problems of stability are usually observed in Sherry wines due to calcium. It should also be highlighted that the effect of the electro dialysis on the sodium concentration was very reduced, as shown by Riponi et al. (1992).

3.3. Effects of the treatments on the main anion concentrations

It was observed that electro dialysis produced an appreciable reduction in the sulphate content, its quantity being in direct relationship with the DR used (Table 2a–

b). That is especially interesting in wines with a high sulphate content, such as those where the practice of plaster use in winemaking is carried out, which was the case with the wines used in this work. Plaster use (addition of CaSO_4) is a treatment authorised in Sherry wines to reduce the pH of the musts before fermentation (Gómez Benítez, Grandal Delgado, & Diez Martín, 1993). The effect of the electro dialysis treatment on the tartrate ion was much smaller, 5 meq/L being the maximum decrease in Fino wine against a decrease of 38 meq/L in the sulphate ions in the same wine. This indicates that electro dialysis preferentially separates the sulphate (Wucherpennig, 1976), which can be explained because the sulphate is an anion of a much stronger acid and therefore more active in the membranes than the tartrate. In wines with a low sulphate concentration, the effect on the tartrate concentration would be undoubtedly larger. Cold treatment produced a larger decrease in the tartrate content than electro dialysis (13 meq/L in the Fino wine). Lastly, neither the electro dialysis nor the cold treatment produced appreciable effects on the contents of malate, lactate and acetate at the concentrations of these ions in Sherry wines.

Table 2
Variations in main cation and anion concentrations during electro dialysis and cold treatments

Treatments Deionisation rate (%)	Fino							
	Control ^a	ED ^b						CT ^c
		19.6	26.7	30.4	32.3	34.2	40.4	
<i>(a) Fino wine</i>								
Na (mg/L)	31	30	30	32	30	27	25	30
K (mg/L)	695	535	498	450	425	425	330	520
Ca (mg/L)	102	72	72	71	70	70	67	90
Mg (mg/L)	86	66	60	60	60	60	48	78
Sulphate (g/L K_2SO_4)	2.72	1.63	1.36	1.10	1.14	1.08	0.87	2.72
Tartaric acid (g/L)	2.43	2.33	2.16	2.10	2.10	2.08	2.05	1.43
Malic acid (g/L)	0.177	0.189	0.146	0.153	0.153	0.153	0.146	0.161
Lactic acid (g/L)	0.214	0.238	0.249	0.246	0.230	0.247	0.255	0.263
Acetic acid (g/L)	0.140	0.136	0.130	0.125	0.109	0.115	0.140	0.125
		Medium			Cream			
		Control ^a	ED ^b		CT ^c	Control ^a	ED ^b	CT ^c
			20.8	25.2	30.1		18.3	
<i>(b) Medium and Cream wines</i>								
Na (mg/L)	52	46	41	38	54	31	30	33.8
K (mg/L)	1470	825	755	620	820	705	520	695
Ca (mg/L)	97	97	97	92	120	95	70	120
Mg (mg/L)	84	84	84	78	84	66	46	78
Sulphate (g/L K_2SO_4)	2.52	1.47	1.33	1.21	2.52	2.16	1.27	2.16
Tartaric acid (g/L)	1.55	1.41	1.38	1.33	1.13	1.59	1.41	1.35
Malic acid (g/L)	0.200	0.206	0.192	0.192	0.215	0.132	0.148	0.158
Lactic acid (g/L)	1.397	1.355	1.351	1.341	1.418	n.d.	n.d.	1.406
Acetic acid (g/L)	0.590	0.600	0.650	0.675	0.653	0.590	0.575	0.580

^a Control, wine before treatment.

^b ED, electro dialysis.

^c CT, cold treatment.

Table 3

Sensorial analysis of the wines treated by electro dialysis and cold treatment (number of times that the sample has been preferred)

Treatment	ED ^a	CT ^b	ED ^a	CT ^b	ED ^a	CT ^b	ED ^a	CT ^b	ED ^a	CT ^b	ED ^a	CT ^b
<i>(a) Fino wine</i>												
Deionisation rate (%)	19.6	–	26.7	–	30.4	–	32.3	–	34.2	–	40.4	–
Preferences	1	4	1	4	0	5	0	5	0	5	0	5
	Medium						Cream					
	ED ^a	CT ^b	ED ^a	CT ^b	ED ^a	CT ^b	ED ^a	CT ^b	ED ^a	CT ^b	ED ^a	CT ^b
<i>(b) Medium and Cream wines</i>												
Deionisation rate (%)	20.8	–	25.2	–	30.1	–	18.3	–				
Preferences	1	4	1	4	0	5	2	3				

^a ED, electro dialysis.^b CT, cold treatment.

3.4. Effects of the treatments on tartrate stability

The cold treatment ensured tartrate stability in the three studied wines (Table 1a–b). To get a similar stability with electro dialysis it was necessary to apply a DR value higher than 26% in the Fino wine, while Medium and Cream wines needed DR values lower than 20%. The electro dialysis produced a reduction in Ts directly related to the DR used, while the Mc values decreased to values smaller than 10 $\mu\text{S}/\text{cm}$, and then stayed more or less constant. The cold treatment produced considerable decreases in Ts and Mc mainly in the Fino wines, because of its large effect on the bitartrate ion. The stability criteria for white table wines recommended by the manufacturers of the equipment were used (stable wines when $T_s < 12.5^\circ\text{C}$ or $M_c < 20 \mu\text{S}/\text{cm}$) (Gaillard, Ratsimba, & Favarel, 1990). Then, the Mc gave correct information on stability, with all the stable wines having a value of Mc less than 20 $\mu\text{S}/\text{cm}$. Meanwhile Ts gave a misleading information because following Ts criteria all the treated wines would be unstable.

4. Effect on the sensorial characteristics of the wines

Most of the tasters preferred the cold treated samples (Table 3), indicating that the electro dialysis produced a slight reduction in the aroma and flavour of the wine in relation to the applied DR. In their general opinion the observed reductions of the aroma and flavour were acceptable, whenever the value of the DR of 30% was not exceeded. The aromatic loss taking place can have been produced, at least partly, because the installation was not entirely rendered inert.

5. Conclusions

The treatment of Sherry wines by electro dialysis and cold treatment on an industrial scale produces their tartrate stabilization in an effective way. To obtain an

appropriate tartrate stability a deionisation rate of 20% is applied for the Medium and Cream wines and a rate of 26% for the Fino wines. Electro dialysis reduces the concentration of sulphates more than the tartrates. It is observed that the electro dialysis treatment affects slightly the sensory characteristics of the Sherry wines, depending on the applied deionisation rate, but was acceptable whenever deionisation rates lower than 30% were used.

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