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Prediction of tartrate stability of sherry wines by a conductimetric system with rapid response

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Abstract

The particular production process of Sherry wines means that an appropriate level of tartrate stability is not always reached. The objective of the work described here was to find a rapid and reliable method to determine the tartrate stability of this type of wine. After a broad initial characterization, it was shown that cold-treated Sherry wines remain more oversaturated with potassium bitartrate than white and red table wines. The saturation temperature (Ts) and the result of the minicontact test (Mc) have been correlated with the tartrate stability of different Sherry wines, with the conclusion that wines having Mc values lower than 10 μS/cm remain stable at −4 °C during 1 week. In contrast, a direct relationship between stability and Ts was not found. The minicontact test is therefore proposed as a method for the rapid and reliable determination of the tartrate stability of Sherry wines.

Keywords: Potassium bitartrate; Tartrate stability; Sherry wines; Saturation temperature; Minicontact test

1. Introduction

Sherry wines are liquor wines produced in the south of Spain (C.E. 1988, 1999) and there is a wide range of sherry types that are significantly different in nature (Jerez-Xérèz-Sherry, 1977). The production methods for these types of wines have been widely described in the bibliography (González Gordon, 1972; Martínez, Pérez & Benítez, 1997; Pérez, Flores, Ramírez, & Navarete, 1980). Sherry wines show numerous peculiarities in terms of their composition in relation to tartrate stability and the efficacy of stabilization treatments. The alcohol strength can vary from 15 % vol. in the "Fino" wines to 22 % vol. in some old "Oloroso" wines and as the alcohol content increases the solubility of the potassium bitartrate (KHT) decreases (Ratsimba, Laguerie, Biscans, & Gaillard, 1989). The effect of the high sugar content in some types of wines, which can exceed 100 g/l in the Cream wines, acts in addition to the effect of the alcohol content. These sugars come from the natural sweet wines used in blending processes and are produced with sun-concentrated grape (González Gordon, 1972). The natural sweet wines also contain a high colloid content (Usseglio-Tomasset, Bossia, Delfini, & Ciolfi, 1980), which inhibits the growth of the KHT crystals and hinders stabilization by cold treatment (Celotti, Bornia, & Zoccolan, 1999; Guitard, 1983). The addition of "Vinos de Color" (obtained by flame-heating wines directly) in blends has the same effect and such blending is often carried out to adjust the final color of some wines (González Gordon, 1972). The presence of peptides and glycoproteins should also be considered for Fino and Manzanilla wines as these are released from dead yeast settled in the bottom of the casks during the biological ageing. These compounds have an inhibitory effect on KHT crystallization (Gerbaud, Gabas, Blouin, Pellerin, & Moutounet, 1997; Gerbaud et al., 1996; Lubbers, Leger, Charpentier, & Feuillat, 1993). Likewise, both extraction from the wood of the casks and the concentration effect by evaporation during prolonged ageing increase the potassium concentration in the wines with physical-chemical ageing; e.g. as occurs in Oloroso wines (Pérez Rodríguez, 1991). All of these characteristics of Sherry wines hinder tartrate stabilization by cold treatment.

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Therefore, in some types of Sherry wines, for example the Cream wines, problems associated with stabilization appear frequently and, at present, these problems have not been resolved satisfactorily. As a consequence, it is particularly important to develop a reliable method to measure the efficacy of the cold treatment and to predict the tartrate stability for these wines . The traditional method involves observing the formation of deposits in a sample kept at low temperature, but this method suffers from poor reproducibility and is also time-consuming (Berta, 1993; Brugirard & Rochard, 1992; Colagrande, 1984). For these reasons, systems based on conductimetric measurements with a rapid response, such as that based on the saturation temperature (Ts) of KHT in the wine (García Ruiz, Alcántara, & Martín, 1991), are currently recommended. However, in the case of table wines it has been observed that there is an appreciable difference between the saturation temperature and the crystallization temperature (Maujean, Sausy, & Vallée, 1985; Vallée, Bagard, Bloy, & Bourde, 1990), a problem that precludes the use of such a conductimetric method as a predictive tool. Another prediction system is based on the minicontact test,in which cold wine is kept in contact with KHT and the decrease in the conductivity after a certain time is measured. This system has shown good results for table wines (Angele, 1992).

The objectives of this work were to determine the KHT saturation level of the different types of Sherry wines, to compare these levels with those found in other wines and to measure the effect of the cold treatment in order to verify the difficulties encountered in tartrate stabilization for Sherries. Furthermore, it was envisaged that we could determine the efficacy of the saturation temperature and the minicontact test as systems for the rapid prediction of tartrate stability of these wines.

2. Materials and methods

2.1. Determination of the KHT saturation level

This parameter was measured by the ratio between the concentrations product (CP) and the solubility product (SP) of the KHT. CP is the product of the molar concentrations of potassium (K^+) and bitartrate (HT^-) ions in the wine and was calculated from the K^+ and tartaric acid (H_2T) concentrations, using the values of the pH and alcohol content of the wine, according to the method of Berg and Keefer (1958). SP is the CP of a KHT saturated water-alcohol mixture with the same alcohol content of the wine and was directly obtained from the data of Berg and Keefer (1958) at two temperatures: −4 °C and 20 °C (SP_{-4} and SP_{20} respectively).

2.2. Determination of the saturation temperature and minicontact test

The system STABISAT, which is based on the patent of Gaillard, Ratsimba, and Favarel (1987), and manu-

Table 1
KHT saturation levels in different types of sherry wines

	K (mg/l)	H_2T[a] (g/l)	CP[b]$\cdot 10^{-6}$	CP/SP_{-4}[c]	CP/SP_{20}[d]
(a) *Fino wines*					
Untreated	839±73	2.04±0.20	164.8±17.1	12.2±1.1	1.4±0.1
Treated	639±56	1.30±0.16	68.6±15.7	5.9±1.3	0.6±0.1
t Factor[e]	$9\cdot10^{-5}$	$1\cdot10^{-6}$	$2\cdot10^{-9}$	$3\cdot10^{-8}$	$2\cdot10^{-8}$
(b) *Oloroso wines*					
Untreated	1373±167	1.28±0.18	153.7±14.9	17.4±2.1	1.8±0.2
Treated	1176±120	1.03±0.21	103.0±11.2	11.3±1.2	1.2±0.1
t Factor[e]	$2\cdot10^{-1}$	$7\cdot10^{-2}$	$5\cdot10^{-3}$	$4\cdot10^{-3}$	$4\cdot10^{-3}$
(c) *Medium wines*					
Untreated	930±108	1.52±0.21	124.2±16.4	12.3±2.4	1.3±0.2
Treated	790±91	1.11±0.17	83.4±14.5	8.5±1.4	0.9±0.1
t Factor[e]	$6\cdot10^{-2}$	$1\cdot10^{-2}$	$2\cdot10^{-3}$	$8\cdot10^{-3}$	$4\cdot10^{-3}$
(d) *Cream wines*					
Untreated	839±72	1.77±0.17	146.1±2.9	14.7±2.3	2.0±0.2
Treated	823±57	1.30±0.14	106.2±2.0	11.1±0.7	1.4±0.1
t Factor[e]	$4\cdot10^{-1}$	$2\cdot10^{-3}$	$2\cdot10^{-3}$	$2\cdot10^{-2}$	$2\cdot10^{-3}$

[a] H_2T: tartaric acid.
[b] CP: concentrations product.
[c] SP_{-4}: solubility product at −4 °C.
[d] SP20: solubility product at 20 °C.
[e] *t*: Probability associated with the Student *t*-test.

factured by Bioserae Laboratoires (Montolieu, France), was used to determine these parameters. This system uses the polythermal method, in which the wine conductivity is determined at different temperatures in two samples of the same wine: one of them with 4 g/l of KHT and the other without KHT (García Ruiz et al., 1991). The first measurement is carried out using a heating rate of 2.7 °C/min and the second at 1.7 °C/min. The saturation temperature is taken as the temperature at which the measured values in the two samples are equal. The error in the measurement of the Ts, within the range of alcohol strengths of the Sherry wines, is +0.30 °C (Gómez, Palacios, Caro, & Pérez, 1999). This degree of accuracy is suitable for the characterization and control of the tartrate stability process. In the minicontact test the wine is kept at 0±0.1 °C with 4 g/l of KHT added, during a maximum period of 18 min. The result of the test, named Mc, is the decrease in the conductivity during the test in ?Siemens/cm (Gómez et al., 1999).

2.3. Experimental method

In order to determine the KHT saturation level of Sherry wines, a range of 36 samples were used and these included each of the types Fino, Oloroso, Medium and Cream, with representative samples taken before and after the cold treatment. The samplings were carried out in six different cellars and are fully representative of the Sherry area. A standard cold treatment process is used in all of the cellars and this involves refrigerating the wine at a temperature close to its freezing point and storing it at this temperature during one week; the maximum increase in the wine temperature during the treatment is 2.0 °C/week. The minimum volume of the tanks sampled is 20,000 l. The saturation of the white and red table wines was calculated from literature data (Usseglio-Tomasset, Ubigli, & Barbero, 1992). These data cover 30 white and 33 red table wines. The analytical parameters in these wines were determined before and after the cold treatment. For the determination of the tartrate stability in each type of Sherry wine, samples with a wide range of initial values of Ts and Mc were used. Mixtures of cold-treated and untreated wines in the proportions 100/0, 80/20, 60/40, 40/60, 20/80 and 0/100 were prepared, with the stability of samples kept +4.0±0.1 °C and −4.0±0.1 °C being observed up to 24 months. The stability of the samples was noted on a weekly basis during the first month and at varying intervals until 24 months. The size of the sediment was visually quantified as (+) when there were only a few crystals, as (+ +) when many crystals could be observed, and as (+ + +) when the crystals covered the bottom of the bottle.

2.4. Analytical determinations

The alcohol strength and pH were determined using the European Official Methods of Analysis (C.E.E., 1990). The analysis of K^+ was carried out by flame-emission photometry (C.E.E., 1990) and the tartaric acid content was determined according to the method described by Rebelein (1969).

2.5. Statistical analysis

A significance level of 95% was used to determine the confidence interval. The determination of the *t* Student factor was carried out in coupled samples with two-tailed distribution.

3. Results

3.1. Saturation levels in KHT in the different types of wine and their variation during the cold treatment process

Fino and Oloroso Sherry wines showed the highest initial CP values (Table 1a–d). The Oloroso wines had

Table 2
KHT saturation levels in white and red table wines

	K (mg/l)	H_2T[a] (g/l)	CP[b]·10^{-6}	CP/SP_{-4}[c]	CP/SP_{20}[d]
(a) *White table wines*					
Untreated	540±46	2.43±0.23	128.1±18.6	6.0±1.0	0.75±0.12
Treated	329±29	1.62±0.27	45.7±9.7	2.1±0.4	0.270.06
t Factor[e]	$1·10^{-10}$	$2·10^{-5}$	$1·10^{-10}$	$1·10^{-9}$	$4·10^{-10}$
(b) *Red table wines*					
Untreated	810±57	2.33±0.32	200.1±35.1	10.4±1.9	1.3±0.2
Treated	571±52	1.39±0.32	81.1±24.2	4.3±1.3	0.52±0.16
t Factor[e]	$3·10^{-10}$	$6·10^{-6}$	$8·10^{-9}$	$4·10^{-8}$	$3·10^{-8}$

[a] H_2T: tartaric acid.
[b] CP: concentrations product.
[c] SP_{-4}: solubility product at −4 °C.
[d] SP20: solubility product at 20 °C.
[e] *t*: Probability associated with the Student *t*-test.

lower concentrations of tartaric acid and higher concentrations of potassium than the Fino wines, a situation resulting from KHT precipitation and concentration effects, respectively. The higher potassium concentrations are due to the release of potassium during contact with the cask wood and liquid evaporation through the wood during the long ageing time (Pérez et al., 1980). An expected decrease of the CP values is observed in the cold-treated wines in relation to the untreated wines. This decrease is much larger in the Fino wine (58.2%) than in the Oloroso (33.1%) or Medium (32.7%) wines and is smaller still in the Cream wines (27.2%). These values are not directly related to the initial oversaturation (CP/SP), a fact that indicates an inhibitory effect of the KHT precipitation that increases from the Fino to the Cream wines. The values of the

Table 3
Tartrate stability tests of sherry wines with different values of Ts and Mc

	Storage temperature											
	−4 °C						+4 °C					
(a) *Fino wines*												
Ts (°C)[a]	23.5	21.0	18.9	16.7	14.0	11.3	23.5	21.0	18.9	16.7	14.0	11.3
Mc (μS/cm)[b]	86	53	9	7	3	1	86	53	9	7	3	1
1 week	+ +	+ +	–	–	–	–	+ +	–	–	–	–	–
2 weeks	+ + +	+ +	+	–	–	–	+ + +	–	–	–	–	–
3 weeks	+ + +	+ +	+	–	–	–	+ + +	–	–	–	–	–
4 weeks	+ + +	+ +	+	–	–	–	+ + +	–	–	–	–	–
2 months	+ + +	+ + +	+ +	–	–	–	+ + +	–	–	–	–	–
6 months	+ + +	+ + +	+ + +	–	–	–	+ + +	–	–	–	–	–
18 months	+ + +	+ + +	+ + +	–	–	–	+ + +	+ +	+	–	–	–
24 months	+ + +	+ + +	+ + +	–	–	–	+ + +	+ + +	+	–	–	–
(b) *Oloroso wines*												
Ts (°C)[a]	22.10	20.88	19.55	18.21	16.87	15.30	22.10	20.88	19.55	18.21	16.87	15.3
Mc (μS/cm)[b]	47	30	10	8	6	6	47	30	10	8	6	6
1 week	+	+	–	–	–	–	–	–	–	–	–	–
2 weeks	+ + +	+	–	–	–	–	–	–	–	–	–	–
3 weeks	+ + +	+ +	+	–	–	–	+	–	–	–	–	–
1 month	+ + +	+ + +	+	+	–	–	+ +	+	–	–	–	–
2 months	+ + +	+ + +	+ +	+ +	+	–	+ + +	+	–	–	–	–
4 months	+ + +	+ + +	+ + +	+ +	+	–	+ + +	+ +	+	–	–	–
14 months	+ + +	+ + +	+ + +	+ + +	+ +	–	+ + +	+ + +	+	–	–	–
20 months	+ + +	+ + +	+ + +	+ + +	+ + +	–	+ + +	+ + +	+ +	+	+	+
(c) *Medium wines*												
Ts (°C)[a]	21.60	20.55	19.34	18.12	16.91	15.62	21.60	20.55	19.34	18.12	16.91	15.62
Mc (μS/cm)[(b)]	30	9	6	6	5	5	30	8	6	6	5	5
1 week	+	–	–	–	–	–	–	–	–	–	–	–
2 weeks	+ +	+	–	–	–	–	–	–	–	–	–	–
3 weeks	+ + +	+ +	+	+	–	+	–	–	–	–	–	–
4 weeks	+ + +	+ +	+	+	–	+	–	–	–	–	–	–
2 months	+ + +	+ + +	+ +	+	–	+	+	–	–	–	–	–
6 months	+ + +	+ + +	+ + +	+ +	+ +	+ +	+	+	–	–	–	–
18 months	+ + +	+ + +	+ + +	+ +	+ +	+ +	+ +	+ +	+ +	–	–	–
24 months	+ + +	+ + +	+ + +	+ +	+ +	+ +	+ +	+ +	+ +	+	+	+
(d) *Cream wines*												
Ts (°C)[a]	17.80	17.70	17.62	17.55	17.45	17.40	17.80	17.70	17.62	17.55	17.45	17.40
Mc (μS/cm)[b]	3	3	2	1	0	0	3	3	2	1	0	0
1 week	–	–	–	–	–	–	–	–	–	–	–	–
2 weeks	–	–	–	–	–	–	–	–	–	–	–	–
3 weeks	–	–	–	–	–	–	–	–	–	–	–	–
4 weeks	+	–	–	–	–	–	–	–	–	–	–	–
2 month	+ +	+	–	–	–	–	–	–	–	–	–	–
4 months	+ + +	+ +	+ +	+	–	–	+	–	–	–	–	–
14 months	+ + +	+ + +	+ + +	+ +	+	+	+ +	+	–	–	–	–
20 months	+ + +	+ + +	+ + +	+ +	+ +	+ +	+ + +	+ +	+ +	+	+	+

Results of the tartrate stability test: (–) nothing observed; (+) small sediment; (+ +) medium sediment; (+ + +) large sediment.
[a] Ts: saturation temperature of potassium bitartrate.
[b] Mc: result of the minicontact test.

Student *t*-factor for the different parameters are much smaller in the Fino wines than in the other Sherry wines, which confirms the data discussed above. Furthermore, the *t* factors are much smaller in the table wines than in Sherry wines, demonstrating a more marked effect of the cold treatment in these wines (Table 2a,b). The CP values of KHT are similar to those found in Californian Sherry-type wines (De Soto & Yamada, 1962).

3.2. KHT oversaturation in the cold treated wines

In the cold-treated Sherry wines the oversaturation levels in KHT at −4 °C are much higher than unity, with mean values between 5.9 and 11.3 (Table 1a–d). However, it should be noted that during the stabilization treatment these wines are kept at temperatures between −6 and −9 °C during 1 week. These saturation levels are much higher than those found in white table wines (mean value 2.1) and red table wines (mean value 4.3) (Table 2a,b). The decrease in the ratio CP/SP during the cold treatment is similar to the trend found for CP, with the decrease being more pronounced in the Fino wines and less marked in the Cream wines. At 20 °C the KHT saturation levels of Sherry wines approach unity, varying from 0.6 in Fino wines to 1.4 in Cream wines. These results are in agreement with those of Berg and Keefer (1958), who found in table wines treated at −4 °C values of the ratio CP/CS_{20} varying from 0.96 to 1.19.

3.3. Relationship between tartrate stability, time and storage temperature

It was observed in some cases that the formation of KHT sediments takes a long time (Table 3 a–d), especially in Cream wines and Sherries with low Ts values. In all cases it was found that the size of the sediment increased with time and that, after a time period that depended on the sample in question, most of the samples did form KHT sediment. The temperature of the test had a great influence on the results, as demonstrated by the fact that deposit formation occurs a −4 °C before it occurs at +4 °C. Therefore, the absolute tartrate stability does not exist, rather the conservation conditions (time and temperature) should be specified.

3.4. Relationship between tartrate stability, saturation temperature and the minicontact test

In cellars a wine is usually considered stable when sediment is not formed after storage for one week at low temperature. Indeed, a relationship between the stability of the wine under these conditions and the values of Ts and Mc has been the subject of research interest. It can be observed (Table 3a–d) that the wine stability does not show any direct relationship with Ts, and wines with similar values of Ts show different tartrate stability. It is the case of the Oloroso wine with a Ts of 20.88 °C which is unstable at −4 °C, and the Medium wine with a Ts of 20.55 °C wich is stable at the same temperature. That confirms previous observations (Maujean et al., 1985; Vallée et al., 1990). However, the stability does have a high degree of correspondence with the Mc value because the wines with Mc values higher or equal than 80 μS/cm form sediment at −4 or +4 °C within 1 week. The wines with Mc values of about 50 μS/cm form sediment within one week at −4 °C but not at +4 °C, and the wines with Mc values lower than 10 μS/cm require more than 1 week to form sediment at −4 °C and a much longer time at +4 °C. The maximum values of Mc that give acceptable tartrate stability during one week at −4 °C for the different types of sherry wines are 9 μS/cm for Fino wines, 10 μS/cm for medium wines, 9 μS/cm for Medium wines and 3 μS/cm for cream wines. These values are in accordance with those published by Gomez, Palacios, Skely, Veas, and Pérez (in press). As can be observed, a value of 10 μS/cm can be taken as the limit for tartrate stability for all of the sherry wines considered. In other words, a wine with an Mc value equal or lower than 10 μS/cm will not form sediment under the stated conditions.

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