Tartaric stabilization of a Reserve Ruby Port Wine by cold and electrodialysis processes: Effect on chemical and sensory characteristics

Nuno Jorge\(^a\), Alice Vilela\(^a\)*, António Braga\(^b\) Fernando M. Nunes\(^c\), Fernanda Cosme \(^a\)

\(^a\) Institute for Biotechnology and Bioengineering, Centre of Genomic and Biotechnology, (IBB/CGB-UTAD), University of Trás-os-Montes and Alto Douro, 5001-801 Vila Real, Portugal.
\(^b\) Sogrape Vinhos, S.A., Departamento de Enologia | Winemaker, 4430 - 809 Avintes, Portugal
\(^c\) Chemical Research Centre (CQ-VR) University of Trás-os-Montes and Alto Douro, 5001-801 Vila Real, Portugal.

*avimoura@utad.pt

**Keywords**: Port Wine, tartaric stabilization, cold stabilization, electrodialysis, sensory analysis

**ABSTRACT**

Cold stabilization is the most common process for wine tartaric stabilization. This process consists in cooling the wine at temperatures near the wine freezing point, to induce potassium bitartrate crystals precipitation. However, this is a slow and costly operation. Electrodialysis is a technique that allows the separation of anions and cations according to their electrical charge through a selective semi-permeable membrane in which a direct current is applied between two electrodes. This system can be used to remove or reduce calcium and potassium, therefore electrodialysis can be used to stabilize the wine against tartaric instability. So, the focus of this work was to determine if electrodialysis could be an alternative for cold stabilization of a Reserve Ruby Port Wine. The results indicated that wines treated by cold stabilization showed a significant decrease on colour intensity, total anthocyanins, polymeric and total pigments. The wines treated by electrodialysis showed a significant increase in limpidity and a significant decrease in sodium, potassium and calcium content. Wines sensory analysis revealed that those treated by electrodialysis had higher limpidity, colour intensity, persistency, balance, flavour and aromatic quality. This study provides information that could be used to select an appropriate process for wine tartaric stabilization.

1. **INTRODUCTION**

Cold stabilization is a method of separating unstable natural ionic salts from wine by cooling the wine at temperatures near the wine freezing point [1]. Precipitation occurs due to the instability of tartaric acid in its bitartrate salt form, which is known as potassium hydrogen tartrate (KHT) as a supersaturated solution. Cold stabilization may have some disadvantages, since it is a process with high energy and equipment costs [2]. During the time at low temperatures, a rapid precipitation of wine potassium bitartrate crystals will occur that is easily removed by filtration at low temperature to prevent the redissolution of the crystals [3]. Electrodialysis is a process that allows the separation of anions and cations according to their electrical charge using a charged selective membrane sheets under the influence of a charged current [4]. Electrodialysis is ideal for removing mineral cations such as potassium, calcium,
sodium and tartaric acid. Polyphenols, polysaccharides and amino acids are unaffected, as well as colloidal material of red wines [4]. Electrodialysis has advantages over the traditional methods of wine tartaric stabilization, such as, no influence on wine sensorial characteristics [5]. Therefore, the aim of this study was to evaluate alternative processes for cold tartaric stabilization such as electrodialysis of a Reserve Ruby Port Wine

2. MATERIAL AND METHODS

2.1. Wine Samples

A Reserve Ruby Port Wine (Douro Region) obtained with traditional grape varieties such as Touriga Francesa, Tinta Roriz, Touriga Nacional, Tinta Barroca and Tinto Cão was used. It presented the following characteristics: Specific gravity at 20º C (g/cm³) 1.024; alcohol content (% vol.) 19.9; titratable acidity (g/L tartaric acid) 4.9; volatile acidity (g/L acetic acid) 0.3; pH 3.5. Analysis of conventional oenological parameters were performed according standardized procedures [6].

2.2. Cold stabilization and electrodialysis experiments

Tartaric stabilization of a Reserve Ruby Port Wine by a cold stabilization process and electrodialysis was carried out at a semi-industrial scale, with and without addition of Arabic gum. For the cold stabilization process, wine was cooled to a temperature of -6 ºC, and placed into tanks of 250 L, for 4 days and for 7 days. At the end of both processes the wines were filtered (plates - KS Seitz 50). Arabic gum (40 g/hL) was added after filtration to the wine that was cold stabilization during 4 days and to one part of the assay carried out with the electrodialysis process. Finally the wines were bottled for physicochemical and sensory evaluations. All experiments were carried out in duplicate.

2.3. Colour intensity and hue, total and coloured anthocyanins, polymeric and total pigments

Colour intensity and hue was determined by O.I.V. [6] method. Total and coloured anthocyanins, polymeric and total pigments according to Somers and Evans [7]. All analyses were performed in duplicate.

2.5. Monomeric anthocyanins profile

Performed by Dionex UltiMate 3000 HPLC-DAD. Reverse phase C18 column (25cm, 4.5mm diameter, 5μm particles). The eluent was constituted by 5% aqueous formic acid (solvent A) and methanol (solvent B). The elution program was: 5% of B from zero to 5 min followed by a linear gradient up to 65% of B until 65min and from 65 to 67min down to 5% of B. The flow rate was 1mL/min. Injection volume 25μL. The identification was made considering their retention times and UV spectra. The chromatograms were recorded at 525 nm. All analyses were performed in duplicate. All analyses were performed in duplicate.
2.6. Calcium, sodium and potassium

Calcium, sodium and potassium were analyzed by flame atomic absorption spectrophotometry according to OIV [6]. All analyses were performed in duplicate.

2.7. Sensory evaluation

The sensory analysis was performed by a trained panel of 7 judges specialized in this type of evaluation. All evaluations were conducted from 10:00 to 12:00 a.m. in an individual booth and according to standardized procedures [8]. Eight sensory attributes were evaluated including visual, aromatic, and taste attributes.

2.8. Statistical analysis

Both physicochemical and sensory data were statistically tested by analysis of variance (ANOVA) using the software Statistica 2010 (StatSoft Inc., 2010).

3. RESULTS AND DISCUSSION

Wines treated by cold stabilization method had a significant decrease on colour intensity, coloured and total anthocyanins, polymeric and total pigments. This treatment was responsible for removing pigments and compounds responsible for the red wine colour (Table 1)

Table 1 – Colour intensity, hue, total and coloured anthocyanins, polymeric and total pigments for both untreated Reserve Ruby Port wine and for Reserve Ruby Port wine after cold stabilization and electrodialysis stabilization (mean ± SD).

<table>
<thead>
<tr>
<th>Stabilization methods*</th>
<th>Color intensity (mg/L)</th>
<th>Hue</th>
<th>Total anthocyanins (mg/L)</th>
<th>Colored anthocyanins (mg/L)</th>
<th>Polymeric pigments (mg/L)</th>
<th>Total pigments (mg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T</td>
<td>1.98±0.00</td>
<td>0.842±0.000</td>
<td>231±0</td>
<td>2.52±0.00</td>
<td>6.49±0.00</td>
<td>14.73±0.01</td>
</tr>
<tr>
<td>F7</td>
<td>1.27±0.00</td>
<td>0.687±0.000</td>
<td>91±1</td>
<td>1.86±0.02</td>
<td>4.87±0.01</td>
<td>10.81±0.00</td>
</tr>
<tr>
<td>F4</td>
<td>1.34±0.00</td>
<td>0.693±0.001</td>
<td>101±1</td>
<td>1.96±0.04</td>
<td>5.18±0.04</td>
<td>12.22±0.00</td>
</tr>
<tr>
<td>E</td>
<td>1.61±0.00</td>
<td>0.677±0.000</td>
<td>107±1</td>
<td>2.35±0.00</td>
<td>6.26±0.03</td>
<td>13.38±0.07</td>
</tr>
<tr>
<td>EGA</td>
<td>1.66±0.00</td>
<td>0.682±0.001</td>
<td>109±0</td>
<td>2.38±0.13</td>
<td>6.49±0.14</td>
<td>14.14±0.00</td>
</tr>
</tbody>
</table>

*Untreated wine (T); four days cold with arabic gum (F4); seven days cold (F7); electrodialysis with arabic gum (EGA); electrodialysis (E); means within a column followed by the same letter are not significantly different (Tuckey, 5%).

Moreover, wines treated by electrodialysis presented a significantly higher limpidity and a significantly decrease on tartaric acid, potassium and calcium content. The HPLC analysis showed that no significant differences between the wines treated by cold stabilization and electrodialysis method were observed regarding monomeric anthocyanins. This could be related with the fact that a loss of monomeric anthocyanins occurred during Ruby Port wines aging and an increase in the concentration of polymeric pigments. As using HPLC it is possible to quantity only the concentration of monomeric anthocyanins.
Sensory analysis of the Reserve Ruby Port wine showed that wines treated by electrodialysis process (Figure 1) are more scored for limpidity, colour intensity, persistency, balance, flavour intensity and aromatic quality and intensity.

![Sensory profile graph](image)

Figure 1 – Sensory profile for untreated Reserve Ruby Port wine and for Reserve Ruby Port wine after cold stabilization and electrodialysis stabilization.

Based on the results, it is possible to suggest that electrodialysis technique could provide a possible alternative to cold stabilization process for Reserve Ruby Port wine tartaric stabilization.

**Acknowledgments**

This work was partially funded by IBB/CGB-UTAD and Chemistry Research Centre (CQ-UTAD). Additional thanks to SOGRAPE VINHOS, S.A., for the availability of wines, equipment and analysis.

**References**