PROTEIN STABILIZATION OF WHITE WINES BY STABILIZING FILTRATION: PILOT STUDIES •

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Key words: protein stabilization, zirconium, adsorption.

1. INTRODUCTION

Heat-unstable soluble proteins present in grapes, grape juices and wines may become insoluble and precipitate causing the formation of undesirable hazes or deposits in white wines after bottling and during storage. This is commonly prevented using bentonite, though this technique presents some drawbacks, like adverse effect on the sensory properties of wine, loss of wine volume as lees, long working time and waste disposal problem (Waters *et al.*, 2005).

Zirconia is a metal dioxide of special interest due to its surface properties, which allow it to be employed commonly as catalyst or support material (Mallick *et al.*, 2006). We have been studying a new method to remove unstable proteins from white wine by a continuous adsorption process packed in column using zirconia as adsorbent material operating in open loop (Pashova *et al*., 2004a, b; Salazar *et al*., 2006, 2007).

The aim of this work was to extend the study of white wine protein stabilization in a continuous process operating in a closed loop with zirconium oxide in a pilot scale for three different monovarietal wines of 'Xarel.lo', 'Chardonnay', and 'Muscat', and to analyse the effect of this treatment on the total protein concentration and stabilization.

• QUAD. ViTIC. ENOL. UNIV. TORINO, 31, 2009-2010

2. MATERIALS AND METHODS

2.1. Wine samples

Three monovarietal white wines were used in this study corresponding to 'Xarel.lo', 'Chardonnay', and 'Muscat' cultivars, vintage 2008. Xarel.lo, Chardonnay and Muscat wines were elaborated by the Bellvei Agricultural Cooperative, Tarragona, Spain. The wines were elaborated with must clarified by settling. The fermentation was controlled at 18 °C in industrial scale (50 000 L). The wine samples were used immediately after fermentation and settling stages with no additional treatment.

2.2. Equipment

The shape of zirconia pellets (Saint Gobain NorPro, Staw, OH, USA) was small disks with a diameter of 3 mm and a thickness of approximately 1 mm, a pore size of 3.6 nm, a surface area of 241 m^2 g^{-1} and with amorphous morphology. It was thermally treated (500 ºC for 12 h) to modify to tetragonal morphology and packed (6.5 L) in a stain steel column (0.50 m high and with an internal diameter of 129 mm).

Wine samples were pumped, up-flow mode, through the column by a centrifugal pump at a recirculation flow rate constant of 300 L h^{-1}, operating in a closed loop for all experiments. The total protein concentration and protein stability were determined in function of time of treatment.

2.3. Analysis and characterization

Superficial properties of ZrO_2 were determined by the adsorption model of Brunauer-Emmett-Teller (BET) and morphology of the material by X ray diffraction (XRD). Total protein concentration was measured by Bradford method. The wine protein fractions before and after each treatment were evaluated by gel permeation chromatography, using the method described by Czekaj *et al.*, 2001. Protein stability was determined by thermal test.

3. RESULTS

The initial content of total wine proteins was 24.2 ± 0.8 mg BSA L^{-1} for Xarel.lo wine, 26.9 ± 0.5 mg BSA L^{-1} for Chardonnay wine, and 22.2 ± 0.1 mg BSA L^{-1} for Muscat wine.

After the stabilizing filtration process, thermal stability tests showed that wines were stable, and their total protein concentration had been reduced, with no negative effects on quality.

The total time to achieve the protein stabilization of Xarel.lo, Chardonnay, and Muscat wines was 8, 24 and 139 hours, respectively, with a total protein content

of 13.4 ± 1.6 mg BSA L^{-1}, 14.6 ± 1.8 mg BSA L^{-1} and 12.1 ± 0.1mg BSA L^{-1}, respectively.

During the stabilizing process, the protein reduction in all wines was similar. Protein content decreased more at the beginning, and remained practically constant after reaching the stability point.

The results show that there is no relation between stabilizing time and total protein content, even though the initial total protein content was similar in all wines.

HPLC analyses of the macromolecular profile allowed to identify four macromolecular fractions in all wines: around 15 kDa, 25 kDa, around 67 kDa and > 190 kDa, although the 67 kDa fraction was not found in Chardonnay wine.

The 15 kDa, 67 kDa and > 190 kDa protein fractions seem not to be related to protein stability, because they stabilize in a different moment than when the wine reaches protein stability.

The 25 kDa fraction was found in all three wines and was directly implicated in protein stability because the stability point of wines matched the lowest value of this fraction. Similar results were obtained with Chardonnay and Muscat wines at laboratory scale by Pashova *et al*. (2004 a, b).

Acknowledgments

The authors thank the Cooperativa de Bellvei, Tarragona (Spain) and Oriol de Guevara for donating the wines used in this work. M. Lira would like to thank for the project scholarship to the Generalitat de Catalunya (2005SGR-01066). Finally the authors are also grateful for the financial support provided by the Spanish Ministry of Education and Science (AGL2006-07034/ALI).

Abstract

Protein stabilization is an important part of the winemaking process of white wines, and in this work we present the results of protein stabilization of different monovarietal wines (Xarel.lo, Chardonnay, and Muscat) by a continuous stabilizing filtration process using a column packed with zirconium oxide operating in a continuous regime in a closed loop at pilot scale. The treatments in these conditions allow stabilizing the wines and reducing their protein content. No relation was found between the time necessary to stabilize the wine and the total protein content. The protein profile of the wine along the treatment shows four different fractions of macromolecules, of 15 kDa, 25 kDa, around 67 kDa and >190 kDa, except the 67 kDa fraction not found in Chardonnay. A fraction of 25 kDa was found in all three wines and could be directly involved in protein stability because the stability point of wines matched the lowest value of this fraction.

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