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Isabel red wines produced from grape pre-drying and submerged cap winemaking: A phenolic and sensory approach

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ABSTRACT

The aim of this work was to determine the detailed phenolic composition and sensory profile of red wines produced from Isabel grape using two alternative winemaking: grape pre-drying (IPD) and submerged cap (ISC). IPD wines were produced by the grape drying using a tray dryer at 60 °C and 1.1 m/s of airflow and the ISC wines were produced using stainless steel screens inside the fermentation vessel aiming at avoiding the rise of the cap due to the carbon dioxide. As expected by the thermal degradation, IPD wines presented not quantifiable concentration of anthocyanins and were described as bitter, acid, herbaceous and astringent due to their higher content of galloylated (6.94 mg/L), monomeric flavan-3-ols (49.66 mg/L) and proanthocyanidins (6.28 mg/L), which were less affected by thermal degradation. Submerged cap wines were described as colorful, pungent and persistent in mouth probably due to the anthocyanin content (2.43 mg/L) and hydroxycinnamic acid derivatives (280.5 mg/L), respectively. Submerged cap is a promising procedure because these wines presented higher yield and anthocyanin content and color intensity similar to traditional wines; pre-drying winemaking can be considered less promising since it presented lower yield and sensory features that are not very appreciated by the consumers.

1. Introduction

Table wines are produced from American grapes and their hybrids (*Vitis labrusca L.*) and, in Brazil, exceeded the production of wines produced from European grapes. Isabel grape (*Vitis labrusca L.* x *Vitis vinifera*) plays an important role in the production of red wines and derivative products, since it accounts for approximately 50% of Brazilian grape production (Nixdorf & Hermosín-Gutiérrez, 2010). The wine elaborated from this grape cultivar presents foxy and raspberry aroma and flavor, features that are very appreciated by Brazilian consumers (Rizzon, Miele, & Meneguzzo, 2000).

To date, there has been little published research on the Isabel wine phenolic composition and they have revealed a discreet anthocyanin and pyranoanthocyanin content ranging from 149.8 to 212.8 mg/L and 12.9–20.8 mg/L, respectively (Nixdorf & Hermosín-Gutiérrez, 2010) and another study reported the anthocyanin content of Isabel juices ranging from 102.9 to 216.0 mg/L (Yamamoto et al., 2015). The few mentioned studies were focused only on the phenolic composition of Isabel red wines or juices and presented no data related to sensory descriptors. In addition, no studies were found dealing with Isabel red table wines produced from variations in winemaking procedures.

Despite the fruity flavor and rusticity of the American grapes, the low soluble solids content in their optimal stage of ripening and the low color potential of these grape cultivars are problems that need to be solved by the winemakers. In this context, wineries have used alternative winemaking procedures aiming at enhancing the phenolic extraction, mainly anthocyanin, in order to produce colorful red wines, responding positively in the sensory acceptance. In general, winemakers have employed several variations on winemaking by the application of drying process of the grapes (Marquez, Serratosa, Lopez-Toledano, & Merida, 2012) and

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submerged cap during alcoholic fermentation (Bosso et al., 2011; Suriano, Ceci, & Tamborra, 2012).

Studies dealing with grape drying prior fermentation showed that the heating caused an irreversible damage in the cellular structure of the grape skin increasing the extraction of the phenolic compounds to the wine during maceration (Marquez et al., 2012). In contrast, the thermal degradation of anthocyanins is a well-known phenomenon that could occur in parallel to the phenolic extraction enhancement (Patras, Brunton, O'Donnell, & Tiwari, 2010). Additionally, submerged cap winemaking procedure provides a balance between gains and losses concerning phenolic concentration, i.e., the constant contact between pomace and must causes gains in the phenolic concentration, mainly anthocyanins (Bosso et al., 2011); however, the absence of mechanical pumping or punching-down steps during maceration negatively affects the flavan-3-ol concentrations (Suriano et al., 2012). All the above mentioned studies presented relevant results; however, they presented data regarding *Vitis vinifera* red wines and provided no relationships with sensory data.

In this context, the aim of this work was to evaluate the detailed phenolic composition and the antioxidant activity of Isabel red wines produced from two alternative winemaking procedures: pre-drying and submerged cap in comparison with the traditional winemaking procedure employed in Brazilian wineries. In addition, wine sensory descriptors were associated with the chemical data as a result of a multivariate chemometric approach aiming at assessing the potential of the alternative winemaking procedures.

2. Material and methods

2.1. Chemicals

All solvents were of HPLC quality, all chemicals were of analytical grade (>99%) and the water was of Milli-Q quality. Commercial standards from Phytolab (Vestenbergsgreuth, Germany), Extrasynthese (Genay, France) and Sigma Aldrich (Tres Cantos, Madrid, Spain) were used for analysis. Other non-commercial flavonol standards were previously isolated from Petit Verdot grape skins (Castillo-Muñoz et al., 2009). Procyanidin B4 was kindly supplied by Prof. Fernando Zamora (Department of Biochemistry and Biotechnology, Universitat Rovira i Virgili, Spain). The *trans* isomers of resveratrol and its 3-glucosides (piceid) were converted into their respective *cis* isomers by UV irradiation (366 nm light for 5 min in quartz vials) in methanol:water 250/750 mL/mL solution.

All the standards were used for identification and quantitation by calibration curves covering the expected concentration ranges. When a standard was not available, the quantitation was done using the calibration curve of the most similar compound: malvidin 3,5-diglucoside for 3,5-diglucoside anthocyanin type and malvidin 3-glucoside for the 3-glucoside type, quercetin 3-glucoside for flavonol 3-glycosides and their free aglycones, caffeic acid for hydroxycinnamic acid derivatives, (+)-catechin for polymeric flavan-3-ols (total proanthocyanidins), and individual flavan-3-ol monomers and dimers by their corresponding standards considering their total sum as (+)-catechin equivalents.

2.2. Microvinification

Three red wines were produced in duplicate: Traditional Isabel wine (IT), Pre-dried Isabel wine (IPD) and Submerged Cap Isabel wine (ISC). The grapes were harvested in the city of Jales (20° 16′ 7″ South and 50° 32' 58" West), São Paulo state, Brazil, and they presented, at the start of the winemaking procedure, soluble solids content of 16.4 ± 1.0°Brix, pH value of 3.38 ± 0.03 and total acidity of 5.90 ± 0.07 g.L^{-1} as tartaric acid equivalents.

All the treatments followed the standard winemaking procedure described by De Castilhos, Conti-Silva, and Del Bianchi (2012). Two batches of 7 kg grape each were used for the microvinification process which contemplates the addition of sulfur dioxide (0.086 g/L) and dry active *Saccharomyces cerevisiae* yeasts Y904 (Amazon Group®) in the proportion of 0.2 g/L in order to induce the alcoholic fermentation.

The submerged cap treatment maintained the cap at the bottom of the fermentative vessel by using stainless steel screens, avoiding its rise due to the production of carbon dioxide. The pre-drying treatment consisted of drying the grapes to 22°Brix to avoid chaptalization and obtain wines with an alcoholic content between 8.6 and 14%v/v, as required by Brazilian legislation (Brasil, 2004). This winemaking process was carried out using a convective drying method with a tray dryer at 60 °C and airflow of 1.1 m/s (De Castilhos et al., 2012). At the end of drying procedure, Isabel wines presented 22.9°Brix, with 22.2% of the water evaporated in relation to the initial weight. Both traditional and submerged cap de-stemmed grapes were chaptalized by the addition of 52.2 g/L of sugar.

The following conventional enological parameters were measured: total and volatile acidities as g/L tartaric and acetic acid equivalents, respectively and pH (Brasil, 2004); total dry extract (g/L) (AOAC, 2005); reducing sugars (g/L) by the Lane-Eynon method (AOAC, 2005), alcoholic content (% vol/vol) (AOAC, 2005) and total phenolic content using gallic acid as standard (Slinkard & Singleton, 1977).

2.3. Analysis of the phenolic compounds

2.3.1. Preparation of the wine for the determination of the non-anthocyanin phenolic compounds

The flavonol fractions were isolated from diluted wine samples following the procedure described by Castillo-Muñoz, Gómez-Alonso, García-Romero, and Hermosín-Gutiérrez (2007), using Bond Elute Plexa PCX solid phase extraction cartridges (Agilent®; 6 cm^3, 500 mg of adsorbent, Waldbronn, Germany). The flavan-3-ols (monomers, B-type dimers and polymeric proanthocyanidins) and stilbenes were isolated following the procedure described by Rebello et al. (2013), using SPE C18 cartridges (Waters® Sep-Pak Plus, filled with 820 mg of adsorbent, Saint-Quentin En Yvelines, France).

2.3.2. HPLC-DAD-ESI-MS^n analysis of the phenolic compounds

The HPLC separation, identification and quantitation of the phenolic compounds was carried out on an Agilent 1100 Series HPLC system (Agilent®, Waldbronn, Germany) equipped with DAD (G1315B) and a LC/MSD Trap VL (G2445C VL) electrospray ionization mass spectrometry (ESI-MS^n) system, coupled to an Agilent ChemStation (version B.01.03) data-processing unit. The mass spectra data were processed using the Agilent LC/MS Trap software (version 5.3). The anthocyanin, flavonols and hydroxycinnamic acid derivatives (HCAD) were analyzed according to a previously described method (Lago-Vanzela, Da-Silva, Gomes, García-Romero, & Hermosín-Gutiérrez, 2011). The wine samples were injected (10 μL for anthocyanins and 20 μL for flavonols; flow rate at 0.19 mL/min) onto a Zorbax Eclipse XDB-C18 reversed-phase column (2.1 × 150 mm; 3.5 μm particle; Agilent, Germany) with the temperature controlled at 40 °C.

For anthocyanin and flavonol analysis, the eluents used were as follows: solvent A (water/formic acid/acetonitrile 885/85/30 mL/mL/mL); solvent B (acetonitrile/water/formic acid 500/415/85 mL/mL/mL) and solvent C (methanol/formic acid/water 900/85/15 mL/mL/mL). For both analysis, it was performed a gradient elution; for anthocyanin analysis the gradient was as follows: 0 min (97% A and