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Fining with purified grape pomace. Effect of dose, contact time and varietal origin on the final wine phenolic composition

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ABSTRACT

Fining, which involves the addition of adsorptive material in order to reduce or eliminate certain unwanted components, is a common winemaking practice. Fining agents affect the wine phenolic compounds, some of which may be reduced. When this reduction is experimented by the tannins, a positive effect may result by decreasing astringency in the wine, although a decrease in the wine color may also take place when the anthocyanins are involved, affecting its quality. Recently, grape cell wall material has been tested as a potential fining agent in wines, since it shows a high affinity for tannins so that its use could reduce wine astringency. In this work, the use of purified grape pomace as fining agent is proposed and the effect of different doses and contact times on wine chromatic characteristics was investigated as well as how differences in the composition of the purified pomace could alter the phenolic composition of a red wine. The results showed that a Monastrell purified grape pomace dose of 6 mg/ml and a contact time of 5 days could be suitable for decreasing the wine tannin content without producing great changes in the wine chromatic characteristics. When comparing the effect of purified pomaces from four grape varieties, some differences in their capacity to interact with the wine tannins and anthocyanins were found, however, the results confirm that the purified grape pomace, a byproduct of the enology industry could be a new interesting fining material.

1. Introduction

Fining is a technique that is used to remove unwanted wine components. Sometimes, an excessive extraction of phenolic compounds during winemaking may affect clarification, astringency, color and bitterness. The fining agent reacts with wine components either chemically or physically, to form a new complex that can separate from the wine. Traditionally, proteins such as gelatin, egg albumin, casein and isinglass have been used for fining tannin compounds, due to their high affinity for proanthocyanidins. However, these proteins are of animal origin and their presence could cause allergenic reactions in sensitized individuals (Peñas, di Lorenzo, Uberti, & Restani, 2015).

Lately, those fining agents of vegetal origin have become more popular as substitutes of animal-origin potentially allergenic proteins and to avoid the legal obligation of indicating their presence on the label (Vassilopoulou et al., 2011). Moreover, some strict vegetarians, such as vegans, do not accept any beverage treated with products of animal origin. Consequently, various fining agents derived from plants, including proteins from cereals, legumes, and potato (Gambuti, Rinaldi, & Moio, 2012) have been proposed as an alternative. Protein extracts from yeast (Iturmendi, Durán, Marín-Arroyo, & Marin-Arroyo, 2010; Lochbühler et al., 2015) and seed extracts from grape (Gazzola, Vincenzi, Marangon, Pasini, & Curioni, 2017) have also been shown a valid fining agent for the treatment of red wines to decrease their astringency.

Also, grape marc cell walls have been tested as new fining agents for their ability to bind phenolic compounds (Bautista-Ortín et al., 2015; Jiménez-Martínez, Gómez-Plaza, Molero, & Bautista-Ortín, 2017). Using cell walls, isolated from the pomace obtained after fermentation and devatting, may be an interesting way of adding value to this natural non-allergenic grape product. Cell walls are composed of about 10% structural proteins and 90% polysaccharides, grouped into three categories: cellulose, pectins and hemicelluloses (McNeil, Darvill, Fry, & Albersheim, 1984) whose hydroxyl groups have the ability to form hydrogen bonds and hydrophobic interactions with certain molecules, tannins and anthocyanins among them (Bautista-Ortín, Martínez-Hernández, Ruiz-Garcia, Gil-Muñoz, & Gómez-Plaza, 2016).

Previous studies have reported that pomace cell walls present a

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quite similar composition to that of fresh grapes, the percentual carbohydrate composition not significatively changing between fresh skin cell walls and pomace cell walls (Bautista-Ortín et al., 2015). Guerrero, Smith, and Bindon (2013) also reported that pomace cell walls presented a chemical and polysaccharide composition quite similar to that of fresh grapes. However, the isolation of purified cell walls is a tedious and time-consuming process so as a model for a fiber preparation that might be commercially accessible from processing byproducts, and that would make its obtention faster and less expensive, purified grape pomace (PGP) was used in this study.

As stated before, previous investigations showed that the use of cell walls from grape pomace could largely reduce the amount of anthocyanins and tannins in wines (Bautista-Ortín et al., 2016; Bindon & Smith, 2013). However, such a reduction may also significantly affect wine color (Guerrero et al., 2013), leading to less colored and structured wines, making necessary an optimization in its addition ratio and contact time in the wine. Boulton, Singleton, Bisson, and Kunkeem (1996) stated that the time required for tannin and fining agent to interact is very fast, taking anywhere from 15 min to an hour, whereas the time for the particles to settle can take some days, the duration of the settling period depending on the wine density, volume, temperature, and protein amount added.

The aim of this study, therefore, was to determine the best PGP addition ratio and contact time to reduce tannins in wine without producing great changes in its chromatic characteristics, considering three different pomace doses (6, 10 and 13 mg/mL) and three contact times (5, 10 and 20 days). Moreover, since the PGP composition may be important in the interaction processes with wine phenolics, the effect of PGP obtained from the pomace of four different varieties on the wine phenolic composition and color was also investigated.

2. Materials and methods

2.1. Purification of grape pomace to obtain the material for fining purposes

Marcs from Monastrell, Syrah, Cabernet Sauvignon (CS) and Macabeo grape varieties, grown in four plots located in Murcia (southeastern Spain) and harvested at commercial ripeness, were obtained after the devatting and pressing of a small-scale winemaking process where a maceration time of 7 days was applied. PGP from Macabeo variety were obtained after a direct press white wine vinification. The skins were separated from the seeds and pulp residues with a scalpel, and then were placed in covered Erlenmeyer and treated with a solution of 70% ethanol (1:3, w/v). The samples were shaken at 150 rpm in an orbital shaker at room temperature for 24 h and in the dark in order to eliminate any residual phenolic compounds and residual polysaccharides. This purification process was carried out twice. The purified grape pomaces were then washed with HPLC grade water, lyophilized and grind until a powder. Before of their use, a composition analysis was carried out in triplicate.

2.2. Compositional analysis of PGP from different varieties

2.2.1. Protein quantification

The protein content of the PGP was determined colorimetrically by the Bradford method (Bradford, 1976) after the extraction of 10 mg of PGP with 1 mL of 1 N NaOH (100 °C, 10 min). Then, 10 μL of the supernatant previously centrifuged (13,000 rpm) was combined with 790 μL of H_2O and 200 μL of the Bradford reactive, measuring the absorbancia at 595 nm after 15 min. The assay was calibrated with a standard curve of bovine serum albumin (250 μg/mL), giving a final concentration of between 0 and 46.88 μg/mL in 0.8 mL.

2.2.2. Phenolic quantification

The PGP total phenolic content was measured colorimetrically using the Singleton and Rossi method (1965) after the extraction of 10 mg of PGP with 1 mL of 1 M NaOH (100 °C, 10 min). Then, 10 μL of supernatant previously centrifuged (13,000 rpm) was combined with 40 μL of 1 M NaOH, 790 μL of H_2O and 40 μL of Folin-Ciocalteu reactive (1:2 v/v), measuring their absorbance at 700 nm after 1 h. The assay was calibrated with a standard curve of gallic acid (500 μg/mL) giving a final concentration in 920 μL between 0 and 16.3 μg/mL.

2.2.3. Lignin, uronic acids, cellulosic glucose and no-cellulosic glucose analysis

PGP samples (10 mg) were analysed in triplicated following the method described in Castro-López, Gómez-Plaza, Ortega-Regules, Lozada, and Bautista-Ortín (2016). Cellulosic glucose and no-cellulosic glucose and acid-insoluble residue (Klason lignin) were estimated after a pretreatment (30 °C, 1 h) with aqueous 72% sulfuric acid, followed by hydrolysis with 1 M sulfuric acid (100 °C, 3 h). Hydrolysis using only 1 M sulfuric acid (100 °C, 3 h) was used to determine noncellulosic glucose. Cellulosic glucose was obtained as the difference between the total glucose and non-cellulosic glucose content. In the same hydrosilate, uronic acids were determined colorimetrically with the 3,5-dimethylphenol assay. Pure galacturonic acid was used as a standard. The residual material was filtered, dried and weighed as Klason lignin.

2.3. Interaction of purified grape pomace with phenolic compounds

For the optimization of the doses and contact time, purified grape pomace from Monastrell grape variety was added to a Monastrell young wine with 12% v/v alcohol elaborated in our experimental winery with a small-scale winemaking technique. For that, 100 mL of wine were introduced in 125 mL glass bottles and combined with three different PGP doses (6, 10 and 13 mg/mL) reamining in contact for 5, 10 and 20 days.

To study the effect of the grape pomace varietal origin, pomaces obtained from Monastrell, Syrah, Cabernet Sauvignon and Macabeo were combined with the Monastrell young wine (100 mL) using a dose of 6 mg/mL and a contact time of 5 days.

All trials were performed in triplicate and a control wine without PGP was also prepared. To avoid possible oxidations, nitrogen was added in the headspace of the bottles, and kept in the dark. The assay was performed at room temperature (20 °C) and without agitation, simulating the fining process used in the winery. Then, the wines were centrifuged at 13,000 rpm and their phenolic concentration and composition and chromatic characteristics were analyzed.

2.4. Analysis of wine chromatic parameters

The color intensity (CI) was calculated as the sum of the absorbances at 620 nm, 520 nm and 420 nm (Glories, 1984), the total and polymeric anthocyanins were determined by the method described by Ho et al. (2001), the total polyphenol index (TPI) by the method proposed by Ribereau-Gayon and Pontallier (1983) and the total tannins by the method of methylcellulose (Smith, 2005).

2.5. Analysis of wine anthocyanins and tannins by HPLC

The determination of the concentration and composition of anthocyanins and tannins, by the reaction with the floroglucinol, by HPLC was performed using the methodology proposed by Bautista-Ortín et al. (2016).

2.6. Analysis of molecular distribution of tannins by SEC

The method described by Kennedy and Taylor (2003) has been used, which provides information on the mass distribution of tannins. The method for the analysis of proanthocyanidins consists of 2 gel columns, 300 × 7.5 mm, 5 μm, 500 Å (effective molecular mass range up to 4000 using polystyrene standards) and 100 Å (mass range molecular weight