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Phenolic compounds, organic acids and antioxidant activity of grape juices produced in industrial scale by different processes of maceration

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ABSTRACT

The effect of maceration process on the profile of phenolic compounds, organic acids composition and antioxidant activity of grape juices from new varieties of *Vitis labrusca* L. obtained in industrial scale was investigated. The extraction process presented a high yield without pressing the grapes. The use of a commercial pectinase resulted in an increase on extraction yield and procyanidins B1 and B2 concentrations and a decrease on turbidity and concentration of catechins. The combination of 60 °C and 3.0 mL 100 kg^{-1} of enzyme resulted in the highest extraction of phenolic compounds, reducing the content of acetic acid. The juices presented high antioxidant activity, related to the great concentration of malvidin, cyanidin, catechin and caffeic, cinnamic and gallic acids. Among the bioactive compounds, the juices presented high concentration of procyanidin B1, caffeic acid and *trans*-resveratrol, with higher levels compared to those reported in the literature.

1. Introduction

The world consumption of grape juice has grown each year, being the Brazil and United States the largest producers and consumers (OIV, 2011). In Brazil, the production of juices has been spread in the region of Vale do Submédio São Francisco (VSF), located at the Northeast region. The main grape varieties for the juice production are Isabel Precoce (*Vitis labrusca*) and the hybrids BRS Cora and BRS Violeta, new Brazilian varieties for the production of juices of high quality (Lima et al., 2014).

The grape juice is a representative source of phenolic compounds and studies have demonstrated that the consumption of this beverage is associated to several health benefits to consumers (Krikorian et al., 2012; Vauzour, Rodriguez-Mateos, Corona, Oruna-Concha, & Spencer, 2010). The phenolic compounds associated with these benefits are, mainly, flavonols, flavanols, anthocyanins, phenolic acids and stilbenes (Krikorian et al., 2012; Sautter et al., 2005; Xia, Deng, Guo, & Li, 2010). Among the biological activities related to the phenolic compounds, the antioxidant activity is one of the main characteristics of grape juices (Burin et al., 2010; Dani et al., 2007; Dávalos, Bartolome, & Gómez-Cordovés, 2005; Xia et al., 2010). Several factors exert influence on the juice composition, as the processing techniques (Morris & Striegler, 2005). Among these, variables as temperature, maceration time and use of enzymes have influence on the analytical characteristics, phenolic compounds, antioxidant activity and yield of juices (Fuleki & Ricardo-da-Silva, 2003; Leblanc, Johnson, & Wilson, 2008; Mojsov, Ziberoski, & Bozinovic, 2011; Talcott & Lee, 2002).

In an industrial scale process, the techniques of juices production are divided basically in "Hot press" (HP) and "Cold press" (CP), corresponding to the pressing the heated grape (HP) or pressing at ambient/chilling temperature (CP) (Morris, 1998; Morris & Striegler, 2005). In the HP process the crushed grape is heated at 60–62 °C, pectinase enzyme is added and samples are placed in stainless steel tanks with shaking to promote the extraction of compounds present in grape films, step known as maceration, for 30–60 min. Then, the juice is drained and the bagasse is pressed, obtaining a cloudy juice, which will be submitted to clarification

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treatments to remove suspended solids, using vacuum rotary filters or industrial centrifuges. After, the juice is stabilized, pasteurized and bottled hot (Iyer, Sacks, & Padilla-Zakour, 2010; Morris, 1998).

In the CP process, the maceration of crushed grape is carried out at ambient/chilled temperature, with addition of sulfur dioxide to inhibit the action of oxidative enzymes, and addition of pectinases to degrade the structures of grape films, facilitating the liberation of phenolic compounds to the juice (Leblanc et al., 2008; Morris & Striegler, 2005).

Another technique used is the "Hot Break" (HB), where the grapes are crushed and heated at temperatures higher than 75 °C, for a short time (5–10 min), to deactivate the polyphenoloxidases (PPO), and cooled at 60 °C to add pectinase, following the procedures used in HP process (Iyer et al., 2010; Morris & Striegler, 2005).

In the preparation of grape juices, the maceration process has relevant importance, since is in this phase where the incorporation of the compounds present on the grape film to the juice occurs, as phenolic compounds and aroma components (Fuleki & Ricardo-da-Silva, 2003; Iyer et al., 2010). The heating of crushed grape has as objective to promote the plasmolysis of the membrane and ruptures on the fruit cell wall, facilitating the liberation of liquid and anthocyanins responsible for the color. The enzymes (pectinases) on the grape juices are used to hydrolyze the pectin present on the film, facilitating the liberation of shell compound; to reduce the viscosity of the juice and to increase the juice yield and also to reduce the turbidity (Cascales, Garcia, Roca, & Plaza, 2012; Gomes, Guez, Martin, & Silva, 2007). The addition of pectinases during the grape maceration can be considered a complex process, resulting in important alterations on the chemical composition of grape juice, mainly related to phenolic compounds. There are several commercial enzyme formulations available for application in fruit maceration, consisting of pectinases, cellulases and galactosidades (Arnous & Meyer, 2010; Landbo & Meyer, 2004).

The maceration process comprehends a critical step on grape juice production, directly related to the phenolic quality and bioactive properties of the product. Chemical modification as monomeric anthocyanins degradation and increase of phenolic acids concentration has been reported in grape derivatives treated with enzymatic pool based on pectinases and cellulase (Arnous & Meyer, 2010; Toaldo et al., 2014). However, the chemical alterations from the grape maceration and their influence on grape juices processed in industrial scale are not yet known. In this context, the objective of this work was to evaluate the influence of maceration process at industrial conditions using a commercial enzymatic pool, based on pectinases, on the main group of phenolic compounds, organic acids and antioxidant activity of grape juices from new varieties of *V. labrusca* L.

2. Materials and methods

2.1. Chemicals

Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid), 2,2-azino-bis(3-ethylbenzthiazoline-6-sulfonic acid) (ABTS) and 2,2-diphenyl-1-picrylhydrazyl (DPPH) were purchased from Sigma–Aldrich (St. Louis, MO, USA). Ethanol, potassium persulfate and Folin–Ciocalteu reagent were obtained from Merck (Darmstadt, Germany). Methanol, acetonitrile and 85% phosphoric acid were obtained from Vetec Ltda (Rio de Janeiro, Brazil), J.T. Baker (Phillipsburg, NJ, USA) and Fluka (Switzerland), respectively. Analytical standards of tartaric, malic, citric, succinic, lactic, acetic and ascorbic acids were purchased from Vetec Ltda (Rio de Janeiro, Brazil). Malvidin 3,5-diglucoside, cyanidin 3,5-diglucoside, malvidin 3-glucoside, cyanidin 3-glucoside, peonidin 3-glucoside, delphinidin 3-glucoside and pelargonidin 3-glucoside, kaempferol 3-glucoside, myricetin, quercetin, rutin (quercetin 3-rutinoside), isorhamnetin 3-glucoside, (+)-catechin, (−)-epicatechin, (−)-epicatechin gallate, (−)-epigallocatechin, procyanidins A2, B1 and B2, and *trans*-resveratrol were purchased from Extrasynthese (Genay, France). Gallic, cinnamic and caffeic acids were purchased from Chem Service (West Chester, USA). *p*-Coumaric and chlorogenic acids were obtained from Sigma–Aldrich (St. Louis, MO, USA). Ultra-pure water was obtained by purification using a Purelab Option Q Elga System (USA).

2.2. Grape samples

The grapes of Isabel Precoce and BRS Cora varieties were collected from a specific area destined to the production of commercial juice at Empresa Brasileira de Frutas Tropicais (EBFT/ASA), located at Projeto de Irrigação Senador Nilo Coelho – lote 07, PA III, Petrolina, Pernambuco State, Brazil, situated at 09° 27′S latitude and 40° 38′W longitude, at an altitude of approximately 350 m. The grape juices were produced in an industrial facility belonging to EBFT/ASA.

The plants were grown in vineyards with an average age of tree years, grafted on *IAC 572* rootstock, planted in a field with 3.0 and 2.0 meters of spacing between lines and plants, respectively, and following a trellis system. The irrigation was by micro-aspersion and the vineyards were pruned (production pruning) on October and the grapes were harvested on February, when they reached the required standard maturation: soluble solids between 18 and 20 °Brix, titratable acidity (TA) from 0.7 to 0.9 g 100 mL^{-1} of must, expressed as tartaric acid, and °Brix/TA ratio between 20 and 25.

The climatic data (average/month) for the region in the months of October and February, that is, from pruning to harvest time, were: temperature 28.4 °C, rainfall 16.6 mm, relative humidity 51.4%, evaporation 9.1 mm, luminance 546.3 lx/day and insolation 7.6 h, measured at the weather station of Bebedouro (Petrolina, Pernambuco State, Brazil, 09°09′S 40°22′W).

2.3. Grape juice processing

The juice was elaborated following the formulation adopted by industries of Northeast of Brazil, a mixture of Isabel Precoce 80% and BRS Cora 20%, the cut (blend) was made by mixing the grapes at weighing. All juices were obtained by hot extraction without bagasse pressing, in an in-line system manufactured by JAPA® (Garibaldi, Rio Grande do Sul State, Brazil). The grapes were destemmed and crushed in an automatic equipment model DZ-35, with the addition of an enzymatic liquid mixture based on pectinase, called Endozym® Pectofruit PR, produced by Spindal – Pascal Biotech (Gretz-Armainvilliers, France) at doses of 0, 1.5 and 3.0 mL 100 kg^{-1} of fresh grapes. The grapes were then pumped into a maceration tank with controlled temperature and the mixture was heated at 50 and 60 °C and held at this temperature for 1 h with constant pumping of the liquid. After maceration the juice was separated by draining, aided by a suction pump. This procedure did not require pressing of the bagasse. The juice was homogenized and then submitted to pasteurization at 85 °C for 60 s in a tubular pasteurizer. The samples were then packaged, through hot filling of non-colored glass bottles of 300 mL capacity manufactured by Saint-Gobain® (São Paulo-SP, Brazil), using a gravimetric automatic filling machine (EVR12 model). The filled bottles were capped, closed and tumbled. The closed bottles were cooled in a cooling tunnel by water spraying until reaching an average temperature of 45 °C.

The juices were obtained by the following different maceration conditions: 50 °C without the addition of pectinase (T50E0), 60 °C