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Whey-grape juice drink processed by supercritical carbon dioxide technology: Physicochemical characteristics, bioactive compounds and volatile profile



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ABSTRACT

The effect of supercritical carbon dioxide technology (SCCD, 14, 16, and 18 MPa at 35 ± 2 °C for 10 min) on whey-grape juice drink characteristics was investigated. Physicochemical characterization (pH, titratable acidity, total soluble solids), bioactive compounds (phenolic compounds, anthocyanin, DPPH and ACE activity) and the volatile compounds were performed. Absence of differences were found among treatments for pH, titratable acidity, soluble solids, total anthocyanin and DPPH activity (p-value > 0.05). A direct relationship between SCCD pressure and ACE inhibitory activity was observed, with 34.63, 38.75, and 44.31% (14, 16, and 18 MPa, respectively). Regards the volatile compounds, it was noted few differences except by the presence of ketones. The findings confirm the SCCD processing as a potential promising technology to the conventional thermal treatment.

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1. Introduction

The lifestyle of consumers around the world has changed. Large and complex economic, social, cultural and political movements have led to a strong tendency to change the consolidated consumption habits (Brazil Food Trends 2020, 2010). The search for healthier life practices has led the industrial sector to develop healthpromoting products with convenience and high quality, besides adopting sustainable and clean processes.

In this sense, the consumption of whey-grape juice drink is an alternative for obtaining a range of nutrients that promote health, along with the new tendencies of the global market. The nutritional value of whey and its high bioavailability have motivated its incorporation as an ingredient in food products due to the nutritional appeal and functional properties. In addition, grape juice is appreciated all over the world due to its unique taste, besides being a source of phenolic compounds that exert health benefits

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when consumed regularly (Granato, Margraf, Brotzakis, Capuano, & Van Ruth, 2015).

Conventional heat treatments promote the degradation of nutrients and bioactive compounds, as well as affect the taste of food due to the cooking process (Marszałek et al., 2015). Therefore, non-thermal treatments have been studied aiming at the production of foods with sensory characteristics closer to those of the fresh product.

Supercritical carbon dioxide (SCCD) is a clean technology, recognized as environmentally friendly, which has been gaining prominence in the non-thermal treatment of foods, due to the absence of the harmful effects of heat, thus keeping the physicochemical, nutritional, and sensory characteristics of the fresh product (Ceni et al., 2016; Ramírez-Rodrigues, Plaza, Azeredo, Balaban, & Marshall, 2012, Marszałek et al., 2015). Carbon dioxide (CO₂) has critical properties at low temperatures (31.1 °C) and moderate pressure (7.38 MPa) (Chen et al., 2010). Therefore, SCCD processing for pasteurization can occur under temperature conditions lower than those in the conventional heat treatments, characterizing this technology as a non-thermal pasteurizing process of food products. Supercritical technology has also emerged as an alternative to hydrostatic high-pressure processing, which can reach up to 1000 MPa, while SCCD treatments are close to 10 MPa (Ceni et al., 2016). Other terms adopted for the technology are dense phase carbon dioxide (DPCD), and liquid carbon dioxide (LCD) and/or high-pressure carbon dioxide (HPCD) (Garcia-Gonzalez et al., 2007).

Several studies have demonstrated the efficiency of SCCD processing in the preservation of juices such as orange, melon, hibiscus, kiwi, pear, and strawberry (Ramírez-Rodrigues et al. 2012; Yuk, Sampedro, Fan, & Geveke, 2014). However, there is no report about the effect of the SCCD on the physicochemical characteristics of whey-juice drinks. Therefore, the purpose of this study was to investigate the effects of SCCD treatment (14, 16, and 18 MPa at $35 \pm 2 \,^{\circ}$ C, for 10 min) compared to the conventional heat treatment by HTST (72 °C for 15 s) on the physicochemical characteristics of whey-grape juice drink in relation to pH, titratable acidity, total soluble solids, bioactive compounds, angiotensin converting enzyme (ACE), and volatile compounds.

2. Material and methods

2.1. Whey-grape juice drink processing

The following ingredients were used for the preparation of whey-grape juice drink: 50% (v/v) of whole red grape juice (Mitto[®], Brazil, natural juice without preservatives, sugar or sodium) and 50% (v/v) whey powder (Alibra[®], Brazil) reconstituted in distilled water, according to the manufacturer's instructions. About 0.108 g xanthan gum (SatiaxaneTM, Cargill, Spain) and 7.53 g sucrose (Union[®], Brazil) were added per 100 g of sample. After mixing the ingredients, the beverage was homogenized in a rotor-stator type homogenizer (Walita[®], Brazil) for 5 min. Then, the beverage was subjected to conventional thermal processing (HTST pasteurization) and non-thermal treatment using SCCD technology. The HTST was applied at 72 °C for 15 s in a temperature-controlled water bath (Marconi[®], MA126 / BO, Piracicaba, Brazil). The temperature of the sample was monitored using a digital thermometer (Digital Thermometer Hanna[®], Check temp,

HI98501, Nusfalau, Romania), and the samples were cooled to 10 $^{\circ}\mathrm{C}$ immediately after treatment.

For supercritical dioxide carbon technology, a homemade equipment was used assembled by the research group in LASEFI (Laboratory of Supercritical Technology: Extraction, Fractionation, and Identification of Vegetal Extracts) - Department of Food Engineering (DEA)/School of Food Engineering (FEA), University of Campinas (UNICAMP)/Brazil. Fig. 1 shows the flow diagram of the experimental apparatus. A volume of 400 mL of whey-grape juice drink was manually introduced into the 316 stainless steel reactor, fitted with a flange closure system, with a maximum capacity of 630 mL. Carbon dioxide (99.9% CO₂, Gama Gases Especiais Ltda., São Bernardo do Campo, Brazil) cooled at -6 °C in a thermostatic bath was pumped by a pneumatic pump to the high-pressure reactor, promoting the homogenization of the sample until reaching the operating pressure. The 10-min processing time was counted from the temperature stabilization at 35 ± 2 °C. monitored by means of a digital thermocouple. At the end of each treatment, the equipment was depressurized and the sample collected and immediately cooled. All procedures of hygiene and asepsis were carefully carried out during the experiments, considering the microbiological safety and quality aspects.

The samples were subjected to SCCD at 14, 16, and 18 MPa and $35 \pm 2 \,^{\circ}$ C for 10 min, as described in the next section. All experiments were performed in duplicate. Samples were collected immediately after the process and frozen ($-18 \,^{\circ}$ C) for further analysis, except the pH and soluble solids determinations, which were performed immediately after the processing.

2.2. Physicochemical characterization

The pH was measured using a pH meter (AKSO, AK103, São Leopoldo/RS, BR) calibrated with pH 7 and pH 4 buffer solutions, according to A.O.A.C. Association of Official Analytical Chemists (2000). The total soluble solids were measured with a digital refractometer (Atago[®], PAL-1, Tokyo, Japan). The titratable acidity was determined using a bench titrator (PHS-3B, Phtek, China) by titration with 0.01 M NaOH, according to A.O.A.C. Association of Official Analytical Chemists (2000) and the results expressed as g

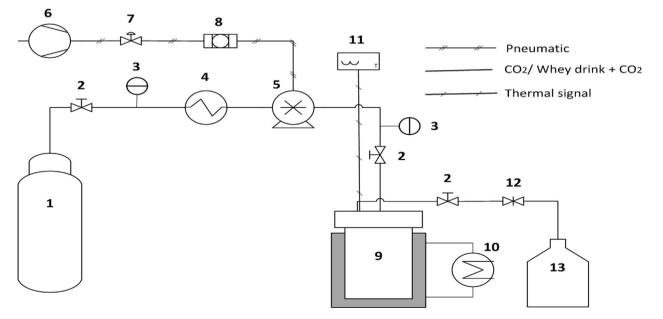


Fig. 1. Diagram of the homemade equipment for the supercritical dioxide carbon technology. 1 – CO₂ Cylinder; 2 – Control valve; 3 – Manometer; 4 – Cooling bath; 5 – Pump (Booster); 6 – Air compressor; 7 – Control valve (air flow); 8 – Air filter; 9 – Batch reactor; 10 – Heating bath; 11 – Temperature indicator; 12– μm valve; 13 –Whey-grape juice drink + CO₂.

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