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### **ABSTRACT**

This brief literature review aims to analyze relevant studies on the application of the cryoconcentration process in wine and grape juice. The cryoconcentration consists of a non-thermal process of concentration of juices and wines capable of preserving most of the chemical, nutritional, functional, and sensory characteristics of the products, resulting in concentrated extracts of high quality and water with great purity.

**Keywords:** Concentration by freezing, Cryoconcentrate, Grape juice, Must, Wine.

## **1 INTRODUCTION**

Water is the main component of liquid foods. The study of its behavior is essential to promote adequate preservation of the constituents of the raw material, as well as proper processing (MIAYWAKI, 2018). The concentration of liquid food is a very important unit operation since concentrated foods take up less space and are lighter. In the food industry, concentration can contribute to the reduction of costs with processing, storage, and transportation. The main methodologies described in the literature for liquid food concentration are evaporation, membrane concentration, and freezing concentration (PETZOLD et al., 2016).

Evaporation or concentration by boiling, consists of the partial removal of water present in liquid foods using boiling and the release of water vapor. Despite being a low-cost method, capable of originating highly concentrated products, when compared to membrane and freezing concentration, it

has higher energy consumption. In addition, the high temperatures used in the process can compromise the sensory and nutritional characteristics of the food, thus affecting its quality (SÁNCHEZ et al., 2011).

The membrane concentration method, known as reverse osmosis, consists of unit operations in which water and some solutes in a given solution are selectively removed from a semipermeable membrane. Because it is a process that does not require phase change, it is the method that requires the lowest energy consumption among the others. However, the membranes used generally have high cost and the maximum concentration of total solids obtained with the process is up to 30% (SÁNCHEZ et al., 2011). Product quality and processing costs are average between evaporation and freezing concentration (MIYAWAKI; INAKUMA, 2021).

The concentration by freezing or cryoconcentration promotes the concentration of liquid food from the freezing and subsequent separation of a part of the frozen water (AIDER; HALLEUX, 2009). The degree of concentration of the products obtained is higher than in reverse osmosis, however, lower than in the concentration by evaporation (SÁNCHEZ et al., 2011). The cost and yield of freezing concentration vary according to the method chosen (AIDER; HALLEUX, 2009).

Cryoconcentration is characterized as a non-thermal technology with a low environmental impact that, by operating at low temperatures, causes minimal sensory changes in the final product, resulting in a high-quality concentrate. Because of this particularity, this is an emerging technology in the food and nutraceutical industry (PETZOLD et al., 2016).

Among the fruits, the grape stands out as one of the largest sources of compounds with antioxidant activity in the diet, due to the presence of flavonoids, anthocyanins, resveratrol, and tannins in its composition (DA SILVA et al., 2019; TAVARES et al., 2019). In addition to the antioxidant potential, the phenolic compounds present in grapes also have neuroprotective, antimicrobial, anticancer, anti-inflammatory, antifungal, antithrombotic, antidiabetic, and anti-obesity action (SMERIGLIO et al., 2016; KOOH et al., 2017). For this reason, grapes and their derivatives, such as juices and wines, are increasingly being studied and consumed, due to their relevance to health. In this sense, the present study aimed to gather the main works published so far on the use of cryoconcentration technology in grape-based, alcoholic, and non-alcoholic beverages, presenting the main scientific findings and future perspectives.

## **2 METHODOLOGY**

Exploratory research was carried out in digital repositories of the area of Food Science and Technology, in the following databases: Science Direct, Web of Science, Scielo, Google Scholar, and Portal de Periódicos da Capes. The keywords used in the search were: cryoconcentration,

cryoconcentration technology, freeze concentration, freeze-concentrated, grape, grape juice, grape wine, grape pomace, and grape most, being used alone and in combination. Articles published in English from the last 20 years were selected.

### **3 RESULTS AND DISCUSSION**

#### **3.1 CRYOCONCENTRATION TECHNIQUE**

Cryoconcentration methods can be classified into two categories: methods with the formation of small ice crystals and methods with the formation of large ice crystals (MIYAWAKI; INAKUMA, 2021). Suspension cryoconcentration is a complex method that involves scraped-walled heat exchangers for the generation of small ice crystals, a recrystallization tube for crystal growth, and a wash tower for the separation of ice crystals. Due to its high complexity and high cost, it has limited application in the food industry (AIDER; HALLEUX, 2009).

The method of progressive cryoconcentration is characterized as a process in which the formation of a single large ice crystal occurs, thus facilitating its separation from the mother solution. This system is composed of a cylindrical compartment, a cooling bath, and a sample conduction system, and the crystal is formed on the cooling surface of the crystallization reservoir (MIYAWAKI; INAKUMA, 2021).

Cryoconcentration in blocks is a method that consists of the partial or total freezing of liquid food, forming a single large crystal. The process takes place in three stages: freezing, thawing, and the separation of the concentrated liquid from the ice fraction. In partial freezing, a refrigerant tube is introduced into the center of the feeding solution, while in total freezing the solution is frozen integrally, both processes being followed by simple gravitational thawing (AIDER; HALLEUX, 2009) or by assisted processes, such as centrifugation (PETZOLD; AGUILERA, 2013) and vacuum pump (PETZOLD et al., 2013). However, the efficiency of cryoconcentration in blocks is limited, when compared to other methods, requiring the performance of several operations to obtain an extract with a high level of concentration (MIYAWAKI; INAKUMA, 2021).

In recent years, cryoconcentration in blocks has been reported as promising and effective for obtaining food products with greater nutritional value and greater preservation of sensory and functional properties. With this, cryoconcentration comes from the concentration of yerba mate (BOAVENTURA et al., 2013), green tea (*Camellia sinensis*) (MENESES et al., 2021), coffee (Moreno et al., 2014) and some types of fruit juice, including apple (ORELLANA-PALMA et al., 2020), blueberry ( ORELLANA-PALMA et al., 2021), and orange (PETZOLD et al., 2019a; PETZOLD et al., 2019b).

### 3.2 CRYOCONCENTRATION OF GRAPE JUICE

The cryoconcentration of liquid foods, such as grape juice and must, consists of the fractional crystallization of water (liquid) to ice (solid) through freezing and the subsequent removal of ice, using washing column techniques or mechanical separation. This process can retain high concentrations of volatile aromatic compounds and preserve the nutrients of the food, generating a concentrated ingredient of high nutritional and functional quality, which can be used as a natural basis for the development of new products, and also contribute to the reduction of transport and storage costs in the industry (MIYAWAKI; INAKUMA, 2021). However, when compared to thermal concentration processes, cryoconcentration has disadvantages such as low production yield, reduced effect on microorganisms and enzymes, and high costs of equipment acquisition, refrigeration, and operation (AMRAN et al., 2016; SÁNCHEZ-MACHADO et al., 2009).

In recent years, the cryo-conception of grape juices has been the object of study in some studies conducted in European countries and Malaysia. In Italy, Albergamo et al. (2020) studied a by-product ("grape water") of the cryoconcentration of grape must of the varieties 'Grillo' and 'Moscato D'Alessandria'. The authors stated that there is a great potential for its use since it has physicochemical parameters similar to mineral water and still presents nutritional, flavoring, flavoring and functional properties characteristic of the grape, in addition to representing an innovative and profitable solution for the wine agroindustry. Another important point highlighted by the authors is the reuse and valorization of a by-product that represents about 65% (v/v) of the initial grape juice, which ceases to be waste to be consumed as a pasteurized beverage or generate a possible ingredient for the food industry.

In Spain, Hernández et al. (2010) examined the cryoconcentration process in grape must of the Macabeo variety using descending film cryo concentrator. As a result, they obtained an average concentration rate of 1.38 °Brix/h, and the content of soluble solids reached was 29.5 °Brix, equivalent to an increase of 89.94% of the °Brix of the initial grape juice (16.4 °Brix). The operating flow of the equipment was 0.8 L/s and consumed between 0.58 - 1.00 kWh/kg of ice. Ice production varied between 1.32 and 1.05 gm<sup>-2</sup> s<sup>-1</sup> for the initial juice and the most concentrated juice, respectively. The association of parameters such as the relative impurity of the ice, calculation of the concentration efficiency, estimation of the energy consumed, and the monitoring of the soluble solids content (°Brix) of the juice and ice proves the viability of the cryoconcentration process of descending film.

In Malaysia, the cryoconcentration of grape juice using a system of cryo concentrators in sequence coupled to a coil crystallizer was evaluated by Safiei et al. (2017), who observed a high retention of polyphenols and an efficient concentration of grape juice. Operating parameters such as refrigerant temperature (-8.0 °C), circulation flow (3.0 L/min.) and operating time (20 min.) were



determinants to confirm the viability and operational and productive efficiency of the evaluated system.

Despite the growing interest in the study of this method of concentration of liquid foods, to date, there is no specific legislation in force in Brazil for cryoconcentrated products based on grapes or any other food.

### 3.3 CRYOCONCENTRATION OF WINE

The concentration of liquid food is one of the most important unit operations in the food industry. Among the technically feasible processes for obtaining liquid concentrates, cryoconcentration stands out for being an efficient method in the preservation of alcohol and aromatic compounds, which are characteristics present in wine.

Research conducted in the last decade has demonstrated the potential of cryoconcentration applied to this raw material in Chile and China. Wu et al. (2017) evaluated the influence of block cryoconcentration on the quality of red wine produced from Cabernet Sauvignon grapes from the Xinjiang region, of China. The results showed that cryoconcentration improved the attributes of color, appearance, aroma, and flavor of the sample, significantly increased the levels of alcohol and glycerol, increased the concentration of esters and anthocyanins, reduced the levels of volatile acids and maintained the other physicochemical properties when compared to the initial sample. In addition, in the sensory evaluation, the cryoconcentrated wine presented better fragrance characters and better sensation in the mouth. Given the findings, the authors suggested that the treatment of wine concentration by freezing may be a good choice to improve the quality of the wine.

Petzold et al. (2016) studied the efficiency of cryoconcentration in vacuum pump-assisted blocks in red wine, produced from grapes of the Cabernet Sauvignon variety from the Valle Central region of Chile. The authors observed that after applying five minutes of suction with a vacuum (40 kPa), the solids content of the concentrated fraction increased significantly (8 °Brix to 25 °Brix), about the solids content present in the initial sample, also noting that the highest concentration occurred mainly in the first cryoconcentrated fractions. The methodology adopted obtained an efficiency of 90% in a short time interval (15 minutes), also observing high purity values in the ice fraction obtained. Regarding the levels of alcohol, acidity, total polyphenols, and pH, the authors observed a significant increase in the concentrated fraction, with color intensification. Thus, they suggest that the technique used in the study is efficient in obtaining samples with possible gastronomic and industrial application, due to its highly attractive properties.

Zhang et al. (2016) evaluated the viability of cryoconcentration of white grape juice and ink with low sugar contents for use in wine production. The juices used in the research were obtained from

grapes from the Xinjiang region, China, and were cryoconcentrated in cryoconcentration equipment by suspension on a pilot scale. After obtaining the cryoconcentrated juices, they were applied in the production of white and red wine, which were submitted to the evaluation of physical and chemical parameters, polyphenol content, and sensory attributes. The results showed that the methodology used was able to raise the content of soluble solids, from 14 °Brix to 23 °Brix, as well as the phenolic content. In addition, the white and red wines obtained from the cryo-concentrated must presented superior sensory quality to the control wines (made from chaptalized grape must), according to the results of the sensory analysis. Thus, the authors suggested cryoconcentration by suspension as a promising alternative to traditional chaptalization technology, highlighting the practicality of applying freezing concentration technology in the wine industry.

Because it is a contestable process, chaptalization is not allowed in some countries, such as Italy, Argentina, Spain, Portugal, and South Africa. But it is adopted and regulated in other countries, such as Brazil, Canada, New Zealand and the United Kingdom (ENO CULTURA, 2020).

### 3.4 CRYOCONCENTRATION OF GRAPE-BASED PRODUCTS: CURRENT AND FUTURE PERSPECTIVES

Given the high nutritional, sensory, and functional content of the products obtained from the grape, it is notorious the growing interest in the development of research that enables the implementation of more sustainable processing technologies, from the economic, environmental, and efficient in terms of yield and quality of the final product, and safe. Thus, it is observed that in the last decade, the concentration by freezing of grape-based products has been gaining more and more space in the scientific environment.

Analyzing the works already published within this theme, it is observed that they have been conducted mainly in some countries of the European continent, the main wine-growing region of the world, especially Spain and Italy. In addition, several studies have been carried out in countries of the Asian continent, especially in China, which includes the second-largest wine-growing area in the world (OIV, 2019). However, a limiting factor for better understanding and wide dissemination of the data is that many of the works produced by the Chinese are published in the official language of the country, Mandarin.

To date, the cultivars most studied in the research of cryoconcentration by freezing are those of the grapes Macabeo, Red Globe, Thompson, Cabernet Sauvignon, Grillo and Moscato. Regarding the most used freezing concentration method, it is observed that cryoconcentration in blocks stands out, possibly due to the lower investment required and also the ease of execution (MIYAWAKI; INAKUMA, 2021). Given the excellent results verified so far, about qualitative and quantitative

aspects of cryoconcentrated extracts, it is believed that these are gaining increasing popularity in the scientific and industrial environment.

#### **4 CONCLUSION**

The findings of this review show that, to date, there are a limited number of studies that have evaluated the application of freezing concentration in grape-based products, which have been carried out mainly in Chile, China, Spain, Italy, and Malaysia. However, the results already observed reinforce the viability and efficiency of the method in improving the nutritional, sensory and functional characteristics of grape juice, must and wine. It is known that concentrated liquids are advantageous options for the food industry since they can cause a decrease in logistics and processing costs, increased shelf life, and reduced waste production.

Because it is a non-thermal technology with low environmental impact, which presents promising results not only in grape-based beverages but also in several other liquid foods already studied, it is understood that the research and improvement of the method can bring numerous benefits to the food and pharmaceutical industries, concerning the quality of the products, processing and cost reduction, and also for the final consumer.

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