

CRYOCONCENTRATION PROCESS IN THE FOOD INDUSTRY: A REVIEW OF THE TECHNIQUE AND ITS APPLICATIONS

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Abstract: Cryoconcentration, known for its effectiveness, simplicity, and cost-effectiveness, has been gaining increasing attention in the food industry. The aim of this work was to provide a review of the cryoconcentration technique and its applications. The study was conducted through a systematic search, from 2013 to 2023, using the descriptor "cryoconcentration" in databases. Inclusion criteria required that the term "cryoconcentration" appear at least once in the title and/or body of the text, with a focus on "block freezing", "food cryoconcentration" and "applications of cryoconcentration in beverages". Thus, it was possible to verify that the cryoconcentration technique has already demonstrated its effectiveness in the preservation of bioactive compounds in fruit juices and wines, in the production of apple, orange, grape and tea juice concentrates. As cryoconcentration continues to evolve, its potential as a promising approach for the development of food products becomes increasingly evident. With the ability to preserve bioactive compounds, improve nutritional quality and intensify flavor concentration, cryoconcentration plays a significant role in the food industry and aligns with the principles of sustainability and circular economy by maximizing the use of regional raw materials and industrial by-products.

Keywords: block concentration; clean technology; emerging technology; freeze concentration.

Processo de crioconcentração na indústria de alimentos: uma revisão da técnica e suas aplicações

Resumo: A crioconcentração, conhecida por sua eficácia, simplicidade e custo-benefício, vem ganhando cada vez mais atenção na indústria alimentícia. O objetivo deste trabalho foi fornecer uma revisão da técnica de crioconcentração e suas aplicações. O estudo foi conduzido por meio de uma busca sistemática, de 2013 a 2023, usando o descritor "crioconcentração" em bancos de dados. Os critérios de inclusão exigiam que o termo "crioconcentração" aparecesse pelo menos uma vez no título e/ou corpo do texto, com foco no "congelamento em bloco", "crioconcentração de alimentos" e "aplicações da crioconcentração em bebidas". Desta forma, foi possível verificar que a técnica de crioconcentração já demonstrou sua eficácia na preservação de compostos bioativos em sucos de frutas e vinhos, na produção de concentrados de sucos de maçã, laranja, uva e chá.

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À medida que a crioconcentração continua a evoluir, seu potencial como uma abordagem promissora para o desenvolvimento de produtos alimentícios torna-se cada vez mais evidente. Com a capacidade de preservar compostos bioativos, melhorar a qualidade nutricional e intensificar a concentração de sabor, a crioconcentração desempenha um papel significativo na indústria alimentícia e se alinha com os princípios de sustentabilidade e economia circular ao maximizar o uso de matérias-primas regionais e subprodutos industriais.

Palavras-chave: concentração de blocos; tecnologia limpa; tecnologia emergente; concentração de congelamento

1 INTRODUCTION

The concentration of liquid foods is a crucial unit operation in the food industry. This technique aims to remove water from food products, resulting in an increase in total solids content, a reduction in product volume, and subsequently, cost savings in terms of packaging and transportation. Moreover, it offers greater biochemical and microbiological stability, extending the shelf life of the product (Zielinski *et al.*, 2018; Orellana-Palma *et al.*, 2019).

Liquid food concentration can be achieved through various mechanisms, including evaporation, reverse osmosis, membrane technology, and cryoconcentration processes. According to Handojo *et al.* (2019), the evaporation technique, involving the use of heat, is widely employed. However, heat-based methods can lead to the degradation of bioactive compounds due to their high sensitivity to thermal treatment (Castro-Puyana *et al.*, 2013). Membrane concentration utilizes low temperatures and consumes less energy, preserving the quality of heat-sensitive compounds. Nonetheless, it has limitations due to issues like membrane fouling, necessitating frequent replacements and increasing process costs (Aider; De Halleux; Akbache, 2007). On the other hand, the cryoconcentration process offers advantages, such as the production of high-quality concentrates, preservation of heat-sensitive compounds, sensory properties, and nutritional value of products (Orellana-Palma *et al.*, 2019; Miyawaki; Inakuma, 2021).

Cryoconcentration, also known as freeze concentration, is an emerging technology that concentrates aqueous food solutions through partial or complete freezing of water (Petzold; Niranjana; Aguilera, 2013). This process enables the production of high-nutrient, biologically rich, and sensory-rich products with substantial potential for application in the food industry (Orellana-Palma *et al.*, 2017; Zielinski *et al.*, 2018).

The cryoconcentration process comprises three main stages: freezing, thawing, and separation. These stages must be carefully controlled and synchronized to ensure process efficiency (Iritani *et al.*, 2013; Moreno *et al.*, 2014; Adorno *et al.*, 2017; Orellana-Palma *et al.*, 2020; Orellana-Palma *et al.*, 2021). However, when compared to thermal concentration processes, cryoconcentration has some disadvantages, including lower production yields, reduced impact on microorganisms and enzymes, and higher costs associated with refrigeration equipment and operation (Amran *et al.*, 2016; Miyawaki; Inakuma, 2021). In this context, this literature review aims to compile data and key findings on the use of cryoconcentration techniques in food products, with a particular focus on their applications in the food industry.

2 METHODOLOGY

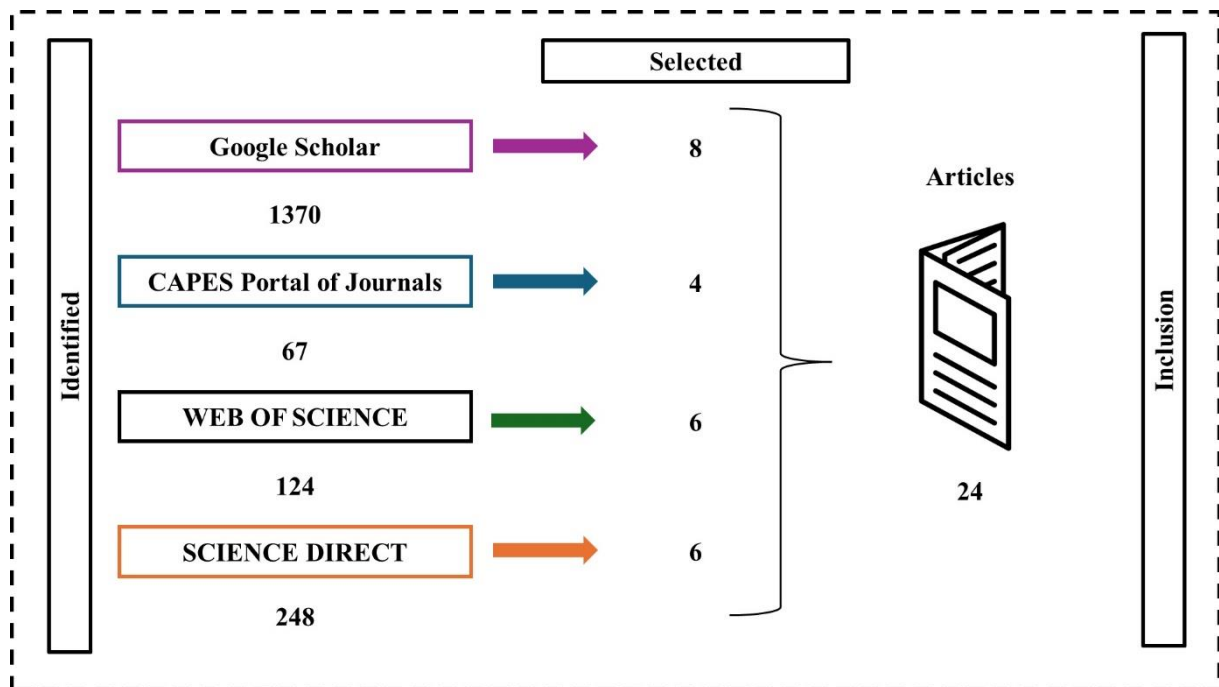
The research was conducted through a quantitative and qualitative literature review covering an 11-year period from 2013 to 2023. To gather relevant materials, the descriptor "cryoconcentration" was used as a search term. Inclusion criteria required that the term "cryoconcentration" appear at least once in the title and/or body of the text. Additionally, to be considered in the research, articles had to specifically address topics related to "block freezing," "food cryoconcentration," and "applications of cryoconcentration in beverages."

To retrieve relevant scientific production, various databases were consulted, including Google Scholar, CAPES Portal of Journals (*Coordenação de Aperfeiçoamento de Pessoal de Nível Superior*), WEB OF SCIENCE and SCIENCE DIRECT.

3 RESULTS AND DISCUSSION

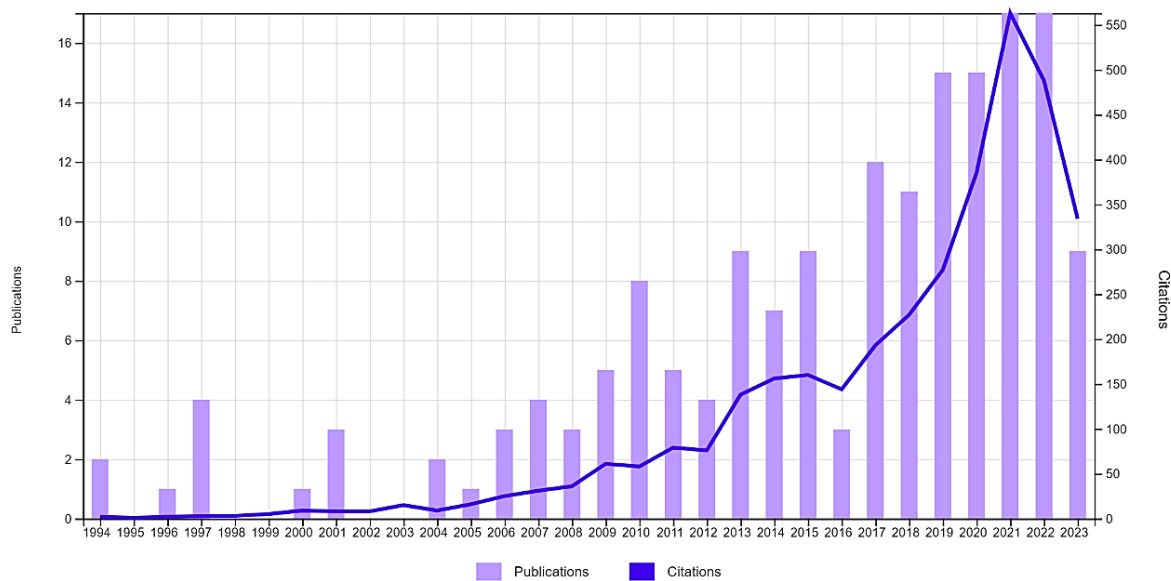
In the databases consulted, a total of 1809 articles were found using the descriptor "cryoconcentration" (Figure 1). It was found that the platform with the highest number of articles related to the topic was Google Scholar, with 1370 articles, considering the research period from 2013 to 2023; and the one with the lowest return was the CAPES Journal Portal, with 67 articles. The application of the exclusion filters established in the methodology resulted in the selection of 8 articles on the Google Scholar platform, 4 articles on the CAPES Journal Portal, 6 on Web of Science, and 6 on ScienceDirect, totaling 24 articles for data compilation and main findings on the use of the cryoconcentration technique applied to food products.

Figure 1. Selection of scientific papers for the composition of the database 2013 – 2023



Source: Authors

It is evident that interest in studying the cryoconcentration technique has increased significantly since 2005. The first works related to this technique were published in 1994, and to date, there are 170 publications focused on cryoconcentration and its applications (Figure 2). In the past five years alone, 73 articles on cryoconcentration have been produced, with 563 citations recorded in 2021 (Web of Science, 2023).

Figure 2. Number of citations and publications related to cryoconcentration over time

Source: Web of Science (2023)

Regarding commonly employed concentration techniques, block cryoconcentration stands out as the most common, simple, and cost-effective method. In this process, the completely frozen liquid is separated from the ice and the concentrate with the assistance of gravitational forces, such as centrifugation (Zielinski *et al.*, 2018). The use of centrifugation in cryoconcentration accelerates the recovery of solutes by applying centrifugal force, enhancing the efficiency of block cryoconcentration. During centrifugation, the ice block acts as a porous solid, allowing the concentrated juice to flow through drainage channels due to a greater driving pressure difference than gravitational force (Zielinski *et al.*, 2018).

Cryoconcentration is highly effective in preserving bioactive compounds found in foods, such as polyphenols, and has been employed to enhance the nutritional quality of liquid foods. This technique is considered a promising tool for concentrating bioingredients in natural products. Recent innovations in cryoconcentration have focused on single-stage systems, using block cryoconcentration or progressive cryoconcentration processes, which involve only one separation step (Boaventura *et al.*, 2015; Orellana-Palma *et al.*, 2017).

In comparison to evaporation techniques, which involve high temperatures, and membrane technology, which operates at low concentrations, cryoconcentration offers significant advantages in the production of high-quality concentrates. This process takes place at low temperatures without contact with the vapor-liquid interface, resulting in minimal to no loss of volatile compounds. This helps maintain the exceptional aroma, taste, and quality of frozen concentrated products, making this technique particularly suitable for concentrating various products, such as fruit juices, tea, coffee extracts, and aromatic extracts (Petzold; Niranjana; Aguilera, 2013; Petzold; Aguilera, 2013).

Zielinski *et al.* (2018) explored block cryoconcentration assisted by centrifugation in apple juice, aiming to produce a high-quality concentrate with potential in the food industry. The authors observed significant increases in phenolic levels with each freezing concentration cycle. Similarly, Meneses *et al.* (2021) evaluated the effect of three stages of block cryoconcentration on the solids content, polyphenols, catechins, antioxidant activity, and sensory quality of aqueous green tea extract. The results indicated that the concentrated green tea extract exhibited increased bioactive compound content following the concentration technique while preserving its functional properties and sensory attributes.

Lazzarotto *et al.* (2021) investigated the effects of adding corn starch to cryoconcentrated yerba mate extract with the aim of developing a potential nutraceutical product. They observed that yerba mate extracts altered the thermal properties of corn starch and increased the amount of incorporated organic compounds, concluding that it is feasible to obtain nutraceutical products through the combination of

bioactives present in yerba mate extract with starches or flours.

Arend *et al.* (2022) applied a multiple-stage gravity-assisted cryoconcentration process with microwave assistance to obtain a concentrated extract enriched with beet byproducts. The results revealed that multiple stage cryoconcentration achieved efficiency exceeding 80%, maintaining high concentrations of phenolic compounds and betalains. Furthermore, the concentrated extract of beet byproducts exhibited remarkable antioxidant activity.

Orellana-Palma *et al.* (2019) assessed the improvement of centrifugal cryoconcentration using the recovery of the concentrated fraction from the ice in orange juice. They found that solutes in the concentrated samples increased sixfold after three cycles compared to the initial solid concentration. In a subsequent study, Orellana-Palma *et al.* (2021) investigated the impact of block cryoconcentration assisted by centrifugal filtering on physicochemical parameters, phenolic content, anthocyanins, and flavonoids in blueberry juice. The results demonstrated an efficiency increase ranging from 81% to 86%, along with higher concentrations of bioactive content.

Several authors have conducted studies related to cryoconcentration due to its wide industrial application and high potential. Petzold and Aguilera (2013) investigated the use of centrifugation to remove the concentrated solution from the ice matrix in a single step, noting a recovery of approximately 0.73 kg of sucrose per 1 kg of initial sucrose, regardless of the initial concentration and freezing procedure. Zhang *et al.* (2016) performed cryoconcentration in grape juice for wine production, finding that the process preserved phenolic compounds and increased sugar levels, eliminating the need for chaptalization. Wines produced from cryoconcentrate were rated more positively in terms of sensory attributes compared to chaptalized wines.

Orellana-Palma *et al.* (2017) explored vacuum-assisted block cryoconcentration under various freezing and processing times to preserve polyphenols in blueberry juice. The results showed high retention of polyphenols in the final concentrate. Safiei *et al.* (2017) assessed the cryoconcentration of grape juice using a sequence of cryoconcentrators coupled to a coil crystallizer, achieving high retention of polyphenols and efficient concentration of grape juice. Wu *et al.* (2017) investigated the effects of cryoconcentration on aromatic compounds, phenolics, color attributes, and sensory properties of Cabernet Sauvignon wine. The results revealed improvements in color, appearance, aroma, and taste attributes, along with reduced levels of volatile acids and increased alcohol, esters, anthocyanins, and glycerol, while maintaining other physicochemical properties of the wine.

Albergamo *et al.* (2020) studied a byproduct known as "grape water," generated through the cryoconcentration of must, highlighting its potential use. This byproduct exhibits physicochemical parameters similar to mineral water and possesses nutritional aspects, flavorings, and typical grape functional characteristics, representing an innovative and profitable solution for the wine industry.

In a similar study, Bredun *et al.* (2023) investigated the recovery of bioactive compounds through the cryoconcentration process of vinification byproducts. The extraction was optimized, leading to grape pomace extracts rich in anthocyanins. Cryoconcentration was applied to enrich the extract, resulting in high concentrations of anthocyanins and color intensity. These studies underscore the effectiveness of cryoconcentration in recovering bioactive compounds from byproducts, contributing to the development of high-value products from previously underutilized resources.

Moreover, the application of cryoconcentration to byproducts significantly contributes to sustainability and the circular economy. The circular economy is an economic system aimed at eliminating waste and continually using resources through principles of reuse, recycling, and sustainable production (Suárez-Eiroa *et al.*, 2019). By recovering valuable compounds from vinification byproducts, cryoconcentration not only minimizes waste but also transforms it into economically valuable products. This process reduces the environmental impact of food production by diverting waste from landfills and promoting the efficient use of natural resources. Consequently, cryoconcentration supports sustainability principles by enhancing resource efficiency and promoting the development of a more eco-friendly and resilient food industry.

Overall, cryoconcentration is a versatile and efficient technique with significant potential for enhancing the nutritional quality of food products and contributing to sustainable food production practices. However, it is essential to address challenges such as optimizing operational parameters and

scaling up the process for industrial applications to fully realize its benefits.

4 CONCLUSION

Cryoconcentration stands out as a highly promising emerging technology, providing the food industry with the ability to develop high-nutritional-value products. Moreover, the application of cryoconcentration principles can be adapted for low-cost commercial equipment, making it accessible to small and medium-sized producers. This accessibility can catalyze the creation and dissemination of high-value-added food products, generating economic benefits and strengthening the circular economy when applied to regional raw material transformation.

The growing research and innovation in cryoconcentration highlight its potential to improve the quality of concentrated foods, preserve bioactive compounds, and enhance process efficiency. The mentioned studies addressing various aspects of cryoconcentration reflect its vital role in producing high-quality concentrates in diverse areas, from fruit juices to wines. Therefore, cryoconcentration represents a promising approach that can benefit both the food industry and small producers, driving the creation of healthier, higher-value-added products, while contributing to sustainability and the circular economy. Continuous research and development in this field can further expand the opportunities for cryoconcentration application, benefiting the food industry and society.

REFERENCES

- ADORNO, W. T. *et al.* Enhancement of phenolic compounds content and antioxidant activity of strawberry (*Fragaria x ananassa*) juice by block freeze concentration technology. **International Journal of Food Science & Technology**, v. 52, p. 1-7, 2017. DOI: <https://doi.org/10.1111/ijfs.13335>
- ALBERGAMO, A. *et al.* Grape water: reclaim and valorization of a by-product from the industrial cryoconcentration of grape (*Vitis vinifera*) must. **Journal of the Science Food and Agriculture**, v. 100, n. 7, p. 2971-2981, 2020. DOI: <https://doi.org/10.1002/jsfa.10326>
- AIDER, M.; DE HALLEUX, D.; AKBACHE, A. Whey cryoconcentration and impact on its composition. **Journal of Food Engineering**, v. 82, n. 1, p. 92-102, 2007. DOI: <https://doi.org/10.1016/j.jfoodeng.2007.01.025>
- AMRAN, N. A. *et al.* Review: parametric study on the performance of progressive cryoconcentration system. **Chemical Engineering Communications**, v. 203, n. 7, p. 957-975, 2016. DOI: <https://doi.org/10.1080/00986445.2015.1075982>.
- AREND, G. D. *et al.* Gravitational and microwave-assisted multi-stages block freeze concentration process to obtain enriched concentrated beet (*Beta vulgaris* L.) by-products extract: Bioactive compounds and simulated gastrointestinal profile. **Food and Bioprocess Processing**, v. 133, p. 77-86, 2022. DOI: <https://doi.org/10.1016/j.fbp.2022.03.004>.
- BOAVENTURA, B. C. B. *et al.* Effect of yerba mate (*Ilex paraguariensis* A. St. Hil.) infusion obtained by freeze concentration technology on antioxidant status of healthy individuals. **Food Science and Technology**, v. 62, n. 2, p. 948-954, jul. 2015. DOI: <https://doi.org/10.1016/j.lwt.2015.02.028>.
- BREDUN, M. A. *et al.* Bioactive compounds recovery by freeze concentration process from winemaking by-product. **Food Research International**, v. 173, p. 113220, 2023. DOI: <https://doi.org/10.1016/j.foodres.2023.113220>.
- CASTRO-PUYANA, M. *et al.* Subcritical water extraction of bioactive components from algae. In: **Functional ingredients from algae for foods and nutraceuticals**. Woodhead Publishing, 2013. p. 534-560. DOI: <https://doi.org/10.1533/9780857098689.3.534>
- HANDOJO, L. A. *et al.* Advancement in Forward Osmosis (FO) Membrane for Concentration of Liquid Foods. IOP Conference Series: **Materials Science and Engineering**, 547, 2019. DOI: <https://doi.org/10.1088/1757-899X/547/1/012053>.

IRITANI, E. *et al.* Improvement of concentration performance in shaking type of freeze concentration. **Separation and Purification Technology**, v. 120, p. 445-451, 2013. DOI: <https://doi.org/10.1016/j.seppur.2013.10.015>.

LAZZAROTTO, S. *et al.* Corn starch incorporated with freeze-concentrated Ilex paraguariensis extracts: a potential nutraceutical product. **Journal of Thermal Analysis and Calorimetry**, v. 146, 171–176, 2021. DOI: <https://doi.org/10.1007/s10973-020-09971-7>

MENESES, D. L. *et al.* Multi-stage block freeze-concentration of green tea (*Camellia sinensis*) extract. **Journal of Food Engineering**, v. 293, p. 110381, mar. 2021. DOI: <https://doi.org/10.1016/j.jfoodeng.2020.110381>.

MIYAWAKI, O.; INAKUMA, T. Development of progressive freeze concentration and its application: a review. **Food and Bioprocess Technology**, v. 14, n. 1, p. 39-51, 2021. DOI: <https://doi.org/10.1007/s11947-020-02517-7>

MORENO, F. L. *et al.* A process to concentrate coffee extract by the integration of falling film and block freeze-concentration. **Journal of Food Engineering**, v. 128, p. 88-95, 2014. DOI: <https://doi.org/10.1016/j.jfoodeng.2013.12.022>.

ORELLANA-PALMA, P. *et al.* Improvement of Centrifugal Cryoconcentration by Ice Recovery Applied to Orange Juice. **Chemical Engineering & Technology**, v. 42, n. 4, p. 925-931, 28 fev. 2019. DOI: <https://doi.org/10.1002/ceat.201800639>.

ORELLANA-PALMA, P. *et al.* Centrifugal Filter-Assisted Block Freeze Crystallization Applied to Blueberry Juice. **Processes**, v. 9, n. 3, p. 421, 26 fev. 2021. DOI: <https://doi.org/10.3390/pr9030421>.

ORELLANA-PALMA, P. *et al.* Influence of cryoconcentration on quality attributes of apple juice (*Malus Domestica* cv. Red Fuji). **Applied Sciences**, v. 10, n. 3, p. 959-976, 2020. DOI: <https://doi.org/10.3390/app10030959>.

ORELLANA-PALMA, P. *et al.* Protection of polyphenols in blueberry juice by vacuum-assisted block freeze concentration. **Food and Chemical Toxicology**, v. 109, p. 1093-1102, nov. 2017. DOI: <https://doi.org/10.1016/j.jfoodeng.2013.12.022>.

PETZOLD, G.; AGUILERA, J. M. Centrifugal freeze concentration. **Innovative Food Science & Emerging Technologies**, v. 20, p. 253-258, out. 2013. DOI: <https://doi.org/10.1016/j.ifset.2013.05.010>.

PETZOLD, G.; NIRANJAN, K.; AGUILERA, J. M. Vacuum-assisted freeze concentration of sucrose solutions. **Journal of Food Engineering**, v. 115, n. 3, p. 357-361, abr. 2013a. DOI: <https://doi.org/10.1016/j.jfoodeng.2012.10.048>.

SAFIEI, N. Z. *et al.* Grape juice concentration by progressive freeze concentrator sequence system. **Journal of Food Processing and Preservation**, v. 41, n. 1, p. 1-11, 2017. DOI: <https://doi.org/10.1111/jfpp.12910>.

SUÁREZ-EIROA, B. *et al.* Operational principles of circular economy for sustainable development: Linking theory and practice. **Journal of cleaner production**, v. 214, p. 952-961, 2019. DOI: <https://doi.org/10.1016/j.jclepro.2018.12.271>.

WEB OF SCIENCE. **Relatório de citações da coleção do Web of Science entre 1994 a 2023**. Tópico “Cryoconcentration”. [Acesso em 01 nov. 2023]. Disponível em <https://www.webofscience.com/wos/woscc/citation-report/ba89a8e3-8164-45ed-87ea-a0b20cb4803a-b0659a2b?page=1>.

WU, Y.-Y. *et al.* Influence of freeze concentration technique on aromatic and phenolic compounds, color attributes, and sensory properties of Cabernet Sauvignon wine. **Molecules**, v. 22, n. 6, p. 899-917, 2017. DOI: <https://doi.org/10.3390/molecules22060899>.

ZHANG, Q. *et al.* Effect of suspension freeze-concentration technology on the quality of wine. **South African Journal of Enology and Viticulture**, v. 37, n. 1, p. 39-46, 2016.

ZIELINSKI, A. A. *et al.* Effect of cryoconcentration process on phenolic compounds and antioxidant activity in apple juice. **Journal of the Science of Food and Agriculture**, v. 99, n. 6, p. 2786-2792, 18 dez. 2018.

DOI: <https://doi.org/10.1002/jsfa.9486>.