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CRYOCONCENTRATION: AN EMERGING TECHNOLOGY FOR THE CONCENTRATION OF BIOACTIVE COMPOUNDS

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ABSTRACT

Conventional thermal concentration methods often degrade bioactive compounds in foods due to high-temperature exposure. Cryoconcentration has emerged as a promising non-thermal alternative, offering improved retention and concentration of these sensitive compounds. This review systematically analyzes 22 articles published between 2020 and 2024, sourced from Scopus, ScienceDirect and PubMed, using the PRISMA methodology. The evidence highlights the effectiveness of block cryoconcentration – with or without assistance – in increasing the concentrations of phenolic compounds, flavonoids, anthocyanins, and antioxidant activity, by up to 9-, 4-, 6-, and 11-fold, respectively. Moreover, the technique achieves retention rates exceeding 80% after three cycles and reduces energy consumption by up to 86% compared to traditional thermal methods. With advancements in scaling for industrial applications, cryoconcentration holds significant potential for the large-scale recovery and incorporation of bioactive compounds into functional food systems, enabling the production of nutritionally rich and functionally enhanced products.

Keywords: freezing concentration, non-thermal concentration, phenolic compounds, volatile compounds

INTRODUCTION

Concentration processes play a vital role in the agri-food industry, particularly for liquid food products such as fruit juices, where they contribute significantly to reducing transportation and storage costs, while also enhancing shelf life (Tavares et al., 2022; Gulied et al., 2023). Conventional concentration is typically achieved through thermal methods such as evaporation (Cochachin et al., 2023). However, high-temperature processing is known to degrade thermolabile components, including bioactive compounds (BC) and certain vitamins (Silva et al., 2020).

To address these limitations, the industry has been exploring innovative concentration technologies that more effectively preserve biological compounds (Orellana et al., 2020b). Non-thermal methods such as reverse osmosis (RO) and membrane concentration (MCC) have emerged as promising alternatives for concentrating BC in liquid foods (Julian et al., 2022; Zhang et al., 2024). However, their broader adoption is constrained by high initial capital costs, relatively low concentration levels (25–40%) (Khan et al., 2024), and

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operational challenges such as concentration polarization, increased osmotic pressure, and membrane fouling (Gulied et al., 2023).

Against this backdrop, cryoconcentration (CC), also known as freeze concentration, has gained recognition as a compelling non-thermal concentration technology (Osorio et al., 2024). The process involves freezing the aqueous portion of a liquid, forming pure ice crystals, which are then removed, leaving behind a concentrated solution with a high content of bioactive compounds (Tobar et al., 2021; Camelo et al., 2021; Ding et al., 2021). Unlike thermal methods, CC operates at sub-zero temperatures, which minimizes degradation of sensitive compounds (Casas et al., 2020; Samsuri et al., 2020) and circumvents the limitations associated with other concentration methods, such as evaporation, RO and MCC (Haas et al., 2022). Additionally, cryoconcentration is notably energy-efficient, requiring as little as 14% of the energy used in conventional evaporative processes (Guerra et al., 2021).

There are three primary types of cryoconcentration:

1. **Suspension cryoconcentration** (CCS) – the oldest and most widely implemented method on an industrial scale (Almeida et al., 2023)
2. **Progressive cryoconcentration** (CCP) – involves layer-by-layer crystallization under constant agitation, which forms a large ice mass on a cold surface to facilitate separation (Prestes et al., 2022)
3. **Block cryoconcentration** (CCB) – entails a partial or complete freezing of the solution, followed by thawing and separation of the concentrated liquid from the ice fraction (Santos et al., 2023; Vásquez et al., 2023).

While CCS remains the most widely implemented, and CCP is increasingly applied (Casas et al., 2020), CCB has gained significant interest in recent years due to its simple equipment requirements, effective separation efficiency (>60%), and capacity to increase the concentration of bioactive compounds by 2- to 10-fold compared to initial levels (Maciel and Teixeira, 2022; Orellana at al., 2021b).

Given this context, a critical synthesis of current research on cryoconcentration is both timely and essential. This review addresses the following research question: What is the current state of scientific knowledge regarding the use of cryoconcentration for the enrichment of bioactive compounds in agri-food systems? The review is warranted by the need for a comprehensive overview of scientific advancements in BC cryoconcentration, which is crucial for future research on the technology's applicability to new food matrices and agro-industrial products. These insights are increasingly important in addressing evolving consumer demands for health-oriented, functional foods (Almeida et al., 2023; Siddiqui et al., 2024).

Accordingly, the objective of this review is to systematically analyze scientific publications from the past five years on the use of cryoconcentration – particularly block cryoconcentration – for the enrichment of bioactive compounds in agri-food systems. Furthermore, it explores potential synergies with other emerging preservation technologies, such as pulsed electric fields (Vidal et al., 2024) and micro- or nanoencapsulation techniques (Rodrigues et al., 2024), to improve the stability, functionality, and scalability of cryoconcentrated products for the functional food industry.

METHODS

Bibliographic sources and search strategy

A systematic literature search was conducted in accordance with the PRISMA methodology guidelines (Page et al., 2021) across the following databases: PubMed, Scopus and ScienceDirect. The search was conducted in November 2024. The search strategy was developed using the PIO (Population, Intervention and Outputs) framework, with boolean operators (AND, OR) to structure the query. The following keywords were used:

- ("Bioactive compounds" OR "biological compounds" OR "thermolabile compounds" OR "phenolic compounds")
- AND ("cryoconcentration" OR "concentration freezing" OR "non-thermal concentration" OR "block concentration")
- AND ("Retention efficiency" OR "Increase bioactive compounds").

Inclusion and exclusion criteria

This review focused on original research articles published between 2020 and 2024 that specifically addressed the cryoconcentration of bioactive compounds. To ensure comparability and replicability, studies were required to provide details on key parameters such as freezing and thawing conditions, retention efficiency, and the number of cycles or applications of cryoconcentrates within the agri-food industry.

Studies were excluded if they did not explicitly address the cryoconcentration of bioactive compounds, or if they were systematic reviews, narrative reviews, books, or book chapters. This approach aimed to minimize biases associated with secondary analyses and avoid redundancy in the collected data.

Data selection, extraction and analysis

Following the database search, the retrieved articles were exported and independently reviewed by five researchers. Any discrepancies in article selection were resolved through consensus. Data selection and extraction were facilitated using Rayyan IA and Microsoft Excel. The selected studies were organized and stored using the Mendeley reference management software.

To identify key trends, patterns, and research networks within the field, a bibliometric analysis was performed using VosViewer and Bibliometrix. This allowed the visualization of co-occurrence networks and the identification of significant research patterns within the scientific literature.

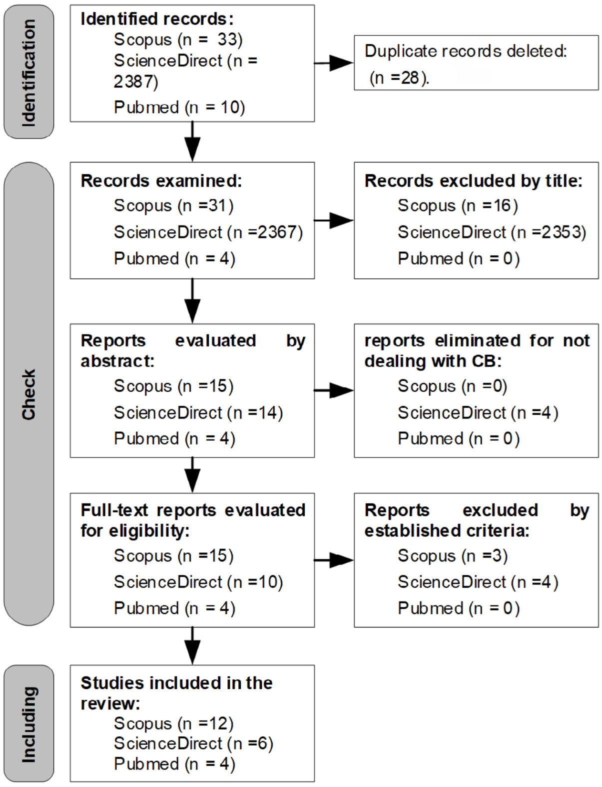

Fig. 1. Study identification and selection process

RESULTS AND DISCUSSION

Selection of studies

The literature search identified 2,430 articles across three databases. After eliminating 28 duplicates using Rayyan IA, five reviewers evaluated the studies based on titles, abstracts, and full texts according to the inclusion and exclusion criteria. Ultimately, 22 articles were selected for the final analysis (Fig. 1).

Characterization of studies and synthesis of results

The selected studies are presented chronologically in Table 1, which categorizes the studies by author, year of publication, product, type of cryoconcentration, freezing conditions, centrifugation parameters, and the results reported by each study.

Cryoconcentration versus traditional concentration methods

Cryoconcentration has demonstrated the ability to concentrate liquid foods at low temperatures by separating pure ice crystals from the final concentrated (cryoconcentrated) solution (Prestes et al., 2022). This process offers distinct advantages over traditional methods, such as evaporation, particularly in preserving thermolabile compounds. (Tobar et al., 2021). For instance, Orellana et al. (2021a) compared cryoconcentration by block centrifugation and evaporation (50°C) for concentrating bioactive compounds in pomegranate juice. They found that cryoconcentration resulted in 5.19, 5.12, 5.29, and 4.05-fold increases in total phenolic content (TPC), total antioxidant capacity (TAC), tannins, and total flavonoid content (TFC), respectively, compared to 3.11, 3.60, 3.87 and 1.97 increases with evaporation. Additionally, cryoconcentration achieved 87% retention efficiency of thermolabile compounds, compared to 68% for evaporation, concluding that cryoconcentration is a non-thermal technology that preserves bioactive compounds in fresh pomegranate juice (Tab. 2).

Similarly, Zhang et al. (2024) compared three concentration methods (vacuum thermal concentration

Table 1. Studies included in the systematic review on cryoconcentration of bioactive compounds

N°	Author and year	Product	Type of CC	Freezing parameters	Defrosting	Results
01	Orellana et al., 2020a	apple juice (14°Brix)	centrifuge-assisted CCB	–20°C 12 h	4000 rpm 20°C 15 min	The research reported an Eff of 73% and 55°Brix, with concentrations of 819 mg GA and 248 mg CEQ for TPC and TFC in 100 g, respectively, achieving a retention of over 60%.
02	Orellana et al., 2020b	juice of calafate (13.9°Brix)	centrifugation-assisted CCB	–20°C 12 h	4000 rpm 15 min	The study reported that CCB increased TBC and AA by 2.5 and 5.2 times, respectively, reaching 42°Brix at the end of the third cycle.
03	Orellana et al., 2021b	blueberry juice (13.8°Brix)	gravitational CCB	–20°C 12 h	20°C 15 min	It was reported that filter-centrifuge CCB achieved higher increases in TPC, TAC, TFC and AA concentration compared to gravity CCB and centrifugal CCB, with increases of 2.1, 1.8, 2.0 and 3.2-fold, respectively, in relation to fresh cranberry juice. Additionally, it achieved an Eff of 86%, a °Brix of 35.9 and retentions of 80%, 75% and 69%.
			centrifuge-assisted CCB		3850 rpm 20°C 15 min	
			CCB assisted by filter-centrifugal		3850 rpm 20°C 15 min	
04	Guerra et al., 2021	murta juice (14°Brix) and arrayan juice (15.1°Brix)	centrifugation-assisted CCB	–20°C 12 h	4000 rpm 20°C 20 min	The CCB process concentrated TPC to 20 and 66 mg EAG/100 g, and TAC to 13 and 25 mg cyanidin-3-glucoside/100 g for murta and arrayan, respectively. AA was 2.1 times higher compared to the initial value, and Effs of 65% and 85% were achieved, with final contents of 48 and 54°Brix.
05	Casas et al., 2021a	gelatin with blueberry juice (13°Brix)	CCB	–20°C 12 h	2800 rpm 15°C 20 min	The study determined that the gelatin incorporated blueberry juice cryoconcentrate presented 55 mg GAE/100g and 530 μmol TE/100g of TBC and AA respectively, reaching a final 45°Brix.
06	Orellana et al., 2021a	pomegranate juice (16.1°Brix)	centrifugation-assisted CCB	–20°C 12 h	1600 rpm 20°C 15 min	It was observed that CCB increased the concentration by 5.19, 5.12 and 4.05 times with respect to the initial value for TPC, TAC and TFC, respectively. Retention was 87%, 71%, 69% and 67% for these compounds, with a final content of 48°Brix.
07	Casas et al., 2021b	blueberry juice (16°Brix)	centrifugation-assisted CCB	–20°C 12 h	4000 rpm 15 min	The results indicated that CCB increased the concentration by 2.7, 3.2 and 1.8 times for TBC, TPC and AA, respectively, compared to fresh juice, achieving a final content of 34°Brix.
08	Vidal et al., 2021	maqui extract (10.5°Brix) and calafate (6.8°Brix)	centrifuge-filtration-assisted CCB	–20°C 12 h	4000 rpm 10 min	Concentrations of TPC and AA increased by 4.8 to 5.0 and 5.8 to 3.4 times, respectively. Eff was 95%, with final contents of 49.72°Brix for maqui extract and 49 and 46°Brix for calafate.
09	Meneses et al., 2021	green tea extract (4°Brix)	gravitational CCB	–20°C 12 h	gravity	CCB was able to concentrate catechins and polyphenols by 4.5 and 3.4 times, respectively, with an Eff of 81.3% and a final content of 14.1°Brix.
10	Bastía et al., 2022	maqui extract (16.2°Brix)	centrifuge-filtration-assisted CCB	–20°C 12 h	4000 rpm 10 min	A concentration of 2.8 and 6.7 times of TPC and TAC was reported, respectively, with Eff of 95% and a content of 70°Brix.

11	Haas et al., 2022	orange juice (10.7°Brix)	gravitational CCB	–20°C 12 h	gravity	A concentration increase of 429% and 343% in polyphenols and AA, respectively, was recorded, with an Eff of 62.52% and a content of 38.06°Brix.
12	Maciel and Teixeira, 2022	pecan Cake Drink	gravitational CCB	–20°C 12 h	gravity	The gravitational CCB process allowed a 2.6-fold increase in TPC content (from 395.29 to 1008.03 mg GAE/100 g) and a 1.9-fold increase in AA measured by the DPPH method. In addition, an Eff of 85% and a retention of 98% were achieved after completion of the five cycles.
13	Rezzadori et al., 2022	guava leaf extract	gravitational CCB	–16°C 24 h	controlled atmosphere at 17 °C	The results reported an Eff of 80% and 73%; a concentration of 3 and 9 times for TPC and 3 and 11 times for AA using gravitational and microwave thawing, respectively.
			microwave-assisted CCB		1400 W	
14	Arend et al., 2022	beet leaf extract	gravitational CCB	–24°C 12 h	gravity	The research concludes that, after 4 cycles of concentration by gravitational CCB, an 8-fold increase in TPC content (from 0.88 to 7.18 mg GAE/mL) and betalains was achieved; and a 5-fold increase in AA, with an Eff of 90%.
15	Almeida et al., 2023	*Moringa citrifolia* leaf tea.	gravitational CCB	–20°C 12 h	gravity	The study achieved an Eff of 91.15% and achieved a 6.82-fold increase in TPC compared to the initial sample after 4 cycles.
16	Márquez et al., 2023	prickly pear juice (13.8°Brix)	centrifugation--assisted CCB	–20°C 12 h	2910 rpm 20 min	The study concludes that, compared to the initial value, an increase of 1.8 and 2.8 times in TPC and TFC was achieved, respectively, with a content of 33.1°Brix.
17	Bredun et al., 2023	grape pomace extract	gravitational CCB	–20°C 12 h	gravity	The research concluded that, at the end of the third cycle, the concentration increased 4 and 5 times for TPC and TAC, respectively, with efficiencies of 93% and 91%.
18	Vásquez et al., 2023	pomegranate juice (15.8°Brix)	centrifuge-assisted CCB	–20°C 48 h	1000 rpm 12 min	The research concluded that CCB increased the TBC by 3 times with respect to the initial value, achieving an Eff of 84.3% and 59.2°Brix when centrifugation was used, while with vacuum an Eff of 81.9% and 47.3°Brix was achieved.
			vacuum-assisted CCB		10 kPa 12 min	
19	Marafon et al., 2024	acerola pulp (6.76°Brix)	gravitational CCB	–20°C 12 h	gravity	The results reported that CCB increased TFC by 166.90%, AA by 112.10%, with an Eff of 78% and 18.3°Brix.
20	Osorio et al., 2024	Craft beers (Witbier, Porter and Bitter)	agitated CCP	–15°C (Witbier Porter), –20°C (Bitter) for 12 h	300 rpm 1 h	The CCP achieved a 1.26-fold increase in TPC, reporting a concentration of 213.5, 438.5 and 370 mg/L in the Witbier, Porter and Bitter beers, respectively.
21	Vásquez et al., 2024	pomegranate juice	centrifuge-assisted vacuum CCB	–20°C 12 h	2360 rpm vacuum 20 kPa 10 min	The results indicated a concentration of 5665 mg GAE/L of TPC, with an Eff of 90.2%, and a content of 54.9°Brix after three cycles of CCB.
22	Zhang et al., 2024	sea buckthorn juice (16°Brix)	centrifuge-assisted CCB	–20°C 12 h	10000 rpm 10 min 25°C	The study concluded that CCB achieved increases of 21.3%-26.01%, 8.4–23.94% and 17.35–22.02% for CPT, CFT and AA, respectively, with superior retention of 78.7%, 86.67% and 82.65% in each case.

Note: Eff = process efficiency; TPC = total phenolic content; TAC = total anthocyanin content; TFC = total flavonoid content; AA = antioxidant activity; TBC = total bioactive compounds.

Table 2. Comparison of cryoconcentration (CC) with traditional methods of concentration (adapted from Tobar et al., 2021 and Prestes et al., 2022)

Concentration method		Process	Advantages	Disadvantages
Thermal	evaporation	removes solvent through heat application	• technology widely used at the industrial level	• degradation of bioactive and heat-sensitive compounds • sensory and nutritional alterations.
Non--thermal	reverse osmosis	removes water by applying external pressure without a phase change	• preserves nutrients and bioactive compounds • low thermal energy consumption	• membranes are prone to fouling and require regular maintenance
	CC (cryoconcentration)	freezes and separates water as ice crystals, concentrating the extract	• preserves bioactive compounds • maintains sensory characteristics • uses only 14% of the energy required for evaporation	• requires optimization of equipment and processes for industrial applications

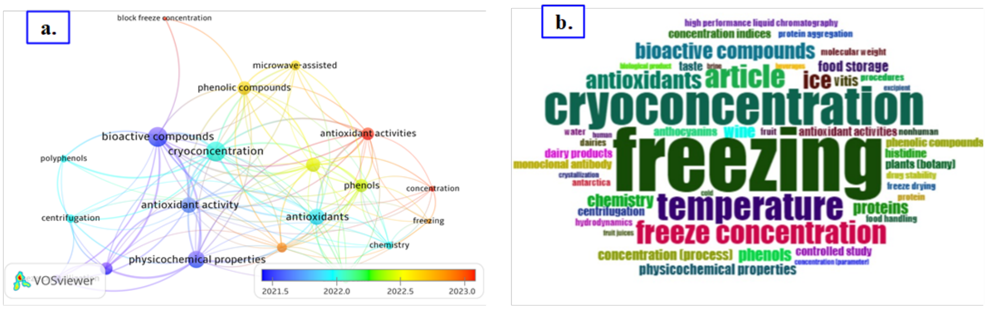

Fig. 2. Word co-occurrence analysis: (a) Network and cluster visualization based on the 22 included studies (generated with VosViewer); (b) Keyword distribution of the studies according to Scopus, categorized according to the PIO strategy (generated with Bibliometrix)

at 60°C, vacuum cryoconcentration, and reverse osmosis membrane concentration) for their effect on volatile components in sea buckthorn juice. Cryoconcentration achieved the highest retention of TPCs (91.6%), compared to 86.67% with thermal concentration and 76.06% with reverse osmosis. Thermal concentration reduced TPC, TFC and ascorbic acid by 26.01%, 23.94% and 22.02%, respectively, and negatively impacted sensory characteristics, causing a 54.7% increase in the browning index and affecting flavor. Overall, cryoconcentration outperforms conventional methods by yielding extracts/products with superior nutritional, sensory, functional and biological properties, while being environmentally sustainable and cost-effective (Prestes et al., 2022).

Identification and co-occurrence analysis of key terms

A bibliometric analysis conducted using VosViewer and Bibliometrix identified and quantified key term frequencies through co-occurrence networks, providing a detailed view of thematic interrelationships across the analyzed articles (Fig. 2a and 2b). Figure 2a highlights frequent terms such as "cryoconcentration" (30 occurrences), "bioactive compounds" (29 occurrences), and "antioxidant activity" (14 occurrences), demonstrating their relevance within the analyzed studies. Figure 2b, based on articles obtained from the Scopus database using the PIO strategy, shows that terms such as "freezing" (36 occurrences), "cryoconcentration" (22 occurrences), and "bioactive compounds"

(9 occurrences) are the most prominent. This analysis not only identifies key concepts but also reveals trends and research priorities in the scientific literature on the cryoconcentration of bioactive compounds, providing a solid foundation for future investigations in this area.

The analysis also revealed that block cryoconcentration (CCB) is the most studied method in the literature. CCB typically involves freezing the liquid solution below the freezing point (usually at –20°C) (Márquez et al., 2023), followed by partial thawing through simple thawing or mechanical forces to separate two fractions (Haas et al., 2022): (I) the cryoconcentrated solution, which serves as feed for subsequent cycles, and (II) the highly pure ice fraction (Marafon et al., 2024). This process can be repeated across multiple cycles (Fig. 3).

As shown in Figure 3, various complementary methods have been employed to facilitate thawing and separation of the cryoconcentrated solution from the ice matrix. These include microwave-assisted thawing (Rezzadori et al., 2022), centrifugation, vacuum processes, and centrifugation-filtration (Bastías et al.,

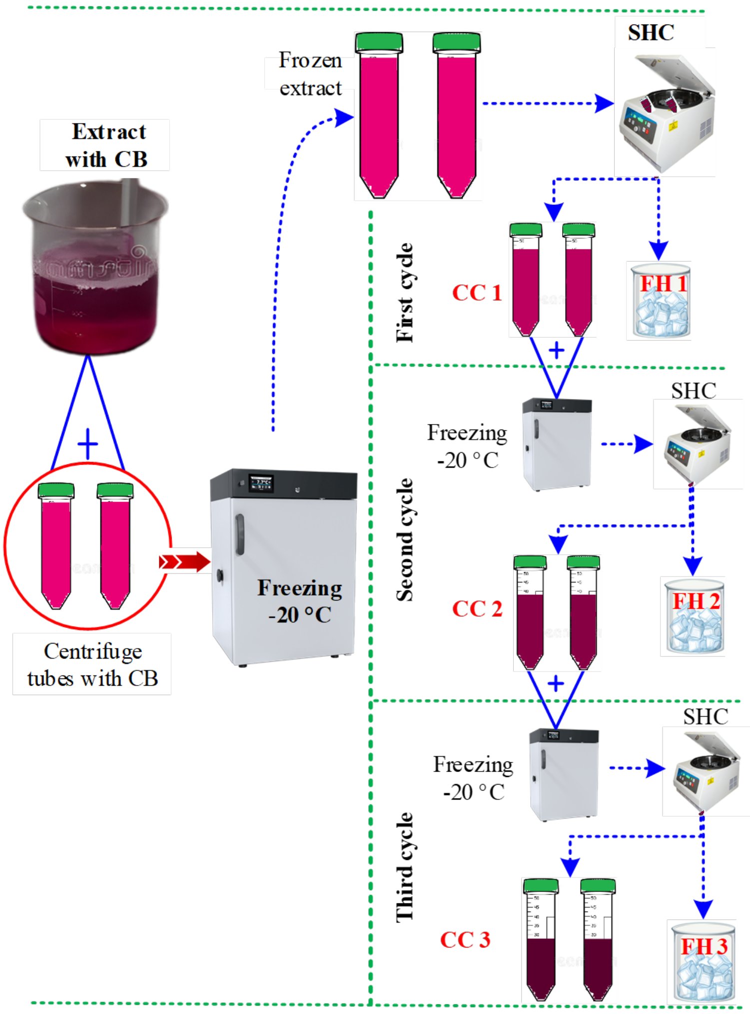

Fig. 3. General procedure of CCB across three centrifugation cycles: SHC – separation of the ice fraction; (CC 1-3) – cryoconcentrates per cycle; (H 1-3) – ice fractions per cycle

2022). Such enhancements help obtain cryoconcentrated extracts with superior bioactive, nutritional, and sensory qualities compared to the original extract and conventional concentration techniques (Vásquez et al., 2024).

Block cryoconcentration: gravity thawing, microwave-assisted thawing, vacuum thawing and centrifugation

According to the reviewed scientific literature, the most extensively investigated thawing methods for block cryoconcentration (CCB) are gravity thawing, microwave-assisted thawing, vacuum thawing, and centrifugation, either applied independently or combined with vacuum or filtration to improve efficiency. Across various studies, the choice of thawing technique has been shown to play an important role in efficiency, solute yield, and concentration percentage (Orellana et al., 2021b; Vásquez et al., 2023).

Gravity-based thawing (CCBG) is a relatively low-cost and user-friendly technique, owing to the simplicity of the equipment involved (Bredun et al., 2023). However, its main drawbacks – namely low separation efficiency and extended processing times – limit its practical applicability (Vásquez et al., 2023). In response to this, techniques such as centrifugal (CCBC) and microwave-assisted (CCBM) thawing have been developed to enhance solute concentration and reduce thawing durations (Casas et al., 2021a).

Recent findings (Fig. 4) underscore the superior performance of CCBM, which has been shown to increase phenolic content and antioxidant activity by factors of up to 9 and 11 respectively, while also improving the bioaccessibility of total phenolic compounds (TPCs) during simulated digestion (Rezzadori et al., 2022). Similarly, Orellana et al. (2021b) reported a polyphenolic retention of 80% in cranberry juice using centrifugal filtration-assisted CCB, compared to 69% using gravity-based CCB. Both CCBM and CCBC can reduce thawing times from several hours to just minutes while achieving high retention and effective separation of bioactive compounds (Arend et al., 2022).

Recovery and cryoconcentration of bioactive compounds from agri-food waste

In alignment with circular economy principles, several studies have highlighted the valorization of agri-food waste (Nabi et al., 2024), which contains high

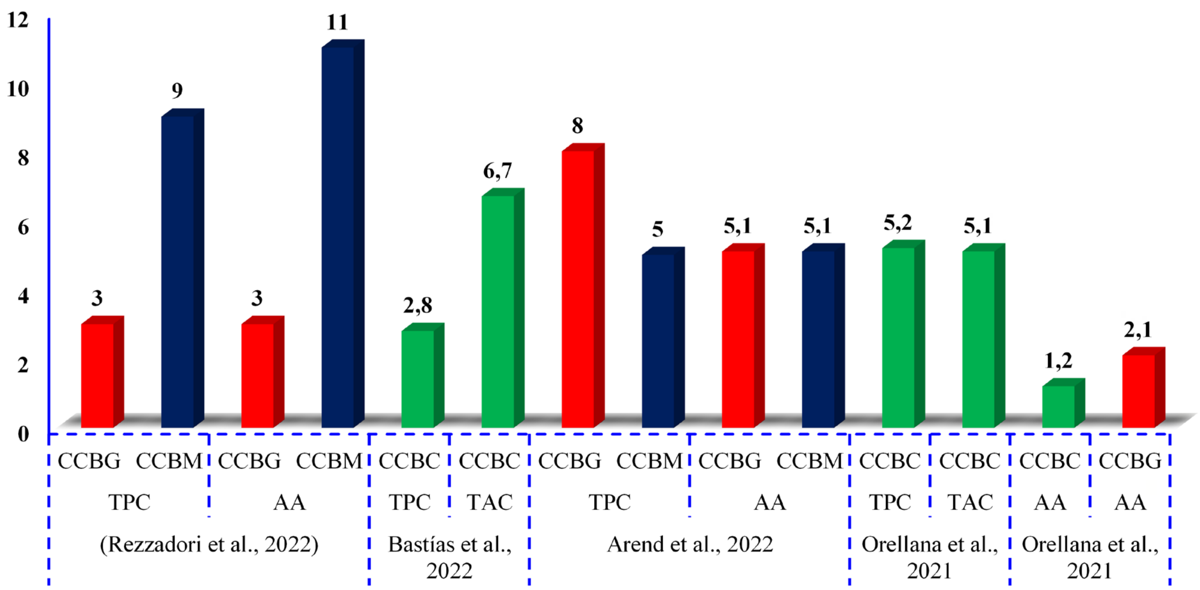

Fig. 4. Number of BC concentration cycles at the end of CCB using different thawing techniques. CCBG – block cryoconcentration with gravity thawing; CCBM – microwave-assisted block cryoconcentration; CCBC – centrifugation and vacuum-assisted block cryoconcentration

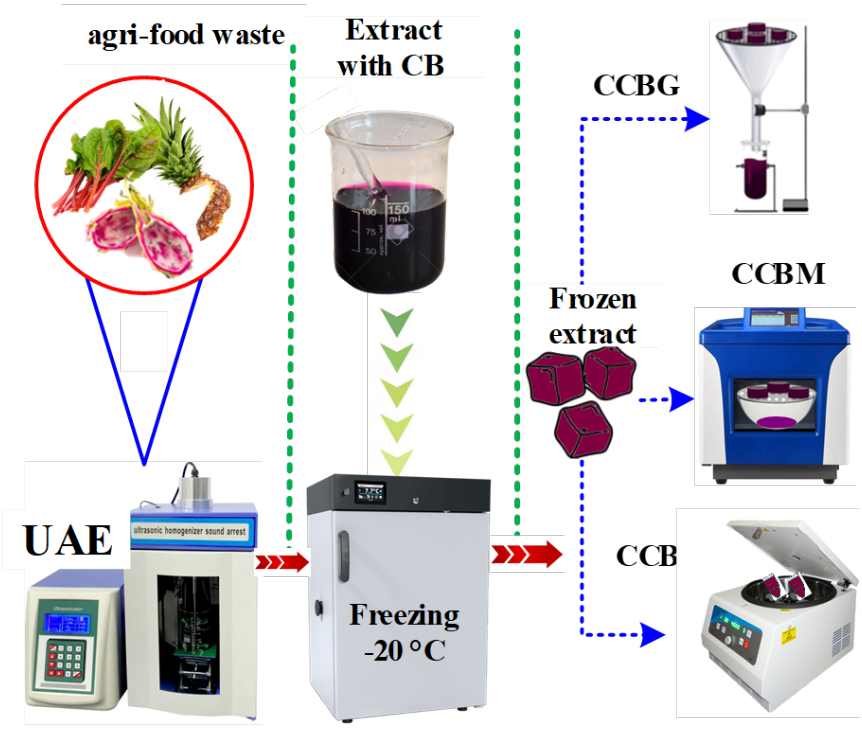

Fig. 5. Extraction and cryoconcentration of BC from agri-food biowaste

levels of bioactive compounds with potent antioxidant properties (Castro et al., 2024). These compounds are typically extracted using green technologies such as ultrasound-assisted extraction (UAE), microwave-assisted extraction (MAE), and supercritical fluid extraction (Ray et al., 2023; Singh et al., 2024). Once extracted, the compounds are subjected to block cryoconcentration or related techniques, as illustrated in Figure 5.

For instance, Bredun et al. (2023) applied gravity-based CCB to grape pomace, achieving phenolic concentrations between 276 and 1264 mg/L and anthocyanin levels ranging from 113 to 567 mg/L by the end of the third cycle. Arend et al. (2022) reported a five-fold increase in phenolic content – from 1.01 to 5.14 mg GAE/mL – in beet bioresidue (leaf) extract after the fourth cycle of microwave-assisted CCB, without compromising the extract's quality. These studies highlight the synergistic potential of combining innovative extraction methods with cryoconcentration technologies to valorize agri-food biowaste within a circular economic framework.

Challenges and future perspectives of cryoconcentration

While cryoconcentration offers a sustainable and effective solution for preserving thermolabile biological compounds (Haas et al., 2022), its industrial scalability remains a major challenge – particularly for block cryoconcentration (CCB). To date, only progressive cryoconcentration (CCP) and suspension cryoconcentration (CCS) have reached industrial scale (Orellana et al., 2020a). Consequently, there is a pressing need for the development of high-capacity equipment designed specifically for CCB, which has consistently demonstrated superior retention and separation performance (Maciel and Teixeira, 2022).

An additional consideration is the stability of cryoconcentrates products. Non-thermal preservation technologies such as pulsed electric fields (PEF) and high-pressure homogenization (HPH) show promise in enhancing microbiological quality and extending the shelf life of bioactive compounds during storage and their integration into food systems (Vidal et al., 2024). Furthermore, advanced delivery systems techniques such as micro- and nanoencapsulation may play a critical role in protecting these compounds under adverse environmental conditions, enabling the development of biofortified foods with enhanced functional properties (Rodrigues et al., 2024; Souza et al., 2024).

CONCLUSION

Cryoconcentration represents an emerging, and eco-efficient technology for concentrating bioactive compounds with minimal degradation, offering clear advantages over traditional thermal methods such as evaporation. Among the various cryoconcentration strategies, block cryoconcentration – particularly microwave-assisted CCB – has demonstrated notable potential, achieving concentration efficiencies exceeding 85% and bioactive enrichment up to ninefold. However, the broader adoption of this technology – especially CCB – hinges on the development of scalable industrial systems.

To fully realize the potential of cryoconcentration, future research should focus on enhancing the stability and functionality of cryoconcentrates through the use of innovative non-thermal technologies, such as pulsed electric fields. Additionally, investigating micro- and nanoencapsulation techniques could be key to optimizing the protection and preservation of bioactive compounds.

DECLARATIONS

Data statement

All data supporting this study has been included in this manuscript.

Ethical Approval

Not applicable.

Competing Interests

The authors declare that they have no conflicts of interest.

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