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## Green extraction technologies for valorising Brazilian agri-food waste

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Brazil generates a large amount of agri-food waste due to its extensive agricultural and food processing activities, presenting both an environmental challenge and an opportunity for value generation. This review examines the current status and key challenges associated with food waste in Brazil, exploring how principles of the circular economy can be applied to address these issues. Emphasis is placed on the use of green extraction technologies to recover valuable bioactive compounds from agricultural byproducts, with applications across the food, pharmaceutical, and cosmetic sectors. A critical evaluation of various extraction methods, including those utilizing green solvents and energy-efficient processes, reveals that while promising, the effectiveness of these techniques varies significantly depending on the specific methodology employed. Different extraction approaches yield extracts with distinct compositions, underscoring the importance of comparative studies and process optimization. By aligning waste management practices with circular economy principles, Brazil can position itself as a leader in sustainable agricultural and food innovation.

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### Sustainability spotlight

The Brazilian agri-food sector generates substantial volumes of underutilized waste rich in bioactive compounds, offering potential for high-value applications. This review examines how green extraction technologies can transform these residues into valuable resources, supporting circular economy strategies, reducing environmental burdens, and improving resource efficiency. By critically assessing current methods and identifying research gaps, the work promotes sustainable innovation in food systems. It aligns with UN Sustainable Development SDG 2, especially Goal 2.4, which aims to ensure sustainable food production systems, as well as SDG 9 (Industry, Innovation and Infrastructure), SDG 12 (Responsible Consumption and Production), and SDG 13 (Climate Action).

## 1. Introduction

The increasing demand for food, driven by global population growth and changing consumption patterns, has placed immense pressure on agricultural and food production systems. While the agri-food sector plays a pivotal role in ensuring food security and economic development, it also generates substantial amounts of waste at every stage of the supply chain, from production and processing to consumption and distribution. In Brazil, a country renowned for its agricultural activity, the large volume of agri-food waste generated represents both an environmental challenge and an untapped reservoir of economic potential.<sup>1,2</sup>

Food waste is not merely an issue of inefficiency but is directly linked to broader global concerns such as hunger, climate change, and resource depletion. This waste includes

a wide array of byproducts, such as bagasse, peels, seeds, shells, and pulp residues, that often retain valuable nutrients and bioactive compounds. When these materials are discarded, essential resources such as water, energy, and labor are also wasted, thereby further enhancing the environmental footprint of the food system.<sup>1,3</sup>

In response to these challenges, the concept of a circular economy has emerged as a promising alternative to the traditional linear economic model. Circular economic principles emphasize reducing, reusing, and recycling materials within the production and consumption cycle, aiming to extend the lifespan of resources and minimize waste. This approach has garnered increasing attention in Brazil, particularly with the enactment of policies such as the National Solid Waste Policy and the recent National Circular Economy Strategy, which support sustainable innovations and waste valorization.<sup>4-6</sup>

Within this context, green extraction technologies represent a vital tool for achieving circularity in the agricultural and food sectors. These innovative methods offer environmentally friendly alternatives to conventional extraction processes, enabling the recovery of high-value compounds from agri-food

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waste without relying on toxic solvents or energy-intensive operations. Green extraction not only aligns with circular economy goals but also presents new opportunities for the development of functional ingredients, bioplastics, pharmaceuticals, and other value-added products.<sup>7,8</sup>

The Brazilian agri-food industry, marked by its vast diversity, offers significant potential for the application of green extraction technologies.<sup>2,9</sup> Commodities such as sugarcane, soybeans, oranges, coffee, and cashew apples produce substantial volumes of byproducts with diverse compositions, including phenolic compounds, fibers, and alkaloids. These materials can be effectively repurposed through tailored extraction

techniques, such as ultrasound-assisted extraction (UAE), supercritical carbon dioxide extraction (SC-CO<sub>2</sub>), and deep eutectic solvents (DESS), each of which is suited to specific compounds and applications.

The agro-industry generates over 2 billion tons of waste globally, highlighting the need for advanced utilization methods to maximize benefits and minimize environmental and economic impacts.<sup>10</sup> Despite the growing interest in sustainable extraction methods, existing reviews primarily focus on either specific waste streams (e.g., citrus peels and coffee grounds) or individual extraction technologies, lacking a comprehensive integration of Brazilian agro-industrial waste



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valorisation within circular economy frameworks. Recent reviews<sup>11,12</sup> address green extraction technologies but do not specifically target Brazilian agricultural systems or provide quantitative comparisons of extraction efficiencies across different waste types. Moreover, current literature lacks a systematic analysis of techno-economic feasibility and life cycle assessments for scaled implementation of these technologies in the Brazilian context.<sup>10,13</sup>

This review addresses this knowledge gap by providing a comprehensive analysis of green extraction technologies specifically applied to Brazilian agri-food waste valorisation within a circular economy framework. Unlike previous reviews, this work integrates quantitative performance data, techno-economic analysis, and sustainability assessments to evaluate the industrial viability of different extraction approaches. This paper focuses on sustainable food technology and waste valorization—a key aspect of the circular economy that is crucial for addressing environmental, economic, and health challenges, providing a comprehensive review of recent publications (2020–2025) regarding Brazilian agro-industrial waste and evaluating the feasibility of using green extraction methods—particularly physical and chemical techniques—to convert these wastes into marketable products. The review emphasizes products for which Brazil is a leading global exporter, underscoring the relevance of this research not only to Brazil's agro-industrial system but also to global sustainable food systems. This review not only addresses the status and challenges of food waste management in Brazil but also places a distinct emphasis on the application of green extraction technologies tailored to the country's specific agricultural byproducts. Unlike previous reviews, which provide a broad overview of extraction methods, this research evaluates the potential of green extraction technologies in Brazil's context, considering the country's diverse agri-food waste streams, regional products, and the economic and environmental implications for Brazil and the whole world.

## 2. An overview of the agri-food industry and the current scenario of food waste

The global agri-food sector plays a crucial role in sustaining the world's growing population, ensuring food security, and driving economic development. While the agri-food industry typically refers to agriculture and food processing activities, the whole sector encompasses all operations within the food supply chain, including harvesting, manufacturing, transportation, marketing, retailing, and consumption.<sup>14,15</sup> Countries with strong agri-food industries, such as the United States, China, and Brazil, play a vital role in feeding the world while facing significant challenges related to resource management, environmental impact, and food waste.

Brazil's significant contribution to global agribusiness is unquestionable. The country plays a leading role as a worldwide supplier of agribusiness products. According to the 2024 Food and Agriculture Organization (FAO) ranking, this country is the world's largest producer of several commodities, including

sugar cane, coffee, soybeans, and oranges.<sup>16</sup> The country's ample agricultural land, favorable climate, and advancements in farming technology have positioned it as a powerhouse in the agri-food sector.

A challenge faced by Brazil and the worldwide agri-food sector is food waste, which has severe environmental, economic, and social consequences.<sup>17</sup> The agri-food industry generates a substantial amount of food waste, accounting for a significant fraction of all food produced. Additionally, food waste represents a substantial loss of resources, including water, energy, labor, land, and capital, further exacerbating environmental and economic challenges.<sup>18</sup>

Globally, approximately 1.3 billion tons of edible food are lost or wasted each year, resulting in the emission of around 3.3 billion tons of greenhouse gases. However, the volume of food waste in Brazil remains uncertain.<sup>1,19</sup> Food waste occurs at all stages of the food supply chain, from post-harvest losses to inefficiencies in distribution, transportation, processing, wholesale, retailing, market dynamics, food service, and household consumption.<sup>9,15,17</sup> Each stage presents unique challenges and opportunities for reducing waste and maximizing the value of resources. Therefore, measuring food waste enables countries to understand the scale of the problem and, thus, the size of the opportunity.<sup>1</sup>

## 3. Circular economy

Governments and the food industry have made reducing or eliminating food waste a top priority, with the circular economy recognized as a promising and strategic approach for achieving this goal. Unlike the traditional linear economy, which follows a one-way flow of resource extraction, production, consumption, and disposal, the circular economy offers a sustainable alternative. The linear model leads to the intensive exploitation of natural resources and significant waste of materials and energy. In contrast, the circular economy aims to close the loop by promoting resource efficiency, reuse, and recycling, ultimately minimizing environmental impact and conserving finite resources.<sup>6,20</sup>

The transition to a more sustainable economy, such as the circular economy, offers a viable approach for addressing these challenges and promoting a balance between production, consumption, and environmental preservation. Although there are several definitions of the circular economy, its main objective is to minimize excessive resource consumption and waste generation by promoting the circulation of materials through multiple cycles of use.<sup>21</sup>

Due to its vast potential, the circular economy has become a key pillar of international policies aimed at sustainable development. It fosters technological innovation, enhances productive efficiency, and supports ecosystem regeneration, making it a globally encouraged approach. Across different regions, government initiatives and international agreements, such as the European Union's Circular Economy Action Plan, have established ambitious goals and facilitated collaborations among governments, industries, and communities to accelerate the adoption of circular practices globally.<sup>8,22,23</sup>



In the circular economy, waste management is essential for fostering sustainability throughout both production and consumption processes. Implementing effective waste management practices helps conserve natural resources and extend their availability. Additionally, it plays a key role in minimizing waste generation and reducing greenhouse gas emissions, thereby delivering substantial environmental benefits.<sup>3,6,21</sup>

The objectives outlined in the 2030 Agenda for Sustainable Development align closely with the principles of the circular economy, particularly through the emphasis on reducing food waste and promoting sustainable consumption and production patterns. Circular economy strategies aim to keep resources in use for as long as possible, extract maximum value from them, and regenerate natural systems—an approach that directly supports Sustainable Development Goal (SDG) 12.3, which calls for halving per capita global food waste and minimizing losses along production and supply chains. Addressing food waste not only contributes to environmental sustainability (SDG 13) and food security (SDG 2) but also promotes social and economic equity by transforming waste into valuable resources. Scientific and industrial efforts to repurpose agricultural and food residues into high-value products, such as bioactive compounds, biomaterials, or bioenergy, reflect the practical implementation of circular economy principles. These initiatives close material loops in the food system and foster collaboration across the entire value chain, reducing environmental impact and supporting the transition toward more resilient and sustainable food systems.<sup>24</sup>

In Brazil, the circular economy has gained increasing prominence, driven by economic, environmental, and social challenges. This topic became particularly relevant after the approval of the National Solid Waste Policy (PNRS) in 2010, through Law No. 12,305, which established essential guidelines for the integrated management and proper disposal of solid waste. The PNRS promotes waste reduction, reuse, and recycling while encouraging shared responsibility for the life cycle of products. Its implementation has spurred public policies and business initiatives aligned with circular economy principles, contributing to the transition toward a more sustainable economic model.<sup>4</sup>

On June 27, 2024, Brazil took another significant step toward consolidating the circular economy with the establishment of the National Circular Economy Strategy (ENEC) through Federal Decree No. 12,082/2024. This initiative lays the foundation for shifting from a linear economic model to a system based on circular economy principles, promoting the efficient use of natural resources and the adoption of sustainable practices throughout production chains. ENEC's guidelines include reducing pollution and waste generation, preserving the value of materials, regenerating the environment, decreasing dependence on natural resources, promoting sustainable production and consumption patterns, and extending the life cycle of materials.<sup>5</sup>

As in other sectors, food production and consumption have a significant impact on the environment due to the intensive use of resources and the large volume of waste generated. In

this context, applying circular economic principles is essential, particularly through the valorization of agro-industrial waste. By reintegrating byproducts such as peels, bagasse, seeds, and other residues into new production cycles, agro-industrial waste valorization delivers environmental, economic, and social benefits, thereby fostering more efficient production chains that align with sustainable development.<sup>21</sup>

While the PNRS establishes shared responsibility for waste, it lacks specific fiscal incentives (*e.g.*, tax breaks and subsidized loans) for companies that invest in high-tech valorisation technologies beyond basic recycling. The lack of budgetary incentives makes the high capital expenditure (CAPEX) of technologies like SC-CO<sub>2</sub> or PLE difficult to justify for many small and medium-sized enterprises (SMEs). Products derived from waste valorisation (*e.g.*, a cosmetic ingredient from cashew bagasse) can face regulatory hurdles. The classification and safety approval processes by agencies like ANVISA (Brazilian Health Regulatory Agency) may not be straightforward for materials from “waste” sources, which can result in entry barriers. Brazil's vast territory and logistical deficits present a significant challenge in collecting, segregating, and transporting this biomass from farms to processing plants. Therefore, a well-established supply chain for managing agro-industrial waste is crucial for implementing a circular economy. The enforcement of the PNRS remains inconsistent across different municipalities and states, resulting in a continued preference for cheaper, less sustainable disposal methods, such as landfills.

#### 4. Green extraction and purification technologies for waste valorization

Emerging innovative and greener technologies have been explored to extract the desired compounds from food waste, as an alternative to conventional methods. These emerging technologies offer several advantages over traditional methods, including reduced time and solvent consumption (up to 75% reduction in extraction time), the use of green solvents, higher yields (29–197% improvement in some cases), improved safety, enhanced consideration of environmental and human health impacts, as well as convenience, economic benefits, sustainability, and lower nutrient loss.<sup>15</sup>

In traditional extraction methods, organic solvents such as hexane, methanol, and chloroform are widely used. However, they pose harmful impacts on both handling workers (unsafe to humans) and the environment (environmental toxicity) due to their volatile and toxic nature. An alternative to replace these toxic solvents is green solvents, such as ionic solvents, DESs, edible oils, inert gases, ethanol, hydroethanolic mixtures, and (acidified) water. Besides having a lower environmental impact than traditional solvents and being safe for humans, they may also present better extraction yields, higher recovery and reuse possibilities, and be suitable for extraction at lower temperatures, which improves the stability of heat-sensitive compounds.<sup>15,25</sup>



The solvent is a crucial factor that affects the extraction of the desired compounds. The choice of solvent depends mainly on the desired properties of the compound. For the extraction of lipophilic compounds, non-polar solvents such as supercritical CO<sub>2</sub>, ethanol, and certain ionic liquids, as well as ethyl lactate and ethyl acetate, which are generally recognized as safe (GRAS), are appropriate options.<sup>15</sup> Also, ionic liquids (ILs) and DESs have emerged as promising alternatives to conventional volatile organic solvents due to their non-volatile, non-flammable, and generally non-toxic nature. The tunable ionic composition of IL allows for the extraction of a wide range of bioactive compounds, including both hydrophobic (*e.g.*, carotenoids) and hydrophilic (*e.g.*, flavonoids and anthocyanins) molecules. Furthermore, their potential for recycling and reuse contributes to lowering the environmental impact of extraction processes, aligning well with the principles of the green circular economy.<sup>15</sup>

ILs are salts with melting points below 100 °C, typically composed of a large organic cation and an organic or inorganic anion. The wide range of cation–anion combinations allows them to tailor their properties, earning them the name “designer solvents”. ILs are chemically and thermally stable, non-flammable, highly conductive, and have low vapor pressure, making them effective for isolating organic compounds. Although concerns about their toxicity are growing, ILs remain promising for sustainable chemical and industrial processes due to their efficiency, recyclability, and biocompatibility. In food applications, toxicity concerns are more related to the concentration of IL in the final product rather than its composition.<sup>26</sup>

DESs and natural deep eutectic solvents (NADESs) represent an emerging class of sustainable solvents that have attracted significant research interest due to their unique properties. They are known as a new generation of solvents that comply with the requirements of green extraction and are environmentally friendly, in addition to possessing the GRAS status.<sup>27</sup>

DESs are composed of a hydrogen bond acceptor, such as quaternary ammonium salts, and one or more hydrogen bond donors, including amines, amides, or carboxylic acids, which together substantially lower the mixture's melting point. DES shares key advantages with IL, including chemical and thermal stability, non-flammability, high dissolution ability, and good ionic conductivity. However, they are more cost-effective, easy to prepare, non-toxic, and often biodegradable, making them highly appealing for green chemistry applications. Despite these benefits, challenges remain, such as difficulty in separating them from reaction products and the complexity of designing suitable solvent systems.<sup>28</sup>

Pressurized natural deep eutectic solvents (P-NADESs) have also been investigated for the extraction of several compounds. High-pressure extraction has gained ground in compound recovery because it requires less energy consumption, as these methods are faster and more scalable.<sup>29</sup> DESs have been widely used for the extraction of hydrophilic compounds, such as phenolic compounds and anthocyanins. More recently, hydrophobic DESs have also been developed for the extraction of non-polar compounds, including carotenoids.<sup>27</sup>

In addition to these alternatives, acidified solvents play a crucial role in the extraction of pH-sensitive hydrophilic compounds, such as anthocyanins. These solvents are specifically designed to maintain a low pH, which stabilizes anthocyanins in their flavylium cation form, enhancing both their color intensity and structural integrity. Commonly used solvents like ethanol or water are acidified with organic or mineral acids to optimize extraction conditions. The acidic environment not only prevents degradation but also improves extraction efficiency, making acidified solvents particularly suitable for maximizing anthocyanin yield and preserving their functional properties.<sup>30–32</sup>

Capello *et al.* suggested replacing traditional solvents with environmentally friendly alternatives and outlined key criteria for green solvents. These include using less hazardous and more biodegradable solvents, adopting bio-based options like ethanol from renewable sources, utilizing environmentally benign supercritical fluids, and incorporating IL with low vapor pressure to minimize air emissions.<sup>33,34</sup>

Besides choosing the adequate extraction process, it may also be relevant to combine it with pretreatments and/or purification or concentration steps, to achieve the highest yield of the desired compound. In Fig. 1, the main pretreatments, extraction techniques, and purification/concentration steps cited in this work are presented.

#### 4.1 Pretreatments

Pretreatment processes are a critical first step in extracting valuable compounds from agro-industrial wastes, as they enhance the accessibility and recovery of target bioactive compounds. These treatments aim to break down complex plant cell structures, reduce particle size, and increase surface area, thereby improving solvent penetration and mass transfer during extraction. Depending on the nature of the raw material and the desired compounds, various physical, chemical, and biological pretreatment methods can be employed.<sup>35,36</sup>

The two most common and largely employed pretreatment methods are drying and grinding. Drying is a key method for preserving food, extending shelf life, and reducing storage costs. Conventional hot air drying is widely used for this purpose, but it may reduce nutritional quality and alter the appearance, depending on the duration of exposure to heat. Freeze-drying is another option that better preserves nutrients and appearance, but it is more time-consuming and expensive, with results varying depending on the drying conditions and food properties.<sup>32,37</sup> In the grinding process, the particle size is reduced. As a result, it increases the diffusivity of the bioactive compounds and promotes the rupture of the cell walls.<sup>38</sup>

Depolymerizing compounds like lignin is a crucial step before the extraction of lignocellulosic materials, as it enhances solvent accessibility to hydrolysable lignin within the rigid structure of lignocellulosic materials. This process significantly enhances the efficiency of phenolic compound extraction by breaking down complex lignin structures, thereby improving solvent penetration and ultimately increasing extraction yields.<sup>35</sup>



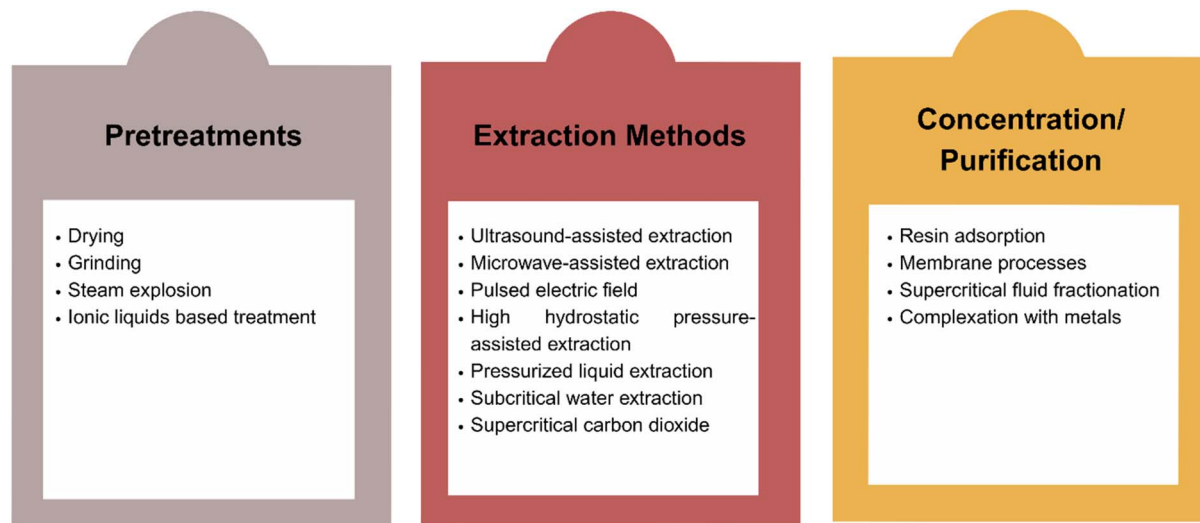


Fig. 1 Pretreatments, extraction techniques, and concentration/purification steps used to extract desired compounds from agro-industrial wastes.

Steam explosion is a process that can be used for this purpose, characterized by low capital cost and moderate energy requirements. It involves exposing lignocellulosic materials to high pressure (5–50 atm) and elevated temperatures (160–260 °C) for a short period, typically ranging from a few seconds to several min. At the end of this treatment, the pressure is rapidly released, causing a sudden “explosion” that disrupts the cell wall structure and breaks the physical bonds within the lignocellulose matrix, thereby altering the internal structure of the biomass. During this process, acetic acid is released from the naturally occurring acetyl groups in hemicellulose, triggering autohydrolysis reactions. These reactions lead to the partial cleavage of hemicellulose acetyl side chains and promote the breakdown of glycosidic bonds in hemicellulose, hemicellulose–lignin linkages, and  $\beta$ -O-4 inter-unit bonds in lignin. As a result, hemicellulose becomes partially hydrolyzed, increasing its solubility in water, while lignin is depolymerized, enhancing its solubility in alkaline or organic solvents.<sup>35,39</sup>

IL also holds significant potential as pretreatment solvents for lignin extraction. Their effectiveness lies in their ability to dissolve lignocellulosic components, including lignin and cellulose, thereby facilitating the disruption of the plant cell wall matrix. ILs can selectively solubilize lignin, reduce the crystallinity of cellulose, and aid in the separation of these structural polymers. This capacity enhances the accessibility of target compounds and improves the efficiency of downstream extraction processes. As a result, IL-based pretreatments have been successfully employed for lignin recovery from various biomass sources. This approach can serve as either a pretreatment step or an extraction method.<sup>36</sup>

#### 4.2 Green extraction methods

The target compounds from agro-industrial waste can be extracted using various conventional methods, including maceration, heat reflux, and Soxhlet extraction. These

techniques remain widely used due to their simplicity and effectiveness. However, they also present significant drawbacks, such as long extraction times and high solvent consumption, which can limit their efficiency and sustainability.<sup>35</sup> For this reason, these technologies have been replaced by innovative green technologies, such as UAE, microwave-assisted extraction (MAE), pulsed electric field (PEF), pressurized liquid extraction (PLE), subcritical water extraction (SWE), and supercritical CO<sub>2</sub> (SC-CO<sub>2</sub>). These innovative methods can be applied alone or in combination to enhance the efficiency of subsequent extraction processes.

UAE is one of the most widely studied extraction methods. This technique utilizes sonication, which involves the creation and collapse of cavitation bubbles. When ultrasound waves, typically in the frequency range of 20–100 kHz, are applied, they induce the implosion of these bubbles within the cells. This process disrupts the cell membranes, allowing solvents to penetrate more effectively and improving mass transfer. As a result, intracellular bioactive compounds are released more efficiently.<sup>40</sup>

The efficiency of the UAE depends on several factors, including equipment parameters (power, amplitude, and pulse use), ambient conditions (temperature and pressure), solvent properties, food matrix properties, and the use of pretreatments. All these factors can impact the extraction efficiency and the quality of the extracted product. In summary, the cumulative findings highlight the significant potential of ultrasound technologies in enhancing yield, reducing processing time, and promoting greater sustainability in industrial applications. UAE and its derivatives, including pulsed UAE, are regarded as innovative methods for extracting natural compounds, making them ideal for the production of functional foods and health products due to their adaptability.<sup>41,42</sup>

PEF is an emerging non-thermal and environmentally friendly technology that shows great promise for applications in



the food industry. It involves the application of a short-duration electric field (ranging from nanoseconds to milliseconds) to a food matrix, resulting in the electroporation of cell membranes. The size and number of these pores increase in proportion to the duration of the treatment and the intensity of the electric field. In this context, PEF can induce either reversible or irreversible electroporation; the latter leads to the leakage of intracellular compounds, facilitating their extraction. The efficiency of PEF depends on several factors, including process parameters (electric field strength, number of pulses, and pulse width), the properties of the food material, and the intended processing objectives.<sup>43</sup>

MAE, which utilizes electromagnetic waves with frequencies ranging from 300 MHz to 300 GHz, is another rapid, energy-efficient, green, and emerging technology for extracting bioactive compounds.<sup>44</sup> MAE operates through two main mechanisms: dielectric heating and ionic conduction. Dielectric heating occurs when dipole moments in molecules rapidly align and realign with the changing electromagnetic field, generating heat through molecular friction. In contrast, ionic conduction involves the movement of ions in solution, which generates heat as they align with the electromagnetic field. Both mechanisms enhance heat generation within the solvent, improving the efficiency of the extraction process.<sup>41</sup>

Microwaves provide energy that heats the intracellular water, increases the pressure within plant cells, and causes cell walls to rupture. This process releases the intracellular compounds, drastically accelerating the diffusion step and significantly reducing the extraction time. As a result, MAE helps minimize the degradation of antioxidants and other bioactive compounds during the extraction process.<sup>45</sup>

High-pressure extraction has become increasingly popular due to its use of GRAS (Generally Recognized as Safe) solvents and lower energy consumption, as the process is faster and more easily scalable.<sup>29</sup> High hydrostatic pressure-assisted extraction (HHPE) is an advanced, non-thermal technique used to enhance the recovery of bioactive compounds from plant and food matrices. Operated at pressures ranging from 100 to 900 MPa, HHPE disrupts cellular structures by generating a significant pressure gradient between the interior and exterior of cell membranes. This results in increased cell permeability, facilitating the efficient release of intracellular compounds. Unlike conventional heat-based extraction methods, HHPE can be performed at ambient temperatures, minimizing the degradation of heat-sensitive constituents and preserving the integrity and functionality of target molecules. As a result, HHPE is gaining prominence as an effective and gentle alternative for extracting high-value compounds in the food, pharmaceutical, and nutraceutical industries.<sup>46</sup>

PLE is an effective and promising method for extracting a wide range of compounds, particularly polar ones, using solvents like ethanol and water. By applying elevated temperatures and pressures above 4 MPa, PLE maintains the solvent in a subcritical liquid state, even at temperatures exceeding the solvent's boiling point. This condition enhances solvent diffusivity and solute solubility, improving extraction efficiency. Additionally, PLE can be combined with UAE in a process

named ultrasound-assisted pressurized liquid extraction (UAPLE), which further increases extraction efficiency by facilitating better solvent penetration into the matrix and enhancing the dissolution of target compounds.<sup>41,47–49</sup>

SWE is a type of PLE, where water serves as the solvent. PLE utilizes water in its liquid state at elevated temperatures, between its boiling point (100 °C) and critical point (374 °C), maintained under high pressure (2–20 MPa). Under these conditions, water exhibits reduced viscosity and surface tension, along with increased vapor pressure and diffusivity, all of which enhance mass transfer rates.<sup>29</sup>

SC-CO<sub>2</sub> extraction is recognized as an effective and sustainable method for extracting desired compounds. Its key advantages include the cleanliness and safety of CO<sub>2</sub>, which is non-toxic, non-flammable, and does not produce environmentally hazardous waste, in addition to being granted GRAS status. The method is also efficient, as supercritical CO<sub>2</sub> penetrates solid matrices more rapidly than conventional liquid solvents. Moreover, it offers high selectivity, enabling the targeted extraction of a wide range of analytes while preserving the integrity of the bulk matrix.<sup>50,51</sup>

In addition to these methods, alkaline hydrolysis offers another valuable approach, particularly for extracting cellulose and lignin from lignocellulosic biomass. This method, which typically involves using a sodium hydroxide solution, is one of the most effective and cost-efficient techniques for lignin extraction, yielding high amounts of lignin. The process works through the saponification of intermolecular ester bonds that crosslink lignin and hemicellulose, leading to the depolymerization of these compounds and resulting in lignin and hemicellulose as the primary products.<sup>52</sup>

### 4.3 Concentration and purification steps

The concentration and isolation process vary depending on the pretreatment and extraction methods applied, as well as the solvent used during extraction.<sup>53</sup> Moreover, one of the main challenges in extracting phenolic compounds is the co-extraction of other substances, such as sugars and polysaccharides, which reduce extract purity and may impact bioactivity.<sup>54</sup> To address this, purification techniques like resin adsorption, membrane processes, acid precipitation, and supercritical fluid fractionation (SFF) have been employed.

Resin adsorption involves retaining phenolic compounds on micro-beads, which are later eluted using a solvent. Typical resins used for this purpose include Amberlite XAD4, XAD16, XAD7, Sepabeads SP207, and Diaion HP20.<sup>54</sup> The purification process can be further enhanced by sequentially washing the phenols loaded on the resin with several solvents at a low pH value.<sup>55</sup>

Membrane processes, on the other hand, utilize ultrafiltration or nanofiltration membranes with varying structures and pore sizes to separate phenolic compounds while also concentrating the sample.<sup>54</sup> This method has been effective in separating sugars from phenolic compounds, utilizing nanofiltration membranes, such as NF90 (commercial), and cellulose acetate membranes.<sup>56</sup> Another study proposed



nanofiltration with composite membranes having a nominal molecular weight cut-off between 150 and 300 Da. Concentrating extracts obtained from agro-industrial byproducts using a membrane separation process is interesting because, in addition to intensifying the properties of bioactive compounds, it increases their final commercial value.<sup>47</sup>

SFF is a promising strategy to concentrate extracts following supercritical fluid extraction (SFE). Among the approaches employed in SFF are the use of adsorbents and sequential pressure reduction, achieved by manipulating the density of the supercritical solvent and column fractionation. These methods allow for selective separation of extracted components, thereby enhancing concentration and purity. On a laboratory scale, the integration of SFF into SFE systems is feasible due to the low consumption of CO<sub>2</sub>, enabling the design of compact equipment that leverages CO<sub>2</sub> selectivity. Furthermore, this configuration facilitates the use of a single collection vessel for the extract, simplifying the process by eliminating the need for precise temperature and pressure control during fractionation.<sup>57</sup>

Complexation with metals has shown promise in enhancing the antioxidant properties of phenolic compounds by protecting against free radicals. Additionally, metal interactions can result in the formation of co-pigments, which produce vibrant colors such as green, blue, and violet, depending on the metal used. Research has demonstrated that ferric ions, for example, can form dark purple complexes with cyanidin-3-glucoside, contributing to the characteristic color of vegetables. Studies also indicate that anthocyanins preferentially bind to iron and magnesium ions, while flavones tend to bind with calcium, highlighting the diverse potential for metal-based co-pigmentation in future applications.<sup>58</sup>

## 5. Green extraction applications for Brazilian agro-industrial waste valorisation

Brazil is one of the world's leading agricultural producers, playing a crucial role in the global supply of several food products, including sugar cane, cashew apples, oranges, and many others.<sup>16</sup> The application of green extraction technologies to these diverse waste streams has shown remarkable potential for value creation, with extraction yields and bioactive compound recovery varying significantly based on feedstock characteristics and the extraction methodology employed. In Fig. 2, several Brazilian products of global significance are presented. These are the products for which Brazil is the largest global exporter. These commodities not only drive the country's economy but also generate a substantial volume of agro-industrial waste throughout their production and processing chains. Waste represents both an environmental challenge and an opportunity for high-value applications.<sup>1,17</sup> Rich in proteins, fibers, bioactive compounds, and natural antioxidants, these byproducts are increasingly being explored for their potential use in food, pharmaceuticals, cosmetics, and bioenergy. By understanding the composition and classification of these

### Brazilian main agro-industrial products

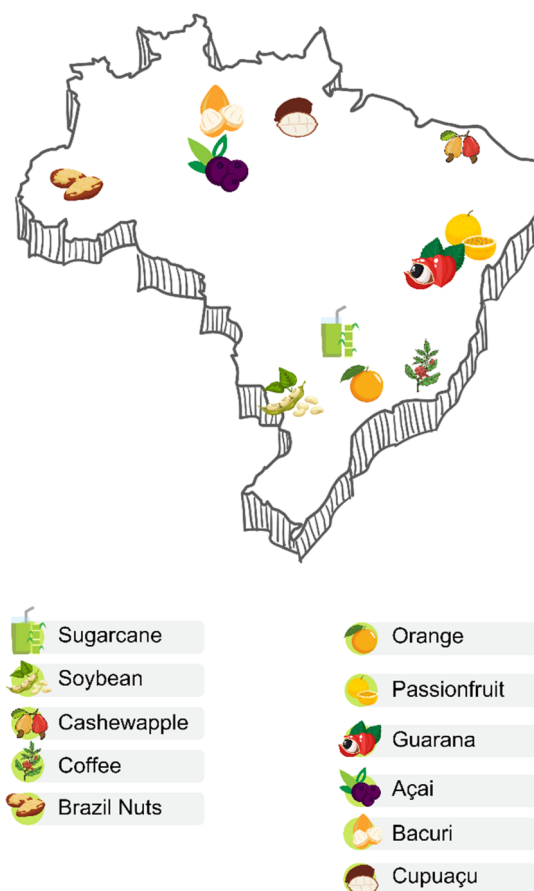


Fig. 2 Major Brazilian agro-industrial products and their key producing region.<sup>16,45,58,62–65</sup>

residues, Brazil can enhance waste valorization strategies, reduce environmental impact, and strengthen its position as a leader in circular economy innovations. Table 1 presents the composition of the main byproducts of some important Brazilian agro-industrial products. As shown in Table 1, the agro-industrial waste exhibits a diverse composition. It includes several bioactive compounds, oils, fats, fibers, alkaloids like caffeine, proteins, and other compounds. Thus, different strategies may be suitable for valorizing them through extraction. To analyze these strategies, the extractable compounds were divided into five groups: bioactive compounds, oils and fats, fibers and lignocellulosic components, alkaloids, caffeine, stimulants, and other compounds. A summary of the strategies used to extract each compound is presented in Table 2.

### 5.1. Bioactive compounds

The bioactive compounds comprise a diverse group of compounds, including phenolic compounds such as flavonoids, anthocyanins, and carotenoids. These compounds offer several health benefits and can therefore be utilized in the formulation of food additives and nutraceuticals. Additionally,



Table 1 Composition of Brazilian main agro-industrial wastes

Product	Byproduct	Definition	Waste composition
Sugarcane	Straws	Sugarcane leaves and tops	Cellulose (33–45%), hemicellulose (18–30%), lignin (17–41%), ashes (2–12%), and extractives (5–17%) (dry basis) <sup>74</sup>
	Bagasse	Primary industrial fibrous residue obtained after pressing sugarcane stalks to extract the juice	Cellulose (39–45%), hemicellulose (23–27%), lignin (19–32%), ashes (1–3%), and extractives (5–7%) (dry basis) <sup>74</sup>
	Vinasse	Fermented liquid medium without ethanol content, from the ethanol distillation stage <sup>74</sup>	—
	Molasse	Product from the sugar industry obtained from sugar crystallization	Significant amounts of vitamin B6 and minerals, including calcium, magnesium, iron, and manganese <sup>74</sup>
Orange	Peel	Orange peel obtained from the juice industry	Soluble sugars, starches, fibers, cellulose, hemicellulose, lignin, pectin, organic acids (citric acid, malic acid, malonic acid, and oxalic acid), vitamins (vitamin C), and phenolic compounds (gallic acid, <i>p</i> -coumaric acid, ferulic acid, caffeic acid, <i>trans</i> -cinnamic acid, flavone, and thymol) <sup>75</sup>
Soybean	Okara	Byproduct generated during the soybean processing of tofu and soy milk	Fiber (30–50%), protein (25–35%), lipid (6–10%), isoflavones, and saponins (dry basis). <sup>76–78</sup>
	Meal	Byproduct of the soybean seed industry	Protein (33–56%), fiber (4.3–7.2%), fat (0.5–3.2%), and carbohydrates (4.7–7%) (wet basis) <sup>79</sup>
Cashew apple	Cashew shell	Agro-waste produced from cashew nut processing factories, rich in cashew nut shell liquid	Anacardic acids, cardanols, and cardols <sup>80</sup>
	Bagasse	A byproduct of this apple after juice extraction	Carbohydrates (27–77%), proteins (1.8–22.7%), lipids (mainly unsaturated) (0.38–12.1%), and ashes (minerals such as zinc, iron, manganese, copper, boron, magnesium, calcium, potassium, sodium, and phosphorus) (dry bagasse). <sup>80,81</sup>
Coffee	Spent grounds	Comes from brewed coffee, meaning it is the waste left after extracting coffee from roasted beans in industrial coffee production <sup>82,83</sup>	Cellulose (12.4%), hemicellulose (39.1%), lignin (23.9%), fat (2.29%), protein (13–17%), micronutrients, and alkaloids like caffeine <sup>84</sup>
	Pulp and husk	Waste of wet (pulp) and dry (husk) processing coffee cherries	Carbohydrates (44–58%), proteins (9–12%), fibers (18–21%), fat (2–2.5%), Caffeine (1.3%), tannins (1.8–8.5%), polyphenols (1%), pectin (12.4%), phenolic compounds, and alkaloids (mainly caffeine) (dry basis). <sup>82,83</sup>
Brazilian nuts	—	—	—
Passion fruit	Rinds	Waste from juice production from the pulp	Dietary fibers (as pectin), phenolic compounds (vitexin, isovitexin, apigenin, isoorientin, cyanidin-3-O-glycoside, and quercetin-3-O-glycoside), and carboxylic acids (edulic acid) <sup>85</sup>
	Seed waste	—	Rich in lipids and essential fatty acids, such as linoleic, oleic, and palmitic acids <sup>86</sup>
Guarana	Wasted seed	—	Phenolic compounds (mainly catechin, epicatechin, and epicatechin gallate, and caffeine (2–6%) (dry basis)) <sup>65</sup>
Açaí	Seed	—	Cellulose and hemicellulose (63–86%), proteins (5–6%), lipids (2–3%), fatty acids, phospholipids, sterols, sphingolipids and terpenes), minerals (2–6%) [10], and polyphenols (mainly procatechuic acid, different dimers of procyanidin, and epicatechin) (dry basis). <sup>45,87</sup>
	Pulp residue/ Bagasse	—	Anthocyanins (cyanidin and pelargonin), flavonoids (homo-orientin, orientin, catechin, epicatechin, and <i>p</i> -coumaric), prothocyanidins, and some other useful products (quercetin, vanillic, ferulic, and gallic acid) <sup>45</sup>
Bacuri	Rinds	—	Water (78.80%), resins (1.40%), crude protein (0.5%), pectin (5%), reducing sugars (2.7%), cellulose (3.90%), total titratable acidity (4.1%), and minerals (0.6%), approximately (wet basis) <sup>58</sup>
Cupuaçu	Seed	—	Protein (3.8–20.6%), lipid (21–24.4%), ash (1.4–5.2%), sugars (13.6–26.4%), fibers (22.2%), phenolic compounds (mainly gallic acid, procatechuic acid, coumaric acid, epicatechin, epigallocatechin gallate, quercetin, and glycosylated quercetin), and flavonoids (isoscutelearein derivatives, hypolaetin derivatives, catechin, kaempferol, and clovamide) (dry basis) <sup>88</sup>



**Table 2** Main technologies applied to extract valuable compounds from Brazilian agro-industrial waste and proposed applications to these extracts<sup>a</sup>

Product	Compounds extracted	Technologies applied	Applications
Sugarcane	Bioactive compounds: phenolic compounds	DES, alkaline hydrolysis, and autohydrolysis <sup>28</sup> SC-CO <sub>2</sub> (ref. 51)	Cosmetic application <sup>54</sup>
	Cellulose and hemicellulose		Film preparation <sup>53,89</sup>
Orange	Phytosterols	UAE + DES, <sup>27</sup> UAE, <sup>66–70,90</sup> PEF + UAE, <sup>91</sup> MAE + DES, <sup>92</sup> and PEF + solid-liquid extraction with green solvents <sup>93</sup>	Food additive and food packaging <sup>69</sup>
	Bioactive compounds: phenolic compounds, flavonoids, limonene, and carotenoids		Functional ingredient <sup>94</sup>
	Pectin		
Soybean	Cellulose and dietary fiber	UAE <sup>69</sup> and PEF <sup>94</sup>	Fragrance and flavor industry <sup>95</sup> Flavoring and preservative agent <sup>96</sup> Drug and byproduct development in food and pharmaceutical industries <sup>77</sup>
	Essential oil	SCE <sup>95</sup>	
	Citric acid	UAE <sup>96</sup>	
Cashew apple	Bioactive compounds: phenolic compounds and carotenoids	MAE, <sup>79</sup> DES <sup>97</sup>	Natural supplement in food processing <sup>98</sup> Encapsulation material <sup>99</sup>
	Fiber	UAE + alkali treatment <sup>98</sup>	
Coffee	Polysaccharide/protein	MAE <sup>99</sup>	Commercial pectin <sup>105</sup> Vegetable oil, substitute for butter in bakery formulations <sup>109</sup> Ingredient in energy drinks and in analgesic formulations, biocide for wood, and repellent <sup>110</sup>
	Bioactive compounds: phenolic compounds	UAE <sup>71</sup> and MAE <sup>44</sup>	
	Pectin	Diluted acid, acid/alkali, and alkaline hydrogen peroxide treatments followed by precipitation <sup>100</sup>	
	Oil	UAE <sup>101</sup> UAE <sup>101</sup>	
	Caffein	HHPE, <sup>46</sup> UAE, <sup>46,72,73</sup> liquid and supercritical CO <sub>2</sub> , <sup>102</sup> SC-CO <sub>2</sub> , <sup>50</sup> MAE, <sup>72</sup> DES, <sup>103</sup> and PEF + MAE <sup>104</sup> MAE <sup>105</sup> SC-CO <sub>2</sub> (ref. 106–108)	
<b>Brazilian nuts</b>			
Passion fruit	Bioactive compounds: phenolic compounds	DME and UAE, <sup>111</sup> UAPLE – PLE, <sup>48</sup> and UAPLE + nanofiltration <sup>47</sup>	Dermo cosmetic formulations <sup>111</sup> Extend the shelf life of food <sup>86</sup>
	Pectin	SEW + P-NaDES <sup>29</sup>	
	Oil	Pressurized ethanol and UAE <sup>112</sup>	
Guarana	Catechins and methylxanthines	High shear mixing <sup>113</sup> and UAE <sup>65</sup>	Alkaloids
Açaí	Alkaloids	MAE <sup>45</sup> and PLE <sup>49</sup>	
	Phenolic compounds	Centrifugation + filtration <sup>114</sup>	
Bacuri	Inulin	Multi-step extraction process <sup>115</sup>	Pigments <sup>58</sup>
	Lignocellulosic byproducts and cellulose		
Cupuaçu	Bioactive compounds: phenolic and carotenoids	Cold maceration, hot maceration, neutral sonication, and acid sonication, <sup>63</sup> and UAE + complexation with metals <sup>58</sup>	
	Bioactive compounds	Percolation process <sup>62</sup>	

<sup>a</sup> DES: deep eutectic solvent; DME: dynamic maceration; HHPE: high hydrostatic pressure-assisted extraction; IL – ionic liquid; MAE: microwave-assisted extraction; P-NaDES: pressurized natural deep eutectic solvent; PEF: pulsed electric field; PLE: pressurized liquid extraction; SEW: subcritical water extraction; SC-CO<sub>2</sub>: supercritical carbon dioxide; SCE: supercritical extraction; UAE: ultrasound-assisted extraction; UAPLE: Ultrasound-Assisted Pressurized Liquid Extraction.



Table 3 Optimal conditions for the extraction of bioactive compounds using emerging technologies reported in the literature<sup>a</sup>

Food waste	Technology applied	Optimal conditions	Main results	Reference
Glycine max (soybean) seed waste	MAE (no control)	MAE at 120 W for 0.16 min with a solvent/dry matter ratio of 60/1	The highest contents achieved were 13.09 mg GAE per g for phenolics and 7.39 mg CE per g for flavonoids	79
Cashew apple	UAE and control (60 min at room temperature)	UAE at 55% amplitude for 12 min and a solid to liquid ratio of 45 g mL <sup>-1</sup> (RMN)	UAE treatment increased total phenolics by 115% and total tannin content by 132%	
Coffee pulp	UAE and MAE	MAE applying a radiation time of 70 min, a 700 W power, and a solvent to sample ratio of 100 : 5 mL g <sup>-1</sup>	MAE increased the content of recovered compounds by 48% (phenolics), 70% (chlorogenic acids), and 42% (caffeine) compared to UAE. Approximately 47 mg of phenolic compounds, 36 mg of flavonoid, 8 mg of chlorogenic acid, and 6 mg of caffeine could be recovered from 1 g of the material	71
Coffee pulp	CE and UAE	UAE at 396 W, 5.5 min and a solid:liquid ratio of 1 : 10 w v <sup>-1</sup> . The optimal temperature was 96 °C for caffeine and 75 °C for phenolic extraction	UAE led to extraction yields 85% higher than that of CE. The highest concentration of caffeine in the extractant was found to be 15.6 ± 0.3 g kg <sup>-1</sup> pulp db, and the concentration of total polyphenols reached 12.4 ± 0.2 g kg <sup>-1</sup>	73
Spent coffee grounds	HHP, UAE, and CE	UAE at 60% amplitude for 15 min	UAE led to greatest content of chlorogenic acid (85.0 ± 0.6 mg kg <sup>-1</sup> FW) and total phenolics (9.51 ± 0.06 mg GAE/100 g FW)	46
Coffee bean	MAE (no control)	MAE at 450 W, 75 °C, solid/solvent content of 2.5 g coffee husk/50 mL citric acid solution, and extraction time of 15 min	The Coffee Husk Pectin (CHP) yield of 40.2% was obtained (weight of dried CHP/weight of coffee husk)	105
Coffee waste	PEF + MAE and CE (no PEF and MAE)	Yellow pulp as the extraction substrate and the combination of MAE + PEF, and a processing time of 15 min	Yellow pulp: 1443 mg GAE/100 g material (PEF + MAE), 1167 mg GAE/100 g (MAE), 799 mg GAE/100 g (CE); red pulp: 1235 mg GAE/100 g (MAE + PEF); 1075 mg GAE/100 g (MAE); 704 mg GAE/100 g (CE)	104
Coffee husk waste	NADES	The best composition of NADES solvent was a choline chloride and proline ratio of 1 : 1. Optimum extraction conditions were used with the addition of 50% water, extraction time of 30 min at 80 °C, and a ratio of sample weight to solvent volume of 1 : 10	A extraction yield of 10.07 mg GAE per g and a polyphenol concentration of 671.4 mg L <sup>-1</sup>	103
Coffee roasting by-products	SC-CO <sub>2</sub> (300 bar, 2 hours) and CE (hexane, 80 °C for 6 hours)	—	No impressive differences in yield between the conventional (3.0 ± 0.1%) and the best SC-CO <sub>2</sub> process conditions (3.1% ± 0.1, 60 °C/300 bar) was found, but the extraction time was drastically reduced from 6 to 2 h	50
Orange by-products	CE (agitation at 80 °C) and UAE using water as solvent	UAE at 790 W L <sup>-1</sup> and 25 °C for 3 min	UAE increased the yields of total phenolics, total flavonoids, and antioxidant activity by 29%, 39%, and 197%, respectively, compared to conventional extraction	66
Orange peel	UAE using water as solvent and stirred-tank extraction	UAE at 80% amplitude for 15 min, using a duty cycle of 2/2	Yields of phenolics obtained using both technologies were similar, but UAE reduced the extraction time and temperature	67



Table 3 (Contd.)

Food waste	Technology applied	Optimal conditions	Main results	Reference
Orange peel	HR and UAE	—	HR and ethyl acetate as solvent improved yields of phenolics; UAE and ethyl acetate led to extracts with the highest content of polymethoxyflavones, and showed the most potent antiglycation, antibacterial, and acetylcholinesterase inhibition activities	68
Orange peel	UAE and olive oil as a solvent	UAE at 45 °C for 35 min and a liquid-to-solid ratio of 15 mL g <sup>-1</sup>	The content of carotenoids achieved a value of 1.85 mg/100 g dry weight	70
Orange by-products	UAE and CE (agitation at 35 °C)	UAE at 90% amplitude for 35 min, pulse 100% and 45/55 ethanol/water (v/v) ratio	UAE led to phenolic recovery up to 60% higher than that of the conventional method. The maximum value achieved for total phenolics was 30.42 mg GAE per g db	90
Orange peel	Combination of PEF (optimized) and UAE (fixed value)	Electric field strength of 5 kV cm <sup>-1</sup> and a total specific energy input of 40 kJ kg <sup>-1</sup>	The application of PEF increased the concentration of limonene by 33% and the extraction of linalool by more than 114% when ethanol was used as solvent	93
Orange by-products	PEF (optimized) + UAE (fixed value)	Electric field of 1.4 kV cm <sup>-1</sup> and 30 pulses of 110 μs	Optimized PEF treatment increased narirutin by 29.4% and hesperidin by 38.9% in the extract. It also boosted antioxidant activity by up to 56% and resulted in a recovery of 40.8 mg g <sup>-1</sup> dry weight of phenolic compounds	91
Passion fruit waste	DME and UAE	UAE at 45 °C for 15 min	The results of antioxidant activity and yield obtained by UAE were superior to those of DME, with the yield being 24% greater	111
Passion fruit rinds	PLE and UAPLE	UAPLE (60 °C, 10 MPa and 360 W cm <sup>-2</sup> ) with a solvent flow rate of 10 g min <sup>-1</sup> and a process time of 68.54 min	UAPLE increased TPC by 100% compared to PLE at the same temperature (60 °C). The economic analysis showed that the lowest manufacturing cost (US\$245.46/g TPC) was at a flow rate of 10 g min <sup>-1</sup> , making it the best option for producing phenolic-rich extracts at the laboratory scale	48
Açai pulp	CE, and pre-treatment of MAE and pressurized MAE	—	When pretreatment was applied, pressurized MAE enabled the achievement of higher concentrations of polyphenols during the extraction. After 30 min of extraction, the TPC content increased 65%. After PMAE (1.5 bar, 300 W) was applied, the maximum content of total phenolics was 424 mg GAE/100 g for extraction from slurry, 8.33 g GAE/100 g for seeds, and 8.83 g GAE/100 db for pulp fractions	45
Bacuri	Maceration and UAE (neutral and acid contions)	Maceration at 50 °C with 100% ethanol for mesocarp extracts, and acidic sonication in 100% ethanol for epicarp extracts	Total phenolics: mesocarp:14.60 ± 0.42% w/w and epicarp: 7.22 ± 0.16% w/w db	63



Table 3 (Contd.)

Food waste	Technology applied	Optimal conditions	Main results	Reference
Glycine max (soybean) seed waste	MAE (no control)	MAE at 120 W for 0.16 min with a solvent/dry matter ratio of 60/1	The highest contents achieved were 13.09 mg GAE per g for phenolics and 7.39 mg CE per g for flavonoids	79
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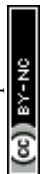


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Orange by-products	PEF (optimized) + UAE (fixed value)	Electric field of 1.4 kV cm <sup>-1</sup> and 30 pulses of 110 μs	Optimized PEF treatment increased narirutin by 29.4% and hesperidin by 38.9% in the extract. It also boosted antioxidant activity by up to 56% and resulted in a recovery of 40.8 mg g <sup>-1</sup> dry weight of phenolic compounds	91
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Bacuri	Maceration and UAE (neutral and acid contions)	Maceration at 50 °C with 100% ethanol for mesocarp extracts, and acidic sonication in 100% ethanol for epicarp extracts	Total phenolics: mesocarp: 14.60 ± 0.42% w/w and epicarp: 7.22 ± 0.16% w/w db	63

<sup>a</sup> CE: conventional extraction; UAE: ultrasound-assisted extraction; NADES: natural deep eutectic solvent; SC-CO<sub>2</sub>: supercritical CO<sub>2</sub> extraction; HR: heat reflux; HHPE: high hydrostatic pressure; PLE: Pressurized Liquid Extraction; UAPLE: Ultrasound-Assisted Pressurized Liquid Extraction; DME: dynamic maceration.



anthocyanins and carotenoids exhibit colorimetric properties that can be used as natural dyes in the food industry.<sup>59–61</sup>

The multifaceted properties of these compounds make them very versatile. Carvalho *et al.* (2021) reviewed several applications of sugarcane phenolic extract, highlighting its use in the cosmetic industry due to its various biological activities, including antioxidant, antimicrobial, anti-inflammatory, and tyrosinase, collagenase, and elastase inhibitors.<sup>54</sup> The authors also stated that the main challenge in extracting phenolic compounds from sugarcane is the co-extraction of other substances, such as sugars and polysaccharides, which can reduce the purity of the extract and impact its potential bioactivities. Therefore, they highlighted the importance of purification steps. Resin adsorption and membrane processes were suggested for this purpose. The study reviewed several studies and suggested the resins Amberlite XAD4, XAD16, XAD7, Sepabeads SP207, and Diaion HP20; and the membranes NF270, NF90, CA400-22 (nanofiltration membranes), and Desal GK (ultrafiltration membranes).<sup>54</sup> Given the importance of these compounds, several processes and innovative technologies have been employed to extract bioactive compounds from industrial waste. A summary of the main results for the extraction of bioactive compounds and their optimized conditions is shown in Table 3. It is essential to note that there are differences in units of measurement across studies, variations in conventional methods used for comparison, and variations in the composition of agri-food byproducts (*e.g.*, differing types of waste and agricultural origins). These factors can make it challenging to compare results across studies in a straightforward manner.

**5.1.1. Extraction of bioactive compounds using ultrasound-assisted extraction (EAU).** The use of water as a solvent in UAE has been recently investigated for extracting phenolic compounds from orange peel. Dalmau *et al.* (2020) demonstrated that the yields of total phenolics, total flavonoids, and antioxidant activity obtained through UAE (790 W L<sup>-1</sup>, 25 °C, 3 min) using water as solvent were 29%, 39%, and 197% higher, respectively, than those obtained through conventional extraction (25 °C for 10 min). It is important to note that the best potency and temperature found were the maximum levels of the parameters studied. Neohesperidin, hesperidin, coumaric acid, and sinapic acid were identified as the main compounds present in the extract. In contrast, naringin and ferulic acid were extracted to a lesser extent, likely due to their more lipophilic nature.<sup>66</sup> Similarly, Belsi *et al.* (2024) proposed an aqueous extraction process to extract phenolics from orange peel using the following process parameters: 150 W sonication, 80% amplitude, and a 2/2 duty cycle for 15 min. They achieved comparable phenolic yields to those obtained using traditional methods (a stirred-tank process for 60 min at 55 °C). The advantages of the ultrasonic process were the less time and energy (room temperature) required.<sup>67</sup>

The extraction of flavonoids from orange peel waste using UAE was also compared to Heat Reflux (HR) extraction, a conventional solvent extraction method, with both tested with various solvents, including 95% ethanol, 70% aqueous ethanol, methanol, and ethyl acetate. The HR extract, using ethyl acetate, yielded the highest total phenolic content, total flavonoid

content, and flavonoid glycosides, and demonstrated the strongest antioxidant and  $\alpha$ -glucosidase inhibition activities. In contrast, the UAE extract with ethyl acetate showed the highest concentration of polymethoxyflavones and exhibited superior antiglycation, antibacterial, and acetylcholinesterase inhibitory effects.<sup>68</sup>

A multi-step approach was also applied to propose the valorization of orange peel waste. In the first step of the process, phenolic compounds were extracted, with the application of UAE significantly reducing the extraction time. In the sequence, cellulose nanofibers were also extracted from this byproduct, further enhancing its valorization. The sequential approach yielded high concentrations of compounds without requiring pretreatments.<sup>69</sup>

Besides its wide use for extracting phenolic compounds from orange peel, UAE was also employed to extract carotenoids from this agro-industrial waste, using olive oil as the solvent. The process was optimized under the following conditions: an extraction time of 35 min, an extraction temperature of 42 °C, and a liquid-to-solid ratio of 15 mL g<sup>-1</sup>. Extraction time, followed by the liquid-to-solid ratio, had the most significant impact on extraction yields. After that, the obtained extract was also encapsulated to enhance its stability.<sup>70</sup>

Patra *et al.* (2021) further optimized UAE from cashew apple bagasse using both response surface methodology (RSM) and artificial neural networks (ANNs). The extraction process was performed under various combinations of treatment time (5–15 min), ultrasound amplitude (30–60%), and bagasse-to-solvent ratio (30–50 g mL<sup>-1</sup>) to maximize the content of phenolics, tannins, and  $\beta$ -carotene. RSM identified the optimal conditions as 12 min of treatment, 55% amplitude, and a 45 g mL<sup>-1</sup> bagasse-to-solvent ratio, whereas ANN predicted optimal parameters of 15 min, 60% amplitude, and a 50 g mL<sup>-1</sup> ratio.<sup>71</sup>

In coffee waste valorization, UAE (60% amplitude for 15 min, which were the maximal levels of the variables studied) proved to be an efficient method for the recovery of phenolic compounds from spent coffee grounds, outperforming classical solvent extraction (at 50 °C for 30 min) and high hydrostatic pressure-assisted extraction (HHPE) by producing higher levels of chlorogenic and caffeic acids.<sup>46</sup> UAE was also applied to coffee pulp, with optimal conditions at 35 min of ultrasonic time, 60 °C, and 250 W, although yields were lower than those obtained by MAE.<sup>72</sup>

Further UAE on coffee pulp using water as the extractant achieved high caffeine and polyphenol recovery with an energy requirement up to 3.4 times lower than that of conventional solid-liquid extraction, highlighting UAE as a sustainable method. The results suggested that the optimal conditions for extracting polyphenols were 5.5 min, 75 °C, and 370 W, which were the average values of the time and temperature studied. It might have occurred because polyphenols have low stability at high temperatures. Moreover, the main polyphenols extracted were protocatechuic acid, gallic acid, and 5-chlorogenic acid, which, together with caffeine, have a wide potential for incorporation in the food, pharmaceutical, and cosmetic industries.<sup>73</sup>



A comparison between ultrasonic and non-ultrasonic methods has also been performed, with four methods being evaluated: cold maceration, hot maceration, neutral sonication, and acid sonication for extracting phenolic compounds from bacuri fruit waste. The results highlighted that maceration at 50 °C with 100% ethanol as the solvent was the best extraction condition for mesocarp extracts, and acidic sonication in 100% ethanol for epicarp extracts, resulting in a high content of phenolic compounds and antioxidant activity.<sup>63</sup>

UAE was also employed to extract phenolic compounds from the bacuri rind. Post-extraction complexation with metal salts (iron and aluminum) enhanced pigment stability, indicating potential for cosmetic applications. Temperature and pH were found to have a significant influence on pigment properties. Overall, the samples exhibited satisfactory technological characteristics, supporting their potential use in various applications, including as pigments in cosmetic formulations, as well as in the form of flour or food powders.<sup>58</sup>

Yellow passion fruit seed waste was subjected to dynamic maceration (DME) and UAE to optimize the recovery of bioactive compounds using hydroethanolic solvents under varying time and temperature conditions. Extracts obtained using UAE yielded superior antioxidant capacity and extraction efficiency compared to those obtained using DME, with optimal conditions being 1 min at 45 °C for DME and 15 min at 45 °C for UAE. Mass spectrometry characterization revealed the presence of fatty acids, phenolic acids, flavonoids, and stilbenes. Both extracts demonstrated photoprotective properties, with sun protection factor (SPF) values of 18.22 (DME) and 11.68 (UAE), supporting their potential application in dermo-cosmetic formulations. Moreover, the UAE extract exhibited anti-proliferative effects on the SIHA cell line and stimulated cell division in the HuH7.5 cell line. These results underscore the effectiveness of UAE as a green extraction method and highlight the pharmacological potential of valorizing passion fruit seed waste.<sup>111</sup>

A combination of UAE and other extraction technologies has also been proposed. Ultrasound can also be combined with pressurized solvents in a process known as UAPLE. The results demonstrated that this technique significantly enhanced the recovery of phenolic compounds from passion fruit rinds. Under conditions of 60 °C, 10 MPa, 360 W cm<sup>-2</sup> ultrasonic intensity, and a solvent flow rate of 10 g min<sup>-1</sup>, 100% of the total phenolic content (TPC) was recovered within 68.54 min, achieving the lowest estimated manufacturing cost (US\$ 245.46/g TPC). Compared to pressurized liquid extraction (PLE) alone, UAPLE increased TPC recovery by 100% at the same temperature and by 55% compared to PLE performed at 75 °C. These findings indicate that, in high-pressure extraction processes, factors such as pressure, temperature, ultrasonic intensity, and solvent flow rate significantly influence the yield of phenolic compounds.<sup>48</sup>

High extraction pressures can enhance solvent access to target compounds by reducing the formation of bubbles in the matrix. However, excessive pressure may cause bed compaction, decrease porosity, reduce solvent-solute contact, and lower interstitial velocity, ultimately decreasing extraction efficiency.

Therefore, pressure should be applied only to the extent necessary to maintain the solvent in a liquid state and facilitate its penetration, avoiding unnecessary increases that could hinder the extraction process.<sup>48</sup> In another study, UAPLE was followed by nanofiltration using polyamide membranes to concentrate phenolics from passion fruit rinds, with membranes with a molecular weight cut-off below 300 Da achieving higher retention of phenolic compounds.<sup>47</sup>

The combination of PEF and UAE was also studied. In a previous study, Razola-Díaz *et al.* (2021)<sup>43</sup> optimized the UAE of phenolics from orange byproducts and recommended the following operating conditions: 45/55 ethanol/water (v/v), 35 min, amplitude 90% (110 W), and pulse 100%. Then, Razola-Díaz *et al.* (2024)<sup>116</sup> utilized these optimized UAE conditions to propose a sequential treatment of PEF and UAE for extracting phenolics from orange peel. The study optimized the PEF treatment and suggested that the optimized conditions were an electric field of 1.4 kV cm<sup>-1</sup> and 30 pulses of 110 μs. These conditions increased the antioxidant activity of the extract up to 56% and the content of narirutin and hesperidin by 29.4% and 38.9%, respectively.<sup>91</sup>

**5.1.2. Extraction of bioactive compounds using pulsed electric field (PEF).** Besides being proposed to extract phenolic compounds from orange byproducts, PEF was also applied in a sequential treatment to extract limonene from the same food matrix using solid-liquid extraction with green solvents. The results of this research showed that the application of PEF improved the extraction of limonene by 33%. Additionally, Gas Chromatography coupled with Mass Spectrometry (GC-MS) analysis revealed no evidence of degradation of individual compounds due to PEF application.<sup>93</sup>

**5.1.3. Extraction of bioactive compounds using microwave assisted extraction (MAE).** MAE is an extraction technology that enhances extraction rates and yields through dielectric heating, and it has been applied to several types of food waste. The application of MAE and UAE was compared for extracting bioactive compounds from coffee pulp, with MAE yielding higher amounts of compounds. The optimal MAE conditions were a radiation time of 70 min, a power of 700 W, and a 50% (v/v) ethanol solvent-to-sample ratio of 100 : 5 (mL g<sup>-1</sup>).<sup>72</sup>

A green valorization protocol for soybean meals, a byproduct of the soybean seed industry, was proposed using MAE to recover polyphenols. The highest contents of phenolic compounds and flavonoids were obtained at 120 W for 0.16 min with a solvent-to-dry matter ratio of 60 : 1, representing the lowest power and ratio levels studied.<sup>79</sup>

Similarly, cashew apple bagasse was subjected to MAE. Optimal extraction conditions were identified as 560 W for 110 seconds with a solvent-to-solid ratio of 30 : 1. Under these conditions, the extracts were rich in phenolics, proteins, tannins, and vitamin C. In contrast to soybean meal, the best results for cashew apple bagasse were achieved at a high power (tested 280–280 W) and at the highest solvent ratio levels tested. In coffee residues, MAE was applied to extract pectin from husks at 450 W and 75 °C for 15 min using a citric acid solution, resulting in the production of antibacterial and anti-inflammatory compounds. All the optimized parameters



correspond to intermediate levels of the parameters studied.<sup>105</sup> Finally, from açai pulp and seed residues, MAE increased the polyphenol content from 370 to 614 mg GAE/100 g DM in 30 min. The study applied ambient-pressure MAE and pressurized MAE as pretreatments for the maceration process and observed that these treatments increased the polyphenol content and antioxidant activity of the extract, mainly when combined with an ethanol/water mixture (1 : 1 v/v) at a temperature of 60 °C. The advantage of the pressurized process was the application of higher temperatures without solvent evaporation, resulting in the highest phenolic content.<sup>45</sup>

MAE was also combined with PEF to propose a green method for recovering phenolic compounds from various coffee agro-industrial wastes, demonstrating the feasibility of the method for all the studied wastes. The study also suggested the use of water as a solvent and highlighted the potential of this green extraction method to extract antioxidant compounds.<sup>104</sup>

DES-based methods were also studied. Isoflavone recovery from soybean and okara, a soy-based byproduct, extracts using different DESs was also studied, with DESs based on choline chloride (ChCl) with carboxylic acids, sugars, and glycerol being investigated. The results of the work showed that isoflavone recovery benefited from ChCl:acetic acid DES with 30% water.<sup>97</sup> Using coffee husk waste as a raw material, a DES composed of ChCl and proline in a 1 : 1 molar ratio yielded the highest polyphenol contents. The results indicated that this ratio was the most effective for polyphenol extraction. Optimal extraction conditions included the addition of 50% water, an extraction time of 30 min, and a sample-to-solvent ratio of 1 : 10 (w/v). Temperature had a positive influence on the extraction efficiency, with 80 °C identified as the recommended temperature for the process.<sup>103</sup>

Pectin is widely used in the food industry for various applications, including gelling, emulsion stabilization, and thickening, and finds relevance in the cosmetic, pharmaceutical, and biomedical sectors. Traditionally, industrial pectin extraction involves prolonged heating and the use of toxic acids, raising concerns about environmental impact and product safety.<sup>29</sup> As a result, alternative, more sustainable extraction methods are being explored. DES has been applied for pectin extraction from orange peel under MAE conditions. The optimal parameters were identified as a ChCl: formic acid concentration of 8% (v/v), an extraction time of 15 min, and a microwave power of 360 W. This MAE-DES approach reduced the extraction time by 75% compared to conventional maceration. Additionally, the pectin obtained through this method exhibited improved water-holding capacity and higher viscosity, indicating enhanced functional properties.<sup>92</sup>

**5.1.4. Other extraction technologies to obtain bioactive extracts.** A comparison between SWE and P-NADES was conducted to explore the valorization of passion fruit waste through pectin extraction from both the rind and its residual biomass. Extraction using the DES system, composed of citric acid, glucose, and water (Ca:Glu:Wa) at 120 °C, proved more effective than both SWE and conventional extraction methods, resulting in higher pectin yields from the passion fruit rind and its residual biomass. Additionally, the results showed that the

extraction solvent influenced the degree of esterification: pectins with a high degree of esterification were obtained using P-NADES (Ca:Glu:Wa). In contrast, lower degrees of esterification were observed with extractions performed using pure or acidified water.<sup>29</sup>

Hydrophobic DES has been utilized for the extraction of nonpolar compounds, including carotenoids. In orange peel, after screening several potential DESs, an extraction process for carotenoids was recommended using menthol: eucalyptol under UAE for 20 min at 120 W, providing an extract with high carotenoid content and stability over 60 days of storage.<sup>27</sup>

Liquid and supercritical CO<sub>2</sub> are other technologies applied to extract desired bioactive compounds. Liquid and supercritical CO<sub>2</sub> with 5% ethanol yielded similar results and extracted bioactive compounds from spent coffee grounds in just 1 hour, matching the yields of conventional extractions that took 5 hours. The extracts obtained contained linoleic, palmitic, oleic, and stearic acids, as well as furans and phenols. The authors also highlighted that the phenolic acids (chlorogenic, caffeic, ferulic, and 3,4-dihydroxybenzoic acids) presented are well-known for their antioxidant and antimicrobial properties.<sup>102</sup>

SC-CO<sub>2</sub> extraction has also been applied in the valorization of coffee roasting byproducts. Compared to traditional extraction methods, SC-CO<sub>2</sub> significantly reduced the extraction time from 6 to 2 hours and eliminated the need for hydrocarbon or chlorinated solvents. Furthermore, laboratory-scale conditions can be easily scaled up to industrial levels. Industrial SC-CO<sub>2</sub> systems also allow for the recovery and reuse of clean CO<sub>2</sub>, ensuring a waste-free, safe, and cost-effective extraction process.<sup>50</sup>

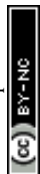
PLE intensifies solute diffusion at elevated temperatures and pressures. Açai byproduct valorization was proposed using a PLE process. The optimal condition was 115 °C with 75 wt% ethanol, resulting in phenolic-rich, antioxidant extracts. The process was also compared to sonication and proved more effective in terms of the evaluated technical responses. Additionally, the process demonstrated technical and economic feasibility.<sup>49</sup>

Finally, cupuaçu byproducts underwent a percolation process with 70% ethanol to extract epicatechin and glycosylated quercetin. The processes involved solid-liquid extraction and the use of green solvents. The results demonstrated that this byproduct still contains significant levels of bioactive compounds with antioxidant activity and notable nutritional potential.<sup>62</sup>

## 5.2. Phytosterols and oils

The recovery of phytosterols and oils from agri-food residues has been extensively investigated using a range of green extraction technologies, mainly SC-CO<sub>2</sub>. These approaches not only offer environmentally friendly alternatives to conventional extraction techniques but also enable the selective recovery of bioactive compounds with potential applications in the food, cosmetics, and pharmaceutical industries.

SC-CO<sub>2</sub> extraction has demonstrated high efficiency in extracting oils and phytochemicals from various agri-food



residues. In the case of sugarcane bagasse, a SC-CO<sub>2</sub> process was optimized for extracting phytosterols. The recommended extraction conditions were 40 °C, a CO<sub>2</sub> flow rate of 6 mL min<sup>-1</sup>, a pressure of 400 bar, and no co-solvent addition. The authors also emphasized that the extract, comprising phytosterols, policosanols, and unsaturated fatty acids, is a potential source of bioactive compounds beneficial to cardiovascular health. Phytosterols are structurally and functionally similar to cholesterol, but they contribute to the reduction of cholesterol in the blood when consumed.<sup>51</sup> Similarly, essential oils from orange peel were successfully extracted using SC-CO<sub>2</sub> at 74.85 °C for 317.51 min, with a solvent-to-sample ratio of 4. The resulting extract exhibited notable aromatic and chemical properties, making it suitable for application in the flavor and fragrance sector, as well as in food preservation.<sup>95</sup>

Okara was subjected to SC-CO<sub>2</sub> to obtain okara oil and defatted okara powder. Although extraction was not optimized, the study confirmed the viability of using SFE for the generation of this dual product. The use of ethanol as a co-solvent significantly increased the content of phenolics and isoflavones in oil and improved antioxidant capacity. Furthermore, SC-CO<sub>2</sub> improved the water absorption and oil-binding capacity of the defatted okara powder, although its swelling capacity was reduced; meanwhile, the oil contained essential fatty acids such as linoleic, oleic, palmitic, linolenic, and stearic acids.<sup>117</sup>

The use of ethanol and other co-solvents, including isopropanol and ethyl lactate, was also explored for oil extraction from spent coffee grounds. The results showed that co-solvent addition reduced extraction time and improved oil yield.<sup>106</sup> Another study employed a two-step extraction process using supercritical SC-CO<sub>2</sub> and ethanol to extract and fractionate oil from spent coffee grounds, with a focus on total phenolic content. Extractions were performed at 333 K and 40 MPa using various solvent systems, including pure SC-CO<sub>2</sub>, ethanol, and a supercritical CO<sub>2</sub>-ethanol mixture (90 : 10 w/w). The addition of ethanol as a co-solvent was shown to enhance the range of extracted compounds by altering the solvation properties of the supercritical fluid. In line with green chemistry principles, the use of ethanol and/or water as co-solvents is recommended to improve extraction efficiency while maintaining environmental sustainability.<sup>107</sup>

The extraction kinetics of spent coffee grounds using absolute ethanol (ET0) and hydrous ethanol (ET6) were evaluated across a temperature range of 60–150 °C under both atmospheric and pressurized conditions. Solvent hydration was found to negatively affect oil recovery, while higher temperatures led to increased extraction yields, reaching values comparable to those obtained with hexane. PLE consistently resulted in faster extraction rates regardless of solvent hydration, with temperature effects on spent coffee grounds oil yields being more pronounced under pressurized conditions. These results highlight the efficiency of ethanol, particularly under high-pressure conditions, for extracting oil and demonstrate the importance of both temperature and pressure in optimizing extraction performance.<sup>108</sup>

Pressurized ethanol and UAE were also employed to extract oil from passion fruit pulp. A study evaluated the content of

phenolic compounds and antioxidant activity of the obtained oil and found that UAE led to the highest overall yield. However, similar to the highest content of phenolics and antioxidant activity, these were achieved with pressurized ethanol. The study also compared various solvents for the UAE and found that the use of polar solvents resulted in higher oil yields.<sup>112</sup>

The oils obtained from agri-food residues have shown promising applications. While previous studies focused on extracting the compounds, a few have also studied the application of the extracts. For example, coffee oil from spent coffee grounds was used to replace butter in cookies at concentrations of 10–30%. At 20% substitution, the cookies exhibited softer textures and higher antioxidant activity, without compromising sensory acceptability.<sup>109</sup>

### 5.3. Fibers and lignocellulosic components

Several fibers and lignocellulosic materials found in plant biomass are primarily composed of cellulose, hemicellulose, and lignin, which form the structural framework of cell walls. These biopolymers offer significant potential for sustainable material development due to their abundance, renewability, and biodegradability. In recent years, the extraction of these components from agricultural residues has become a key focus in efforts to reduce environmental impact and promote circular resource use, with several innovative technologies being used for this purpose.<sup>69,118,119</sup>

Cellulose, a major component of lignocellulosic biomass, has been extracted from various residues using both conventional and green extraction routes. UAE has been effectively applied in multiple approaches for cellulose recovery from agro-industrial residues. In a cascade biorefinery system, UAE was used to extract cellulose from orange peel after the recovery of polyphenols, showcasing the potential for multi-compound extraction from a single biomass source.<sup>69</sup>

Similarly, ultrasound was combined with alkali treatment to extract insoluble dietary fiber, particularly cellulose, from okara waste. The alternating sequence of ultrasound and alkali treatments (*e.g.*, ultrasonic-alkali-ultrasonic and alkali-ultrasonic-alkali) significantly enhanced cellulose content (>83%) and induced greater porosity in soybean fibers. Scanning electron microscopy revealed that ultrasound disrupted the okara matrix, amplifying the collapse effect and improving extractability.<sup>98</sup> Together, these findings illustrate the versatility of ultrasound-based methods in breaking down complex plant matrices and increasing fiber accessibility, whether integrated into biorefinery schemes or combined with chemical treatments.

Another study compared and evaluated green extraction routes – autohydrolysis, alkaline hydrolysis, and DES – for extracting cellulose from sugarcane byproducts. DES composed of ChCl and urea (1 : 2 molar ratio) proved to be the most efficient at removing hemicellulose, while autohydrolysis at 170 °C provided the highest cellulose yield.<sup>28</sup>

Nanocellulose (NC) was extracted from açai bagasse using different hydrolysis and oxidative methods, including mixed acid hydrolysis, TEMPO-mediated oxidation, and ammonium



persulfate oxidation. Mixed acid hydrolysis yielded nanocellulose with the highest crystallinity and thermal stability, whereas oxidation methods improved water dispersion and ionic charge, suitable for various applications.<sup>120</sup> A multi-step extraction and hydrolysis process was also used on açai core fibers to obtain cellulose nanocrystals. The study defined optimal parameters: a low temperature (45 °C), small fiber size (35–125 µm), and a long reaction time (120 min), emphasizing the potential of this biomass for cellulose nanocrystal and lignin production.<sup>115</sup>

Soluble dietary fibers (SDF) were extracted from orange peel using PEF. The optimized parameters were 45 °C, 6.0 kV cm<sup>-1</sup> electric field intensity, 30 pulses, and 20 min. PEF increased SDF's functional properties (*e.g.*, solubility, emulsifying capacity, and binding affinity to metals) by causing electroporation, cell wall collapse, and release of compounds such as pectin.<sup>94</sup> Besides that, a MAE technique was used on okara to obtain a polysaccharide-protein extract, later used as an encapsulating matrix for micronutrients. This method highlights the versatility of MAE in multi-component recovery, while also identifying potential applications of the obtained extract.<sup>99</sup>

Inulin, a valuable prebiotic polysaccharide, was extracted from açai seed flour. The process involved aqueous extraction, centrifugation, and sequential filtration using alcohol, acetone, and water. Optimization through response surface modeling found that 80 °C, a 4 : 1 water-to-flour ratio, and 20 min yielded the highest inulin concentrations.<sup>114</sup> Another study using açai core fibers *via* a multi-stage process also produced lignin as a byproduct along with cellulose nanocrystals and carbohydrates, confirming the potential of integrated extraction approaches for high-value co-products.<sup>115</sup>

Lignin is the second most abundant renewable carbon source on Earth, surpassed only by cellulose. While biorefineries primarily focus on converting polysaccharides from lignocellulosic biomass into biofuels, lignin is often undervalued and mainly used for energy generation. However, due to its high functionality and aromatic structure, lignin holds significant potential for the production of aromatic and fine chemicals. Lignin is insoluble in water and stable in nature, and acts as a 'glue' binding cellulose to hemicellulose. Also, it is a complex polymer made of three types of phenolic units: *p*-hydroxyphenyl, guaiacyl, and syringyl.<sup>100,118</sup>

Finally, lignin was extracted from cashew apple bagasse (CAB) through three different treatments: diluted acid (DA), acid/alkali (AA), and alkaline hydrogen peroxide (AHP). After these treatments, lignin was then recovered by acid precipitation at pH 2. The AA and AHP treatments resulted in high removal levels (98.0% and 96.9%, respectively), producing lignin with high thermal stability.<sup>100</sup>

Lignin was also extracted from sugarcane bagasse using eight different protic ionic liquids (PIL). The results demonstrated that the choice of PIL significantly influenced the properties of the extracted lignins, enabling the production of materials with distinct characteristics suitable for value-added applications. Notably, lignins extracted using acetate and lactate anions combined with monoethanolammonium cations exhibited similar structural profiles. In contrast, lignins

obtained using ethylenediammonium cations paired with acetate or lactate anions showed relatively higher molecular weights compared to the other samples. Elemental analysis further revealed the presence of nitrogen in all lignin samples, indicating that amination reactions may have occurred during the extraction process.<sup>118</sup>

#### 5.4. Alkaloids, caffeine, and stimulants

Coffee and guarana processing wastes are valuable sources of alkaloids, especially caffeine, which can comprise up to 1% of the husk dry weight. Caffeine recovered from these wastes has several applications: as a stimulant in energy drinks, in analgesic pharmaceuticals due to its role in pain relief, as a biocide for wood protection, and in repellent formulations.<sup>110</sup>

Several innovative green extraction techniques have been explored for isolating caffeine and other alkaloids from agricultural and food waste. Among these, UAE has gained prominence for its efficiency. UAE was used to extract caffeine from guarana seeds, demonstrating a superior yield and faster kinetics compared to conventional extraction (CE) at room temperature. Even at elevated temperatures (50–70 °C), UAE maintained kinetic advantages, although the maximum yield became comparable to that of CE. Moreover, life cycle assessment revealed that UAE significantly reduced extraction time, energy use, and environmental impact.<sup>65</sup>

Similarly, UAE was applied to coffee wastes, such as coffee pulp and spent grounds, often in combination with aqueous ethanol, aqueous propylene glycol, or DES. In some studies, UAE was combined with MAE or PEF treatment to enhance the recovery of phenolic compounds and caffeine. Extraction conditions varied widely, with solid-to-liquid ratios ranging from 1 : 10 to 1 : 100 and extraction times from 3 to 35 min.<sup>83</sup> UAE at room temperature, using water as the solvent, also proved highly effective for extracting both caffeine and chlorogenic acid from spent coffee grounds, outperforming simple vortexing and even supramolecular solvents in terms of yield.<sup>121</sup>

Another promising method involves the use of choline-based IL. Caffeine was successfully extracted from coffee husks using aqueous solutions of IL with different carboxylate anions. Among these, choline hexanoate ([Ch][Hex]) stood out, achieving a maximum yield under optimized conditions: 95 °C, 0.021 g mL<sup>-1</sup> solid-liquid ratio, 2.34 M concentration. Notably, the [Ch][Hex] solution could be reused at least twice without significant loss of efficiency.<sup>110</sup>

Lastly, high shear mixing (HSM) was applied to guarana processing waste, including peels and depleted seeds. The integration of pectinase enzymes further improved the recovery of valuable polar compounds, demonstrating the synergy of mechanical and enzymatic treatments.<sup>113</sup>

#### 5.5. Other compounds

Beyond bioactive compounds, polysaccharides, and alkaloids, UAE has also been effectively utilized to recover other valuable compounds, such as proteins and organic acids like citric acid and ascorbic acid, from fruit-based agro-industrial residues.



Table 4 Comparative analysis of green extraction technologies<sup>a</sup>

Technology	Principle	Key advantages	Key disadvantages	Relative yield	Energy use	Scalability	Economic viability (CAPEX/OPEX)
UAE	Acoustic cavitation disrupts cell walls, enhancing mass transfer	Reduced extraction time & temperature; suitable for heat-sensitive compounds	Attenuation in large volumes; potential for free radical formation	Medium-high	Low-medium	Moderate	Medium/low
MAE	Dielectric heating of polar molecules causes cell rupture	Very fast extraction; reduced solvent consumption	Shallow penetration depth; risk of localized overheating; not for non-polar solvents	Medium-high	High	Moderate	Medium/high
PLE/SWE	High temperature and pressure increase solvent diffusivity and solute solubility	Fast and efficient; uses green solvents (water and ethanol); can tune selectivity with temperature	High energy consumption; potential for thermal degradation of sensitive compounds	High	High	Good	Medium/medium-high
SC-CO <sub>2</sub>	Supercritical fluid has liquid-like density and gas-like viscosity, enabling efficient penetration	High selectivity; solvent-free extracts; non-toxic; CO <sub>2</sub> is recyclable	High capital cost (high pressure); not efficient for highly polar compounds without the use of co-solvents (non-polar)	Medium-high (for non-polar)	Medium	Excellent	High/low
DES/ILs	“Designer solvents” with unique properties to dissolve specific components like cellulose or lignin	High selectivity, low volatility, and thermally stable	High viscosity; recovery and recycling of solvent is complex and costly; potential toxicity concerns	High	Low-medium	Low	High/high

<sup>a</sup> Relative ratings (low, medium, and high) are relative. Actual values are highly dependent on the specific feedstock, target compound, and the optimization of the process.

Protein extraction from Brazilian agri-food waste has been successfully explored using ultrasound-assisted extraction (UAE). For instance, UAE was applied to extract proteins, antioxidants, and ascorbic acid from cashew apple bagasse. The study employed both Response Surface Methodology (RSM) and Artificial Neural Network (ANN) modeling to determine the optimal extraction conditions.<sup>101</sup> For protein recovery, the RSM model identified the optimal conditions as 15 min of treatment time, 40% ultrasound amplitude, and a 37 g mL<sup>-1</sup> bagasse-to-solvent ratio. The ANN model provided slightly different conditions: 15 min of treatment, 37% ultrasound amplitude, and a 42 g mL<sup>-1</sup> bagasse-to-solvent ratio. For ascorbic acid recovery, the RSM model suggested optimal conditions of 11.40 min of sonication, 60% ultrasound amplitude, and a CAB-to-solvent ratio of 50 g mL<sup>-1</sup>. The ANN model proposed slightly adjusted conditions: 9.73 min of treatment time with the same amplitude and ratio. These approaches demonstrate how UAE, in conjunction with computational optimization techniques, can substantially increase the yield of functional compounds from food waste streams.<sup>101</sup>

Some organic acids were also extracted from agro-industrial waste. In one study, UAE was employed to extract citric acid from orange and lime peels. An experimental design incorporating five levels of ultrasonic power (50–500 W), extraction time (2–45 min), and ethanol proportion (0–100%) was used in combination with RSM to optimize the process. UPLC-PDA analysis revealed that orange peels had a higher citric acid content than lime peels. For both citrus residues, the best yields were achieved by sonication at low power, using either a low ethanol content or pure water as the solvent. The optimal times varied depending on the solvent and biomass: 5.8 min for low-ethanol solutions and 35.5 min when using only water.<sup>96</sup>

## 6. Scalability and economic viability

The sustainable potential of green extraction technologies demands rigorous scrutiny of their real-world application, including quantitative sustainability metrics and techno-economic analysis. Green extraction technologies demand thorough scrutiny of their real-world application, including quantitative sustainability metrics and techno-economic analysis. LCA studies have demonstrated that green extraction technologies can reduce environmental impacts by 35–60% compared to conventional methods. This reduction is primarily achieved through decreased solvent consumption and energy requirements, which are significant contributors to the environmental footprint of extraction processes.<sup>122,123</sup> The initial investment for technologies like SC-CO<sub>2</sub> extraction can be high, ranging from US\$ 2–5 million for industrial-scale equipment. This is due to the advanced nature of the equipment and the high pressures required for operation.<sup>124</sup>

Recent advancements in automation and the availability of critical machinery have contributed to a reduction in capital costs, making SC-CO<sub>2</sub> extraction more accessible to industries.<sup>125</sup> The cost of SC-CO<sub>2</sub> extraction equipment is often compared to traditional methods, highlighting the need for

high-pressure vessels and specialized maintenance, which contribute to the overall CAPEX.<sup>126</sup>

Their transition from the laboratory to an industrial scale and economic feasibility, and a thorough assessment of their environmental impact are non-negotiable for the effective deployment of their technology. To provide a clearer understanding of the trade-offs between different methods, Table 4 summarizes key performance indicators.

UAE is highly efficient on a laboratory scale, but its scalability presents challenges. Large volumes attenuate the energy-efficient transmission. Thus, large volume systems require multiple transducers and complex reactor designs, which increase capital costs. However, its ability to operate at lower temperatures and reduce processing times can lead to significant operational energy savings. Economically, it is most viable for high-value extracts where a moderate increase in capital expenditure (CAPEX) is justified by improved yield and quality.

The penetration depth of microwaves into the raw material, particularly in dense or aqueous slurries, is a significant limitation to the scalability of this technology. The use of smaller processing batches or advanced industrial microwave systems with multiple magnetrons increases complexity and cost. While it offers rapid heating and reduced solvent use, the high energy consumption during operation can be a drawback for low-value products. Supercritical CO<sub>2</sub> (SC-CO<sub>2</sub>): this technology is one of the most successfully scaled, particularly in the food and pharmaceutical industries. However, its significant barrier is the high capital investment required for high-pressure equipment. The operational costs (OPEX) can be relatively low, especially since CO<sub>2</sub> is inexpensive, non-toxic, and easily recycled within the system. Its economic viability is strongly tied to producing high-purity, solvent-free extracts for premium markets, such as decaffeinated coffee or hop extracts for the brewing industry.

Pressurized Liquid Extraction (PLE) and Subcritical Water Extraction (SWE): these methods have moderate scalability. The primary challenge is safely managing high pressures and temperatures on an industrial scale. The equipment cost is lower than for SC-CO<sub>2</sub>, but energy consumption for heating and pressurizing the solvent can be substantial. Their viability depends on the ability to process large volumes of biomass efficiently and to recycle the solvent (often water or ethanol) to minimize costs.

## 7. Integrated biorefinery approaches for Brazilian agri-food waste

The integration of multiple unit operations within a biorefinery framework may constitute a more robust strategy than individual processes for the valorisation of these food wastes. A systemic approach to biorefineries may be suggested for maximizing resource recovery efficiency and advancing the circular bioeconomy by closing material and carbon loops. The obtained products can be various value-added products, bioproducts, and biofuels.

From an economic perspective, biorefineries are compelling due to their ability to co-produce a diversified portfolio of high-value products from a single feedstock. Some studies have proposed a biorefinery design for managing important agro-industrial wastes. While some studies combine the extraction of value-added compounds and energy recovery, others focus on sequential extraction procedures to extract different compounds.<sup>127</sup> In Fig. 3, a conceptual scheme for a biorefinery system is proposed.

The concept of integrated biorefineries is supported by advancements in process intensification, which involves the use of hybrid and sequential green technologies to improve yield, conversion rates, and energy efficiency. These technologies enable the extraction of high-value compounds from biomass in an environmentally friendly manner.<sup>128</sup>

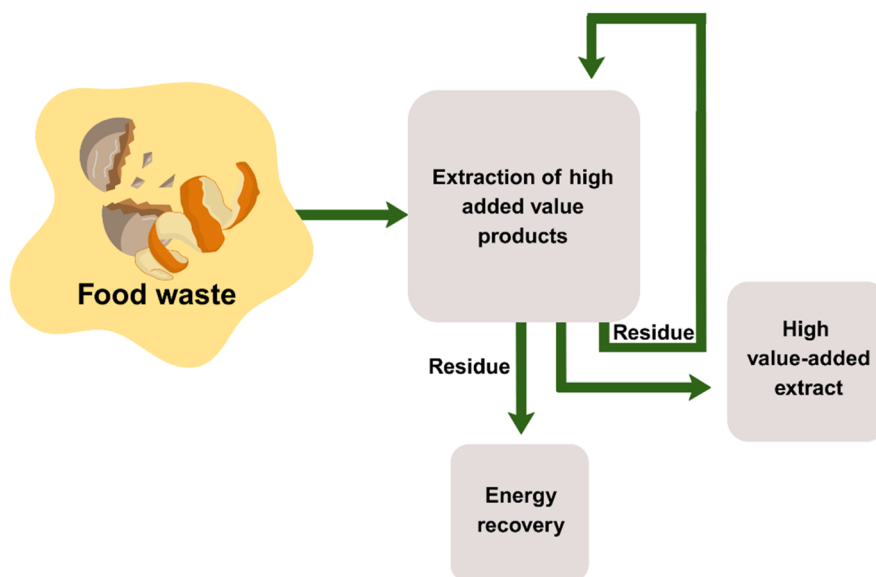


Fig. 3 Proposed scheme for agro-industrial waste valorisation through the extraction of desired compounds in a biorefinery system.



A conceptual framework for a Brazilian agri-food waste biorefinery includes (1) primary extraction of high-value bioactive compounds using green solvents, (2) secondary recovery of structural components (cellulose and lignin) through advanced pretreatments, (3) tertiary processing of remaining biomass for bioenergy production, and (4) integrated waste-to-energy systems for process heat and electricity generation. Pilot-scale studies have shown that integrated biorefineries can significantly enhance economic viability. For instance, the sequential extraction of high-value biomolecules from brown macroalgae has demonstrated promising economic potential, with net present values ranging from € 20 to € 506 k for pilot-scale systems.<sup>129</sup>

Techno-economic modelling indicates that such integrated systems can achieve positive net present values (NPV) from US\$15–45 million over 20 year project lifespans, depending on feedstock availability and product market prices.<sup>130,131</sup> Key success factors for biorefinery implementation include optimizing the feedstock supply chain (addressing Brazil's logistical challenges), developing a regulatory framework for waste-derived products, and establishing strategic partnerships among agricultural producers, technology providers, and end users. Recent investments by Brazilian companies, such as Raizen and Suzano, in biorefinery technologies demonstrate a growing industrial interest in integrated waste valorization approaches. Raizen is projecting a lignin production capacity of 1.5 million tons (wet basis) in at least 7 second-generation ethanol (E2G) plants. The potential production is up to 3.4 million tons (damp basis) across 20 E2G plants.<sup>132</sup> Suzano launched a \$70 million corporate venture capital fund to invest in startups developing sustainable, bio-based solutions, including those focused on biomass and lignin.<sup>133</sup>

An integrated biorefinery design for the complete valorisation of sugarcane straw (SCS) was proposed. The extracted compounds included glucan, hemicellulose, and lignin constituents. The results demonstrated that the approach has successfully converted up to 85% (w/w) of these components into various bioproducts.<sup>134</sup> A spent coffee grounds-based biorefinery is also an emerging approach that promotes a circular bioeconomy. Banu *et al.* (2020) summarized different methods to promote the valorisation of coffee grounds. Several technologies have been employed, and other products have been developed, with emerging technologies standing out as highly utilized alternatives. From the products, polyhydroxyalkanoates and phenolic compounds were the primary products obtained, with flavonoids and caffeine being obtained as secondary products.<sup>135</sup>

Orange waste was the residue most studied in a biorefinery approach. Espinosa *et al.* (2022) proposed a cascade biorefinery approach to extract bioactive compounds from orange peel. The authors proposed a sequential treatment to extract first polyphenols and then nanocellulose.<sup>69</sup> Another biorefinery approach to extract desired compounds from orange peel was also studied. In this work, the proposal was to obtain pectin through enzymatic hydrolysis and then extract phenolic compounds using a combination of extraction and several filtration techniques.<sup>136</sup> Durán-Aranguren *et al.* (2025) proposed a sequence to valorise orange

residues. The proposal produced essential oils, phenolic compounds, pectin, and fermentable sugars, which were later converted into ethanol, xylitol, and single-cell protein.<sup>137</sup>

In the context of biorefinery products, economic considerations play a crucial role in determining the viability of the process. The most economical approach for managing residues must be assessed on a case-by-case basis, as it depends on the operating conditions involved and the selling price of the final product. In this context, a comprehensive techno-economic analysis is indispensable, as the economic viability of such a system is contingent not only on revenue but also on critical parameters such as production capacity and capital expenditures (CAPEX). Empirical evidence from some studies focusing on agro-industrial feedstocks has demonstrated the economic superiority of integrated biorefinery systems. These analyses frequently report favourable financial metrics, including higher net present values (NPV), greater internal rates of return (IRR), and reduced payback times (PBT), collectively validating this approach as a viable and sustainable pathway for converting agro-industrial residues into a new generation of bio-based products and bioenergy.<sup>127</sup> However, there is still a lack of studies that compare the economic aspects of different extraction technologies.

When designing a methodology for a cost-effective integrated biorefinery, several key aspects must be carefully evaluated. These include (i) feedstock availability and associated production costs; (ii) implementation, maintenance, and operational costs of the selected technologies; (iii) processing routes and their corresponding product yields; (iv) identification and resolution of technical challenges during scale-up and operation; and (v) comprehensive assessment of the technical, economic, and environmental impacts of the proposed biorefinery system.<sup>135</sup>

## 8. Research gaps and future directions

Based on the comprehensive analysis of current literature and industrial applications, several critical research gaps and future opportunities have been identified.

### 8.1 General gaps

- Continued preference for cheaper, less sustainable disposal methods such as landfilling due to the absence of supportive regulatory and economic frameworks.
  - Need for government-driven incentives to stimulate industrial adoption of circular economy technologies.
  - The absence of standardized protocols for selecting extraction methods adapted to specific feedstocks and compounds of interest makes the comparison between the methods difficult.
  - Lack of comparative techno-economic analyses between green extraction technologies (*e.g.*, supercritical CO<sub>2</sub>, ultrasound-assisted extraction, and deep eutectic solvents).
  - Limited economic assessments and environmental impact studies of bioproducts from Brazilian agro-industrial wastes.



## 8.2 Technology development priorities

- Development of hybrid extraction systems combining multiple green technologies (*e.g.*, PEF-MAE-UAE) to achieve synergistic effects and maximize extraction efficiency.
  - Advancement of Natural Deep Eutectic Solvents (NADESS) with improved selectivity and recyclability for specific Brazilian waste streams.
    - Integration of artificial intelligence and machine learning for real-time process optimization and predictive modelling.
    - Scale-up studies for promising laboratory-scale technologies, particularly for DES and ionic liquid applications.

## 8.3 Sustainability and economic assessment

- Comprehensive life cycle assessments (LCAs) comparing different extraction pathways for major Brazilian agricultural wastes.
  - Detailed techno-economic analysis, including sensitivity analysis for key economic parameters.
  - Development of sustainability indicators specific to tropical agricultural waste valorisation.
  - Assessment of carbon footprint reduction potential through waste-to-product conversion.

## 8.4 Industrial implementation

- Pilot-scale demonstration of integrated biorefinery concepts for major Brazilian agricultural regions.
  - Development of modular, transportable extraction units to address logistical challenges in remote areas.
  - Regulatory framework development for waste-derived products in food, pharmaceutical, and cosmetic applications.
  - Supply chain optimization models for efficient collection and processing of distributed agricultural wastes.

## 8.5 Product development and market applications

- Standardization of extract quality parameters and analytical methods for Brazilian agri-food waste-derived products.
  - Development of novel applications for extracted compounds in emerging markets (*e.g.*, nutraceuticals and functional foods).
  - Investigation of synergistic effects between different bioactive compounds from mixed waste streams.
  - Consumer acceptance studies for products derived from agricultural waste valorisation.

## 8.6 Future research directions

- Greater collaboration between academia, industry, and governments to foster technology transfer and commercialization pathways.
  - Creation of policy frameworks to integrate waste valorisation with national circular economy and climate targets.
  - Promotion of regional supply chain studies to ensure continuous feedstock availability and reduce transportation-related costs and emissions.

## 9. Conclusions

The valorization of Brazilian agri-food waste through green extraction technologies represents a strategic advancement toward a more sustainable, circular, and resilient food system. As explored in this study, Brazil's diverse range of agricultural byproducts holds significant potential for transformation into high-value compounds applicable in the food, pharmaceutical, and cosmetic industries. Quantitative analysis demonstrates that green extraction technologies can achieve extraction yields ranging up to 95%, with ultrasound-assisted extraction showing 29–197% higher efficiency compared to conventional methods.

In addressing the status and challenges of food waste in Brazil, the research demonstrates that integrating circular economy principles is a promising approach for mitigating environmental impacts while fostering socioeconomic development.

Innovative extraction strategies, particularly those employing green solvents and energy-efficient processes, have proven effective in recovering bioactive compounds from agro-industrial residues, with water, DES, and IL being highlighted as green solvents. It is also important to note that the composition and yield of these extracts are highly dependent on the specific extraction method employed. Comparative analyses reveal that different techniques can yield distinct profiles in the recovered compounds, underscoring the need for method optimization to maximize efficacy and applicability.

To fully harness the potential of these technologies, investments in scalable solutions, cross-sectoral partnerships, and regulatory frameworks that promote innovation and waste reutilization are essential. Furthermore, ongoing research is needed to refine extraction parameters, ensure safety and quality compliance, and evaluate energy consumption and economic feasibility. Through the adoption of circular economy principles and the development of advanced green extraction technologies, Brazil is well-positioned to establish itself as a global leader in sustainable agri-food innovation by converting agricultural wastes into high-value resources.

Future research directions converge on integrating biorefinery approaches, targeting multi-product, zero-waste systems where cascading processes maximize the value of biomass. The development and application of new solvents, with a focus on Natural Deep Eutectic Solvents (NADESS)—biodegradable and non-toxic primary metabolite compounds—are essential, requiring research to optimize their application and recovery. Another promising front is the use of hybrid extraction methods, which combine technologies such as PEF-MAE to achieve synergistic effects. Pretreatment with PEF dramatically improves the efficiency of subsequent extraction, thereby reducing energy consumption and processing time. Also, the application of Artificial Intelligence (AI) and machine learning to *in silico* solvent screening, real-time process optimization, and predictive modeling would reduce the need for extensive empirical experimentation.



## Author contributions

Betina Louise Angioletti Decker: data curation, investigation, methodology, writing – original draft, writing – review & editing. Thatyane Vidal Fonteles: methodology, investigation, writing – review & editing. Sueli Rodrigues and Fabiano A.N. Fernandes: conceptualization, investigation, methodology, project administration, resources, writing – review & editing.

## Conflicts of interest

There are no conflicts to declare.

## Data availability

No primary research results, software, or code have been included, and no new data were generated or analyzed as part of this review.

## Nomenclature

AI	Artificial Intelligence
ANN	Artificial Neural Networks
ANVISA	Agência Nacional de Vigilância Sanitária (Brazilian Health Regulatory Agency)
CAB	Cashew Apple Bagasse
CAPEX	Capital Expenditure
DES	Deep Eutectic Solvents
DME	Dynamic Maceration
ENEC	Estratégia Nacional de Economia Circular (National Circular Economy Strategy)
EPR	Extended Producer Responsibility
GC-MS	Gas Chromatography coupled with Mass Spectroscopy
GRAS	Generally Recognized As Safe
HHPE	High Hydrostatic Pressure-Assisted Extraction
HSM	High Shear Mixing
IL	Ionic Liquids
LCA	Life Cycle Assessment
MAE	Microwave-Assisted Extraction
MAPA	Ministério da Agricultura, Pecuária e Abastecimento (Ministry of Agriculture)
NADES	Natural Deep Eutectic Solvent
NC	Nanocellulose
OPEX	Operational Expenditure
PEF	Pulsed Electric Field
PIL	Protic Ionic Liquid
PLE	Pressurized Liquid Extraction
P-	Pressurized Natural Deep Eutectic Solvent
NADES	
PNRS	Política Nacional de Resíduos Sólidos (National Solid Waste Policy)
PPP	Public-Private Partnerships
RSM	Response Surface Methodology
SC-CO <sub>2</sub>	Supercritical Carbon Dioxide
SDF	Soluble Dietary Fibers
SFE	Supercritical Fluid Extraction
SFF	Supercritical Fluid Fractionation

SMEs	Small and Medium-sized Enterprises
SWE	Subcritical Water Extraction
TPC	Total Phenolic Content
UAE	Ultrasound-Assisted Extraction
UAPLE	Ultrasound-Assisted Pressurized Liquid Extraction

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