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A Detailed Review on Valorizing Oilseed Cakes: Extraction of Bioactive Compounds from Agro-Industrial Waste

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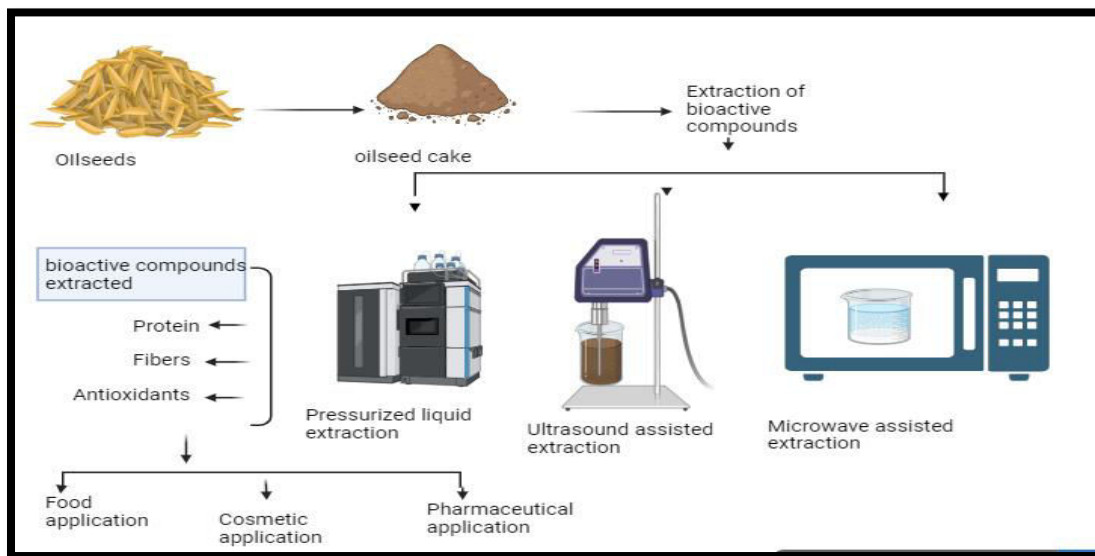
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Abstract: Oilseed cakes are plentiful agricultural by-products resulting from the production of vegetable oils. These cakes contain valuable bioactive compounds like phenolic antioxidants, proteins, peptides, plant sterols, and tocopherols. Extracting these bioactive components from oilseed cakes presents opportunities to create high-value products with applications across the food, pharmaceutical, cosmetic, and nutraceutical industries. This review comprehensively examines various extraction techniques used to recover bioactive compounds from oilseed cakes. Traditional methods such as solvent extraction, Soxhlet extraction, and maceration are discussed alongside emerging technologies like supercritical fluid extraction, ultrasound-assisted extraction, microwave-assisted extraction, and enzyme-assisted extraction. Critical evaluation is provided on factors influencing extraction efficiency, including solvent type, temperature, pressure, particle size, and pretreatment methods. Analytical techniques for characterizing and quantifying bioactive compounds, as well as purification strategies, are also highlighted. Furthermore, the review explores potential applications and economic feasibility of valorizing oilseed cakes through bioactive compound extraction. The importance of valorizing these abundant by-products for a sustainable and circular bioeconomy is emphasized.

Keywords : Agro-waste , bioactive compounds, oilseed cakes, extraction, protein , dietary fibres

Graphical Abstract



Introduction

The present worldwide challenges, Issues like excessive utilization of natural resources, patterns of consumption that cannot be maintained over time, changes in global climate, and the deterioration of environmental conditions, which stem from human activities, necessitate a shift towards a circular economic model. In this model, the concept of waste is eliminated as materials are recycled and reintegrated into the production cycle (Ram lakhhan singh, et al., 2017) . The term "food wastes and by-products" refers to the edible materials that remain after going unused, getting discarded, spoiling, or being consumed by pests at any point along the food supply chain. This encompasses everything from the initial agricultural production stages through to the final household consumption stage (Shikha ojha, et al., 2020). Dealing with food wastes and by-products presents a challenge due to the expenses associated with their management. However, growing environmental awareness and legislative pressures demand the development of new approaches to recover and repurpose food waste, rather than simply discarding it (Beatrice Garske, et al., 2020). Recently, there has been an increasing awareness that food waste represents an abundant resource containing numerous nutraceutical compounds and other beneficial substances.

The food waste contains a variety of valuable components such as coloring pigments, fibrous materials, mineral nutrients, protein sources, and substances that can counteract oxidative damage, can be repurposed and utilized across various sectors such as the food, agriculture, cosmetics, and pharmaceutical industries

(Roxana Nicoleta Rațu, et al., 2023). The extraction of compounds from food waste involves three main steps. First, the raw materials go through a pre-treatment process to remove microorganisms while preserving the beneficial biological activities. Second, the desired compounds are extracted using either conventional techniques like solvents, maceration, or steam distillation, or newer eco-friendly methods such as pulsed electric fields, supercritical fluids, microwaves, ultrasound, or high-voltage electric discharges. In the third and final stage, the extracted product undergoes purification to isolate the target compounds (Ming liu, et al., 2017). To protect public health, it is crucial that high-value components undergo a thorough safety assessment and obtain legal approval before being utilized as input materials. The extraction of bioactive compounds from food waste materials requires the use of separation techniques that do not introduce any harmful or hazardous elements. Moreover, the final products must comply with all relevant food regulations. This meticulous process ensures that these compounds can be safely and legally incorporated into various applications while prioritizing consumer safety (Betty jarma arroyo, et al., 2019). Oilcakes, the main byproducts from oil production, are rich in bioactive compounds such as dietary fibers and proteins. These beneficial substances can be extracted from the oilcakes. When consumed, the extracted bioactive compounds from oilcakes can provide advantageous effects for human health. Additionally, they impart unique properties when incorporated into food products (Santosh kumar, et al., 2020). Their chemical makeup can differ based on factors such as the crop variety, harvest timing, and the specific oil extraction methods employed. Extracting bioactive compounds from these materials has gained traction, as it offers a means to valorize the byproducts generated during oil extraction processes. While previous studies have primarily focused on exploring oilcakes as sources of nutritional supplements and antioxidants for food applications, research has also revealed their potential in the production of enzymes, antibiotics, biopesticides, and vitamins, highlighting their diverse applications beyond the food sector (Maria G. leichtweis, et al., 2021).

This paper provides a critical analysis of different methods aimed at valorizing the oilcakes generated as by-products by the oil industry. It also examines the various techniques utilized for extracting bioactive compounds from these oilcake by-products.

Bioactive Compounds Found in Oilseed Cake

Oilseed crops stand out as major agricultural commodities cultivated worldwide. These crops are grown across the globe primarily for extracting oil. Oilcakes serve as abundant sources containing proteins, fibrous materials, unsaturated fatty acids (both monounsaturated and polyunsaturated varieties), mineral nutrients, antioxidant compounds, and vitamins. (Bhukya Jithender, et al., 2019). During the

2020/2021 growing season, soybeans were the leading crop, accounting for more than 61% of the total production. Following soybeans, the next largest crops were rapeseed, peanuts, sunflowers, cottonseed, and groundnuts (A. L. Rathnakumar, et al., 2022). The processing of oilseeds, whether through solvent extraction or mechanical pressing methods, generates substantial quantities of by-products such as peels, oilcakes, and oil sludge. Specifically, oilcakes are the residual materials left over after the oil has been removed from the oilseeds during extraction (Suka Thangaraju, et al., 2020). The oil extraction process can be carried out either through mechanical means, with oil content of approximately 6-7%, or via solvent extraction, where the oil content is less than 1% (S. P. Jeevan Kumar, et al., 2017). Sesame, walnut, pumpkin, and almond oil cakes retain significant oil content (ranging from 5.10% to 48%) even after the seed defatting process (Petraru Ancuța, et al., 2020). On the other hand, the oilcakes derived from olive, hemp, and sunflower are rich sources of fibrous materials and carbohydrates. (Abedini, et al., 2020). The protein content remains substantial across various types of oilcakes, with groundnut oilcakes having the highest level at around 60% and cottonseed oilcakes containing the lowest amount at approximately 24.79% (Ancuta Petraru, et al., 2022). Through extraction processes, these oilcakes can serve as sources for obtaining proteins, antioxidant compounds, phytochemicals, and dietary fiber.

Protein

Plant proteins derived from oilseeds and their byproducts have attracted considerable attention due to their cost-effectiveness (T.P. Sari, et al., 2022). In the context of oilcakes, aqueous extraction following defatting enables the retrieval of proteins at high concentrations, reaching up to 80%. However, utilizing full-fat material results in lower protein content, ranging from 40-50%. Similarly, employing solvents like ethanol, methanol, or acetone yields concentrates with protein content ranging from 45-60% (C. Chang, et al., 2019). Helling et al. noted that using a highly alkaline solution with a pH exceeding 10 can dissolve fat. The aqueous method aids in eliminating anti-nutrients from the samples, whereas dry methodologies lack a step for removing anti-nutritional factors, which is a significant drawback. Rapeseed proteins possess a well-balanced amino acid profile, containing substantial amounts of sulfur-containing amino acids that exceed the requirements for both adults and children. The two main protein groups, napin and cruciferin, collectively make up 85-90% of the total protein content in rapeseed. Nutritionally, rapeseed protein is comparable to soy protein (Lisa Campbell, et al., 2016). Rapeseed protein extracted via ultrasonic and ultrafiltration methods exhibited desirable functional attributes like solubility, emulsification capacity and stability, outperforming soy protein, suggesting its potential as an alternative protein source (Andreas Fetzer, et al., 2020).

Proteins derived from soybean and groundnut oilcakes demonstrate excellent digestibility and absorbability, with a nutritional value equivalent to animal proteins. While soybean protein lacks methionine, groundnut protein is rich in arginine. Linseed oilcake proteins are abundant in arginine, glutamic acid, and aspartic acid. Additionally, these proteins exhibit antifungal and emulsifying properties (Klaudia Kotecka-Majchrzak, et al., 2020).

The proteins extracted from sunflower oilcakes are highly nutritious and easily digestible. They are a rich source of essential amino acids including cysteine, methionine, leucine, valine, isoleucine, tryptophan, alanine, and phenylalanine (Ancuta Petraru, et al., 2021).

From oilcakes, protein hydrolysates, isolates (with a protein content greater than 90%), and concentrates (with protein content ranging from 30-80%) can be prepared. To obtain the protein isolates, a process is employed that begins with solubilizing the proteins using an alkaline solution at a high pH. This is followed by isoelectric precipitation induced by the addition of an acid. The precipitated proteins are then washed and dried. The resulting protein isolates demonstrate desirable functional properties such as high water holding capacity, emulsification ability, and emulsion stability. Due to these properties, they find applications as emulsifiers and functional ingredients (Thi Linh Nham Tran, et al., 2020). Hydrolysates are produced by subjecting protein isolates to hydrolysis. This process modifies the protein structure, resulting in improved functionality, solubility, hydration, and gelling abilities. Additionally, it leads to the formation of a protein fragment known as a bioactive peptide. These bioactive peptides exhibit various biological activities, including antioxidant, antithrombotic, hypocholesterolemic, and immunomodulatory properties (Petraru Ancuța, et al., 2020). Peptides extracted from sesame and rapeseed sources demonstrate beneficial properties such as the ability to lower blood pressure (antihypertensive), counteract oxidative stress (antioxidant), and bind to bile acids. Meanwhile, peptides derived from peanuts exhibit antithrombotic activity, which can help prevent the formation of blood clots (Hemau yuan, et al., 2022).

Antioxidant

Oilcakes contain natural antioxidants such as free phenolic acids, esterified phenolic acids, condensed phenolic acids, flavonoids, and lignans. These antioxidants help in reducing oxidative stress and consequently may play a role in preventing various types of cancers (Ancuta Petraru, et al., 2018). The antioxidant compounds found in oilcakes can be extracted using various techniques such as organic or non-toxic

solvents, high-pressure methods, microwave-assisted extraction, and supercritical fluid extraction. These extracted antioxidants can be incorporated into diverse food products like bakery items, beverages, and extruded snacks (Maria G. Leichtweis, et al., 2021). The primary antioxidants present in oilcakes from sources like sunflower, rapeseed, coconut, mustard, cotton, and sesame are phenolic compounds (Abedini, et al., 2022). Sesame oilcake is a rich reservoir of various phytochemicals including phenolic compounds, flavonoids, tocopherols, vitamins, carotenoids, lignans, pigments, and steroids. These phytochemicals offer numerous health benefits such as antioxidant, anti-cancer, antiproliferative, antimicrobial, anti-inflammatory, neuroprotective, and cholesterol-lowering properties (Petraru Ancuța, et al., 2020).

Dietary Fibers

With growing consumer awareness about the nutritional benefits of dietary fiber, there has been an increasing demand for high-fiber food products (Yao Olive Li, et al., 2017). To meet this rising demand, it is crucial to explore new alternative sources of dietary fiber. Oilcakes, which are by-products generated from oilseed processing, could serve as an inexpensive source. The dietary fiber derived from oilcakes has the potential to be utilized as a functional ingredient, supplement, or additive in the food and pharmaceutical industries (Nayanika Sarkar, et al., 2022). In 2018, Sun et al. employed a combined ultrasonic and alkaline extraction method for isolating insoluble dietary fiber (DF). Compared to conventional methods, their approach allowed for lower temperature (30°C), reduced time (10 minutes), and up to 95% less alkali consumption. Moreover, the physicochemical properties of the extracted dietary fibers were superior due to the ultrasound treatment altering the DF structure, increasing short-chain components and surface area.

Zheng et al. (2018) investigated the effects of various treatments on the properties of defatted coconut dietary fiber (DF). They subjected the DF to acid treatment, enzymatic hydrolysis, and particle size reduction. The study revealed that hydrolysis and particle size reduction led to structural changes, increased water holding and swelling capacities, and higher soluble DF content, while decreasing oil holding capacity, emulsion capacity, and color. In contrast, acid treatment had the opposite impact on these properties.

Extraction of Bioactive Compounds

The conventional protein extraction method involves two main steps: first, an alkaline solubilization stage where insoluble materials are removed by centrifugation, and second, an isoelectric precipitation step at pH 4-5, followed by

centrifugation and neutralization. However, alternative and improved versions of this method have emerged, which can be categorized into three main approaches: aqueous extraction methods, dry techniques, and combined approaches (Tarun Belwal, et al., 2018).

Aqueous extraction utilizes water, either alone or in combination with acidic, basic, or saline agents, as the extraction medium. The extracted solution undergoes isoelectric precipitation, followed by membrane filtration techniques for purification (Daniel Nunez, et al., 2022). Dry methodologies typically involve a milling or deagglomeration pre-treatment step. These methods yield a lower percentage of extracted protein (around 40%) compared to aqueous extraction methods (around 80%). The advantages of dry methods include minimal physical impact on the particles, preservation of the native structure, and lower associated costs. However, the requirement for expensive equipment remains a significant drawback (Sonia Amariei, et al., 2022).

Enzyme treatments present an intriguing approach due to the selective mode of action of enzymes, allowing them to target specific protein bonds. However, the high cost of enzymes and the difficulty in recycling the treatment stream pose challenges to the widespread adoption of this method (Debkumar Chakraborty, et al., 2023).

Dietary fiber (DF) can be obtained using various conventional techniques, such as dry, wet, chemical, gravimetric, enzymatic, physical, microbial processes, or a combination of these methods. Moreover, there have been investigations into more environmentally friendly or innovative extraction methods, including ethanol, water, steam, ultrasonic, hydrostatic pressure, and pulsed electric field extractions (Yvonne Maphosa et al., 2016).

Dry methods typically involve breaking down the material through milling and then separating the fiber fraction using air classification. Wet milling methods, on the other hand, utilize water for fiber extraction and can be categorized into conventional, alkali, and enzymatic approaches, each differing in the use of reagents and process conditions.

Physical methods aim to maintain the structural integrity of the fibers during extraction. Microbial methods involve fermenting the fibers using microorganisms and enzymes. These various techniques offer diverse approaches to extracting dietary fiber, each with its own advantages and applications. (Maphosa Y., et al., 2016).

When it comes to extracting antioxidants, there isn't a one-size-fits-all method. However, to be deemed appropriate, a method should fulfill several criteria. These include selectivity, achieving a high extraction yield, the capacity to reclaim the solvent, employing environmentally friendly solvents, and maintaining the functional properties of the extracted molecules. Conventional methods for antioxidant extraction encompass maceration (solid-liquid extraction) and solvent-based approaches. The selection of solvent is contingent upon the nature of the material and the particular compounds targeted for recovery. (Daniele Naviglio, et al., 2019). When organic acids were combined with water as the extraction solvent, the yield of flavonoids and proanthocyanidins was enhanced compared to using water alone (Juncao, et al., 2018). Traditional extraction methods require large quantities of solvent and extensive processing times, which can be limiting factors. To address these limitations, alternative extraction techniques such as microwave-assisted, ultrasound-assisted, pressurized liquid extraction, and enzyme-assisted extraction can be employed, offering more environmentally friendly and efficient approaches (Ancuța Petraru, et al., 2022). Antioxidants extracted are typically identified and quantified using high-performance liquid chromatography (HPLC) and spectrophotometric methods (UV-Vis). The antioxidant capacity of these compounds is assessed using various assays, including 2,2-diphenyl-1-picrylhydrazyl (DPPH), ferric reducing antioxidant power (FRAP), 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS), cupric reducing antioxidant capacity (CUPRAC), oxygen radical absorbance capacity (ORAC), and peroxide value determination (Madalina Nisto, et al., 2021).

Methods of extraction

Microwave - Assisted Extraction (MAE)

Microwave-Assisted Extraction (MAE) is a method that harnesses microwave radiation to aid in the extraction of compounds from solid materials into a liquid solvent. The heating mechanism in MAE is based on the ability of certain molecules to absorb and dissipate microwave energy, leading to an increase in temperature.

The effectiveness of microwave heating in this process depends on the dielectric constant of the molecules involved. Molecules with high dielectric constants, such as water (dielectric constant of 78.5 at 25°C) and ethanol (dielectric constant of 24.3 at 25°C), are capable of absorbing and re-emitting microwave energy, resulting in the heating of the system. In contrast, solvents with low dielectric constants, like hexane (dielectric constant of 1.87 at 25°C), are relatively insensitive to microwave radiation and do not contribute significantly to the heating process.

By selectively heating the solvent and solid mixture using microwaves, MAE can accelerate the extraction of target compounds from the solid material into the liquid

phase. This technique offers advantages over conventional extraction methods, such as reduced extraction time, lower solvent consumption, and potentially higher extraction yields.

When employing Microwave-Assisted Extraction (MAE), power is a crucial parameter that needs to be optimized to achieve efficient extraction. A study by Chen et al. demonstrated the impact of microwave irradiation power on the yield of trans-resveratrol extracted from tree peony seed oil extracted residues. The researchers observed that increasing the power from 120 to 385 W led to an improvement in the yield, rising from 1.2 $\mu\text{mol/g}$ to 2.9 $\mu\text{mol/g}$, while maintaining a similar purity level between 80% and 90%. This enhancement in yield can be attributed to the increased diffusivity of the solvent, facilitated by the irradiation energy transferring from the plant matrix to the solvent.

However, a further increase in power beyond 385 W resulted in a decline in yield. When the irradiation power exceeded a certain threshold, excessive internal heating occurred, leading to carbonization and potentially triggering undesirable reactions, such as isomerization or degradation of the target compound. Consequently, it is essential to strike a balance between providing sufficient energy for efficient extraction and preventing detrimental effects caused by excessive heating.

Kaur et al. observed a comparable trend in the extraction yield of swertiamarin, amarogentin, and mangiferin as a function of the microwave irradiation power. The optimal yield for swertiamarin and amarogentin was achieved at around 500 W, while for mangiferin, the peak yield was observed at a slightly higher power of 550 W. This finding highlights the importance of carefully selecting the appropriate irradiation power level to maximize the extraction efficiency of specific target compounds, as different compounds may exhibit varying responses to the applied microwave energy.

Michel et al. explored the application of Microwave-Assisted Extraction (MAE) for obtaining antioxidants from sea buckthorn (*Hippophae rhamnoides* L.) berries. They found MAE to be a suitable alternative technique for extracting polar antioxidants, such as quercetin and isorhamnetin, which are typically not extracted efficiently through conventional methods like aqueous maceration or simple pressing. This study highlighted the potential of MAE to access and recover polar compounds that are challenging to extract using traditional approaches.

Furthermore, the researchers recognized that microwave technology could be extended to other processes beyond extraction, such as hydrodistillation (HD). This versatility of microwave-assisted techniques opens up opportunities for efficient extraction and processing of various valuable compounds across different applications.

Lucchesi et al. employed a solvent-free microwave extraction technique to obtain essential oil from *Elletaria cardamomum* L. using only the residual water present in the plant material. Their study revealed that this approach was more effective than classical hydrodistillation for extracting oxygenated compounds from the essential oil. The absence of an external solvent in the microwave extraction process likely minimized thermal degradation and hydrolysis reactions that could potentially degrade oxygenated compounds, thus preserving their integrity and yielding better extraction efficiency compared to traditional hydrodistillation methods.

In summary, Microwave-Assisted Extraction (MAE) offers the ability to control selectivity through the optimization of irradiation power. This selective heating can influence the extraction yield and composition of target compounds, as demonstrated in the case of extracting trans-resveratrol from tree peony seed oil extracted residues. Additionally, the temperature induced by microwave irradiation can also contribute to selective extraction, as observed in the extraction of different sugars (inositols and inulin) from artichoke external bracts. The selective heating effects observed in MAE may arise from the specific solvent composition or the characteristics of the plant matrix being processed. These findings highlight the potential of MAE to achieve targeted and efficient extraction of desired compounds by leveraging the unique heating mechanisms facilitated by microwave irradiation.

Ultrasound Assisted Extraction (UAE)

Ultrasound technology, involving the use of acoustic waves, was discovered towards the end of the 19th century. This technology has found applications in various fields, including food processing as a new green technology, as well as in the pharmaceutical, nutraceutical, and cosmetic industries.

The application of ultrasound technology can enhance the extraction process by facilitating the release of desired compounds from plant materials. At specific frequencies and amplitudes, ultrasonic waves generate cavitation bubbles, which upon reaching an unstable state, implode, releasing high temperatures and pressures. This phenomenon can disrupt cell walls and promote the release of metabolites from the plant matrix.

Several parameters can be modulated to control the behavior of ultrasonic waves during the extraction process. The two primary parameters are frequency (measured in Hertz, Hz) and amplitude (measured in Megapascals, MPa). Power (expressed in Watts, W) is a function of amplitude over time, while intensity (W/m^2) represents the power per unit surface area. Adjusting these parameters can alter the characteristics of the ultrasonic waves, potentially influencing their interactions with the plant samples and affecting the extraction efficiency.

By optimizing the frequency, amplitude, power, and intensity of the ultrasonic waves, it is possible to maximize the extraction yield and selectivity for specific target compounds from plant materials.

In their study, Chen et al. investigated the influence of various factors, including solvent type, temperature, and ultrasonic frequency (ranging from 2 to 8 kHz), on the extraction of betulin from white birch bark. While the ultrasonic frequency was one of the parameters examined, their findings indicated that its effect on the purity of the extracted betulin was less pronounced compared to the impact of the solvent used. The choice of solvent appeared to play a more significant role in determining the purity of the extracted betulin.

Alvez-Filho et al. conducted a study on the extraction of chlorogenic acids from sweet potato peels, where they investigated the impact of different ultrasound power densities ranging from 0 to 50 W/L (equivalent to 500 W in their experimental setup). Their findings revealed that the yields of certain compounds, such as 4,5-dicaffeoylquinic, 3-caffeoyl-4-feruloylquinic, and 3-caffeoylquinic acids, improved with increasing ultrasound intensity. The formation of these compounds was attributed to hydrolysis reactions occurring on other molecules like tricaffeoylquinic acid. This hydrolysis process was facilitated by the generation of hydroxyl radicals ($\text{OH}\cdot$) caused by the ultrasonic waves, which in turn produced hydrogen peroxide (H_2O_2). The presence of hydrogen peroxide and hydroxyl radicals induced hydrolysis reactions, leading to the observed changes in the composition of the extracted compounds. This study highlighted the potential of ultrasound-assisted extraction as a selective process, where adjusting the applied ultrasound power can promote specific reactions or modifications of the target solutes, enabling selective extraction or transformation of desired compounds.

Wei et al. observed hydrolysis reactions occurring during the ultrasound-assisted extraction of polysaccharides from tea flowers. The ultrasonic waves appeared to have the ability to cleave the polysaccharide chains into smaller fragments, thereby affecting the selectivity of the extraction process. A similar effect on polysaccharides was studied by Zhu et al. during the extraction from pomegranate peels, where they varied the ultrasound power from 100 to 200 W, leading to differences in the extracted polysaccharide composition.

Jacotet-Navarro et al. compared three different ultrasonic devices (bath, reactor, and probe) for extracting rosmarinic acid, carnosic acid, and ursolic acid from rosemary leaves. They observed variations in the yields and purities of these compounds, which were attributed to the different ultrasonic power outputs provided by each device. These findings highlight the influence of the specific ultrasonic equipment and the applied power on the selective extraction and modification of target compounds during ultrasound-assisted extraction processes.

Chemat et al. conducted a comparative study between conventional extraction and ultrasound-assisted extraction (UAE) to evaluate the impact of temperature on the yield and purity of artemisinin extracted from *Artemisia annua* L. Their findings revealed that UAE at higher temperatures led to an increase in the extraction yield of artemisinin. However, the purity of the extracted artemisinin decreased due to the enhanced co-extraction of other compounds present in the plant material.

Similarly, Lavoie et al. investigated the extraction of betulinic acid from *Betula alleghaniensis* and *B. papyrifera*. They observed that the concentration of betulinic acid in the extracts increased with higher temperatures. Nonetheless, the extraction process was more selective at lower temperatures, resulting in purer extracts that were not contaminated with squalene, a co-occurring compound in the plant material. These studies highlight the trade-off between extraction yield and selectivity/purity that can be influenced by temperature in ultrasound-assisted extraction processes. While higher temperatures may enhance the overall extraction yield, they can also lead to the co-extraction of undesired compounds, thereby reducing the purity of the target compound. Conversely, lower temperatures often result in more selective extractions, yielding purer extracts but potentially at the cost of lower overall yields.

In conclusion, ultrasonic waves have the ability to enhance plant extraction processes by facilitating the breakdown of cell walls and promoting the release of desired compounds. However, various factors, such as intensity, temperature, and the specific ultrasonic device employed, can induce different mechanisms that influence the extraction and its selectivity.

Adjusting the ultrasound intensity can trigger reactions like the hydrolysis of chlorogenic acids from sweet potato peels, leading to the formation of specific derivatives. Temperature variations during the ultrasound-assisted extraction of polyphenolic compounds from *Aronia melanocarpa* by-products have also been shown to affect the selectivity of the process.

Moreover, a comparison of different ultrasonic devices, such as baths, reactors, and probes, used for extracting compounds like rosmarinic acid, carnosic acid, and ursolic acid from rosemary leaves, revealed differences in the yields and purities of the extracted compounds due to the varying ultrasonic power outputs of these devices.

Ultrasound-induced mechanisms, such as thermal degradation and hydrolysis, can chemically modify the target solutes, enabling selective extraction or transformation of specific compounds. The studies mentioned demonstrate how ultrasonic waves can alter the extraction process, resulting in different selectivities and compositions of the obtained extracts.

Pressurized Liquid Extraction (PLE)

Many studies have employed the pressurized liquid extraction (PLE) technique for extracting pollutants from natural matrices such as soils. However, this technique has also been utilized for the extraction of metabolites from plant materials. PLE operates by applying pressure to heat the extraction solvent above its boiling point, thereby enhancing the extraction efficiency by reaching higher temperatures compared to conventional extraction methods like maceration or Soxhlet extraction. The elevated temperatures achieved in PLE offer several advantages. Firstly, they increase the solubility capacity of the solvent, allowing for better dissolution of the target compounds. Secondly, the reduced viscosity of the solvent at higher temperatures facilitates better penetration into the plant cells. Additionally, the high temperatures can weaken the interactions between the solutes and the plant matrix, further aiding in their extraction.

While these effects generally lead to improved extraction yields, they may also result in a decreased selectivity during the extraction process. As a consequence of the enhanced solubilization and weakened matrix interactions, non-target compounds may be co-extracted alongside the desired metabolites, potentially reducing the selectivity of the extraction.

Dunford et al. investigated the role of temperature, ranging from 80°C to 125°C, on the selectivity of policosanol extraction from wheat straw, germ, and bran. Their study revealed that while higher temperatures did not significantly impact the extraction yield of policosanol, it led to an overall increase in the global yield. Consequently, this rise in overall yield resulted in a decrease in selectivity for policosanol extraction.

Similarly, Gómez-Mejía et al. observed an increase in the extraction yields of p-coumaric acid, trans-ferulic acid, rutin, and hesperidin when extracting polyphenols from citrus peel waste at higher temperatures of 62°C and 90°C, compared to lower temperatures. This temperature-dependent increase in yield was attributed to the enhanced solubilization and extraction of these polyphenolic compounds at elevated temperatures.

These studies highlight the potential trade-off between extraction yield and selectivity when employing pressurized liquid extraction (PLE) at higher temperatures. While higher temperatures generally facilitate better extraction and higher overall yields, they may also lead to the co-extraction of non-target compounds, thereby reducing the selectivity for the desired metabolites.

Hossain et al. investigated the effect of temperature, ranging from 66°C to 129°C, on the extraction of antioxidants from rosemary using pressurized liquid extraction (PLE). Their study revealed that different compounds exhibited varying trends in their extraction yields as a function of temperature. While the yields of some compounds, such as caffeic acid and gallic acid, increased with higher

temperatures, other compounds, including rosmarinic acid, luteolin-7-O-glucoside, apigenin-7-O-glucoside, carnosic acid, and carnosol, exhibited decreased yields at elevated temperatures.

This study highlights that the effect of temperature on extraction yield is not uniform across all polyphenolic compounds. While higher temperatures generally facilitate better extraction, they can also lead to the degradation or alteration of certain polyphenols, resulting in reduced yields. Therefore, the temperature's impact on extraction yield and selectivity depends on the specific target compounds and their stability under high-temperature conditions.

Alvarez-Casas et al. studied the extraction of polyphenols from white grape marc and observed degradation effects on certain compounds at high temperatures. Specifically, they found that the yields of gallic acid and catechin decreased at temperatures above 100°C, while the total phenolic and flavonoid contents increased even at the highest temperature employed.

Similarly, Dobias et al. investigated the extraction of esculetin, rutin, scopoletin, 7-hydroxy-coumarin, and quercetin from various plant samples. Their findings indicated that while the yields of most compounds improved with increasing temperature, the content of quercetin experienced a decline when the temperature was raised from 80°C to 100°C.

These studies demonstrate that high temperatures employed during pressurized liquid extraction (PLE) can have contrasting effects on different polyphenolic compounds. While elevated temperatures generally enhance the overall extraction yields, they may also lead to the degradation or alteration of certain thermally sensitive compounds, resulting in reduced yields for those specific compounds. Consequently, the temperature's impact on extraction selectivity and the yields of individual compounds can vary depending on their thermal stability and susceptibility to degradation processes.

Benito-Roman et al. optimized the extraction of β -glucans and phenolic compounds from waxy barley at high temperatures ranging from 135°C to 175°C. Their study revealed that increasing temperature had a negative impact on the yield of β -glucans, while it positively influenced the yield of total phenolic compounds. However, they observed that high temperatures can lead to fragmentation of the polysaccharide β -glucan, which may explain the observed decrease in its recovery. In another study, Ruiz-Aceituno et al. extracted different sugars, such as inositols and pinitol, from pine nuts. Their findings highlighted that the effect of temperature on the extraction yields varied among these different sugar compounds. While some sugars exhibited increased yields with higher temperatures, others experienced a decrease in their yields.

These studies collectively demonstrate that predicting the impact of temperature on extraction selectivity and yields can be challenging, as different compounds may

respond differently to elevated temperatures. Although higher temperatures generally improve overall extraction efficiency, they can also lead to the degradation or fragmentation of certain thermally sensitive compounds, resulting in decreased yields. Conversely, for some compounds, higher temperatures may enhance their extraction yields. Therefore, optimizing temperature conditions is crucial to achieve the desired selectivity and maximize the yields of specific target compounds during pressurized liquid extraction.

In conclusion, temperature plays a crucial role as the main optimizing parameter for pressurized liquid extraction (PLE). The effect of temperature on the selective extraction of compounds can be positive or negative, contingent upon their chemical structure, as evident from numerous studies discussed. Furthermore, although the application of pressure allows for the utilization of temperatures exceeding the solvent's boiling point, it is not always necessary to apply excessive heat. Additionally, the influence of the number of cycles, which facilitates a pseudo-dynamic extraction, on the selectivity of extraction has received limited attention in research.

This section has examined three techniques (UAE, MAE, and PLE) based on their selective extraction results. Each of these techniques possesses unique advantages stemming from their respective technical parameters, such as the power of ultrasonic waves, microwave intensity, and temperature for pressurized extraction. Furthermore, they offer benefits over conventional extraction methods, including reduced solvent consumption, faster extraction times, and lower energy requirements, notwithstanding the necessary apparatus. However, the primary factor influencing selectivity is not the physical parameters but rather the chemical nature of the extraction solvent. Consequently, due to the solvent's significant effect, it is challenging to favor one technique over another in terms of selectivity. Additionally, the ease of scaling up these techniques for industrial processes should be taken into consideration.

Bioactive compounds extracted from different oilseed cakes

Compounds derived from oilcakes can find new applications as ingredients in food products, functional foods, pharmaceuticals, cosmeceuticals, and materials for biopackaging. Currently, there's a growing awareness among consumers about the importance of maintaining a healthy diet, as nutrition plays a crucial role in preventing lifestyle-related ailments such as obesity, cancer, and diabetes (S. Simoes, et al., 2021).

Plant proteins find applications in food products due to their desirable properties, such as fat/water absorption, emulsification, and texture modification (Lei sha, et al., 2020). Fibers are added to different food items to modify their texture, consistency, rheological characteristics, and sensory properties. Moreover, fibers can be

employed to improve shelf life by leveraging their capacities to form gels, retain water, mimic fats, and thicken (Ayca Aydogdu, et al., 2018). Incorporating antioxidants into foods rich in lipids helps reduce lipid oxidation processes. Although synthetic antioxidants such as butylated hydroxytoluene (BHT) are extensively utilized in food products due to their effectiveness and affordability, consumers tend to prefer natural antioxidants. (Alberta NA Aryee, et al.,2022).

Innovative food products can be created using two types of ingredients: the first consists of new and alternative food components, while the second involves utilizing ingredients derived from the valorization of by-products. To ensure successful adoption, the marketing strategy must consider new regulations, consumer education, and transparent communication (including clear labeling). When introducing ingredients with notable benefits for consumers, such as functional ingredients, the resulting products are labeled accordingly. By-products from the oil industry have the potential to be repurposed as co-products for creating high-value products, food additives, or supplements (Ricardo Gomez – Garcia. Et al.,2021).

Peanut oilseed cake (PNOC)

Peanut oilcake (PNOC) contains abundant proteins with favorable emulsion and foam characteristics. Its nutritional richness makes it well-suited for improving the physicochemical attributes of pasta and cookies. Studies have investigated the extraction and assessment of functional properties from PNOC (Maridula D, et al., 2016). Protein isolates derived from PNOC typically result in fragile films when used independently. Therefore, it becomes essential to incorporate glycerol or employ a crosslinking technique to enhance their mechanical strength while maintaining effective barrier properties (Jain A, et al.,2015) .

Flaxseed oilseed cake (FOC)

Flaxseed oilcake (FOC) is considered a rich reservoir of omega-3 fatty acids, proteins, both insoluble and soluble dietary fibers, lignans, as well as essential vitamins (A, C, D, E) and minerals. (Petraru Ancut, et al., 2020). These components play a role in preventing colon cancer and reducing the risk of cardiovascular diseases.

The increasing interest in vegetarian/vegan alternatives, especially for individuals with dairy sensitivities, has prompted the creation of fermented beverages resembling kefir, infused with different proportions of flaxseed cake. Beverages containing larger proportions of flaxseed cake demonstrated increased thickness because of the presence of mucilage and proteins, along with heightened antioxidant properties attributed to the generation of phenolic compounds and bioactive peptides (Alaa el-din A. bekhit, et al., 2018).

Rapeseed oilseed cake (ROC)

Rapeseed oilcake (ROC) presents valuable potential in food applications. Sausages formulated with canola protein concentrates as a replacement for casein displayed improved sensory characteristics, including taste, texture, and aroma. The proteins extracted from ROC alone are unsuitable for creating biopolymer films due to their inadequate mechanical and antimicrobial properties. Therefore, the addition of emulsifiers and plasticizers becomes necessary. Addressing this challenge, protein hydrolysates from ROC, when combined with chitosan, have exhibited antimicrobial properties against *Staphylococcus aureus*, *Bacillus subtilis*, and *Escherichia coli* (Ancuta petraru, et al., 2022).

Sesame oilseed cake (SOC)

Sesame oilcake (SOC) holds considerable promise for enhancing the nutritional profile, sensory qualities, and physical characteristics of food items. Lignans extracted from SOC at lower concentrations (150 ppm) can serve as food additives, effectively improving the stability of oils. When compared to using BHT, incorporating SOC has been observed to prolong the time before lipid oxidation occurs in butter, all the while maintaining sensory properties. Furthermore, integrating sesame meal (5-20 ppm) into sunflower and soybean oils has been found to hinder thermal degradation and prevent the loss of polyunsaturated fatty acids, attributed to the redox properties of phenolic compounds found in SOC. (Selin sahin, et al., 2018).

Flavonoids and phenolic acids derived from sesame oilcake (SOC) and coconut oilcake (COC), in addition to BHT, have been integrated into vanilla cake recipes to assess their ability to enhance chemical, microbial, and oxidative stability (Z. Aksoylu Ozbek, et al., 2022). The inherent oilcakes preserved stability for a duration of 13 days, surpassing the 11-day stability of the artificial antioxidant. These natural antioxidants displayed resilience to heat and retained sensory appeal for up to 12 days. Incorporating sesame oilcake (SOC) flour into biscuits has been documented to enhance resistance against microbial spoilage, consequently extending shelf life (Senanayake et al., 2019). Moreover, introducing 10% sesame protein concentrate into extruded snacks has been shown to elevate sensory characteristics, color, and protein content, while simultaneously reducing carbohydrate levels (Matthew Olusola Oluwamukom, et al., 2021).

Sharma et al. formulated films using different combinations of sesame protein isolate (SPI) and gum rosin (GR). The blend with an 80:20 ratio exhibited the highest tensile strength and lowest levels of water vapor permeability, moisture, and solubility. As the percentage of GR increased, optical features improved, such as enhanced transparency and reduced color intensity. Additionally, the inclusion of GR

contributed to structural enhancements by reducing porosity and increasing compactness. Overall, the integration of GR into SPI composite films enhanced their mechanical, optical, morphological, and physical characteristics.

Pumpkin oilseed cake (POC)

Pumpkin oilcake (POC) provides natural macromolecules with the ability to form films. Protein isolates extracted from POC have been employed to create biopolymer films. These films displayed exceptional gas barrier properties, surpassing those of commercially accessible polyethylene and polypropylene films by a significant margin, ranging from 150 to 250 times (Ancuta Petraru, et al., 2023).

Hemp oilseed cake (HOC)

Globally, hempseeds, traditionally cultivated for fabric and paper manufacturing, are now attracting more attention for oil extraction. Despite containing minimal levels (<0.3%) of δ -9-tetrahydrocannabinol (THC), industrial hemp remains viable for agricultural cultivation (Michael D. Kleinhenz, et al., 2021).

The integration of oilcakes abundant in antioxidants, essential fatty acids (with a favorable omega-6/omega-3 ratio), fiber, and minerals has resulted in the creation of nutritious snacks that are both healthy and low in calories. The greatest level of approval was attained when these oilcakes were added at their maximum level (Petraru Ancuta, et al., 2020).

Adding 20% hemp flour to bread formulations enhanced the nutritional content of the products, offering increased levels of essential nutrients like proteins, macronutrients, and micronutrients, notably iron (Iulian Eugen Rusu, et al., 2021).

Conclusion

The food industry generates significant waste, presenting an opportunity for research to improve waste management and promote a circular economy. Currently, these waste materials are not fully utilized. Key strategies for waste reduction include converting waste into energy, incorporating it into animal feed, producing fertilizers and compost, and extracting maximum value (such as phenols, fibers, antioxidants, proteins, etc.) for reintroduction into various products like foods, pharmaceuticals, cosmetics, and textiles. This presents both a challenge and an opportunity to enhance the value of food products, reduce disposal costs, and mitigate risks associated with waste residues.

Bioactive compounds extracted from oilcakes offer health benefits, but the extraction process depends on factors like the method used, the raw material type, and the organic solvent utilized. Conventional methods have drawbacks like high solvent usage and energy consumption, driving interest in greener, more environmentally friendly technologies. Additionally, producing ingredients or

additives from food waste and by-products could address certain nutritional deficiencies.

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