Chapter 2:

Lipid Extraction for Halal and Toyyib Applications: Conventional and Emerging Methods

Amal A. M. Elgharbawy and Sharifah Shahira Syed Putra

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Efficient lipid extraction is vital for producing Halal and Toyyib food products, medicines, and cosmetics, necessitating techniques that adhere to Islamic dietary regulations. This chapter reviews conventional methods like mechanical pressing and hexane solvent extraction, emphasizing the importance of solvent selection and addressing challenges such as solvent residues and cross-contamination. Advanced methods like supercritical fluid extraction, enzymatic extraction, and ultrasound-assisted extraction offer benefits like improved lipid quality and reduced environmental impact, yet present challenges in ensuring Halal-compliant enzymes and solvents. We compare these methods based on factors like source material, desired lipid quality, available resources, and Halal requirements, highlighting the need to balance these elements for compliance. Ultimately, the chapter provides insights into developing sustainable lipid extraction methods that cater to the expanding Halal market, enabling informed decisions by researchers, manufacturers, and consumers.

1 Introduction

Lipids, which include oils and fats, are essential in a number of sectors, including the food, cosmetic, and pharmaceutical industries. They serve various purposes, including stabilising and emulsifying products (Kiyotaka Sato, 2018) and play an essential role in human nutrition (Gurr et al., 2016). The need for effective and sustainable lipid extraction technologies is increasing as the world's demand for lipids rises (Gómez-Guillén et al., 2018). To satisfy the expanding Muslim consumer market, the extraction and processing of lipids in the framework of Halal and Toyyib should adhere to Islamic dietary regulations and ethical principles (Ab Talib & Ai Chin, 2018). Toyyib emphasises the cleanliness, safety, and quality aspects of food, whereas Halal refers to the elements of food that are permitted by Islamic law. Thus, careful consideration of the source material, extraction techniques, and any additives used during the process is necessary to maintain the Halal and Toyyib status of lipids (Riaz et al., 2016).
Quran indeed mentioned oils occasionally. There is a verse in the Quran that particularly refers to olive oil and references oil in general. These are the verse's words:

"Allah is the Light of the heavens and the earth. His light is like a niche in which there is a lamp, the lamp is in a crystal, the crystal is like a shining star, lit from ˹the oil of˺ a blessed olive tree, ˹located˺ neither to the east nor the west, whose oil would almost glow, even without being touched by fire. Light upon light! Allah guides whoever He wills to His light. And Allah sets forth parables for humanity. For Allah has ˹perfect˺ knowledge of all things."

[Surah An-Nur: 24, 35]

The verse emphasizes the value and virtue of olive oil, which is used to symbolize heavenly direction and illumination. The significance of purity and quality in food items, especially oils, is emphasized in this passage, even though it does not expressly address lipid or oil extraction techniques. Therefore, to ensure their purity and wholesomeness, the extraction and processing of lipids and oils should adhere to Halal and Toyyib standards.

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In one more occasion, "- *and (We produced) a tree (of olive) that comes forth from the (mount) Tūr of Sinai, which grows with oil and with a dressing for those who eat.*"

[Surah Al-Mu'minum: 23, 20]

Allah SWT discusses the provision of a tree in this verse that yields food and oil for those who eat it. The verse emphasizes the importance of these natural resources as food sources. This highlights the need for employing suitable and authorized extraction techniques to guarantee that the resulting lipids and oils fulfill Halal and Toyyib standards, maintaining their purity and wholesomeness as per Shari'ah compliances. The guidance provided by the Quran regarding the concept of 'halalan toyyiban' is remarkably precise and warrants comprehensive understanding and recognition. This stems from the complexities associated with discerning its intricate processing, handling and overarching operational procedures. The Quran enlightens humanity on effectively engaging in the production and administration of top-notch halal food, while actively avoiding any semblance of doubt or prohibited elements in our day-to-day dietary requirements.

This chapter reviews various oil and lipid extraction processes, from basic to advanced, analyzing their adherence to Halal and Toyyib standards. It examines factors affecting lipid extraction quality and efficiency, challenges in extracting lipids from non-traditional sources like insects and microbes, and potential future developments. Topics include traditional and modern extraction methods, the impact of raw material properties and extraction environments, and strategies to mitigate lipid oxidation. Emphasizing quality and Halal compliance, the chapter concludes with a discussion of the challenges and future directions in lipid extraction.

2 Extraction of Oil and Lipids

Extraction is a technique for separating oil and lipid from plant and animal sources. There are several methods of oil and lipid extraction, including traditional and modern methods. Mechanical pressing is a traditional technique, whereas chemical extraction, supercritical fluid extraction and enzymatic extraction are modern methods. The type of materials (plants and animals), the required oil and lipid quality and the technique's cost-effectiveness all influence the method selection for oil and lipid extraction (Lamsal et al., 2006). Furthermore, the verses '*ya ayyuha al-nas*' (O mankind) and '*ya ayyuha alladhina amanu*' (O believers) underscore the universality and applicability of the 'Halalan Toyyiban' concept, making it fitting for implementation and practice across all groups of society (Mustaffa, 2019).

2.1 Traditional Methods for Oil and Lipid Extraction

Traditional oil extraction methods, like mechanical pressing and organic solvent extraction, have drawbacks including nutrient loss, high energy consumption, environmental concerns, low efficiency, protein denaturation, and high costs (Colivet et al., 2016). The use of toxic solvents in organic extraction worsens these issues. The Quran has emphasized:

'*And make not your own hands contribute to (your) own destruction (harm)*'

[Surah Al-Baqarah: 2, 195]

Therefore, in this section, we discuss the traditional and modern methods for oil and lipid extraction.

2.1.1 Mechanical Extraction (Cold Pressing)

Traditional lipid extraction techniques include mechanical extraction, also known as cold pressing (Fig. 2.1), which use mechanical forces to separate lipids from their source material. This procedure normally entails a number of processes, such as washing, drying, dehulling, and crushing the source material before collecting the released oil (Cancela et al., 2016; Pohndorf et al., 2016). The usefulness and efficiency of mechanical extraction for lipid extraction have been assessed in several studies. As an illustration, Pan et al., (2017) examined the standard techniques for removing oil from microalgae biomass, which include cell drying, cell disruption by chemical, mechanical, or biological means, and lipid extraction using chemical solvents. According to Patel et al., (2018), simple mechanical techniques, such an oil press or expeller press, are typically used to extract oils from seeds. These techniques may also be used to extract oils from microalgae.

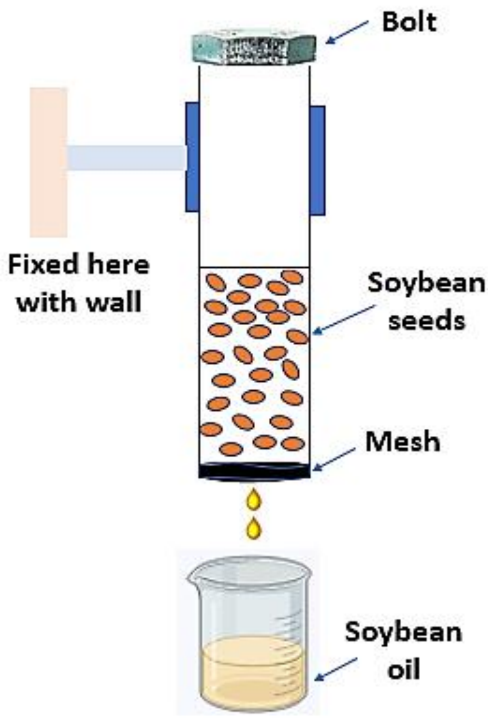

Figure 2.1: Schematic diagram of cold press oil extraction setup (Saleem & Ahmad, 2018)

Mechanical extraction methods recover less oil than solvent extraction techniques, which fills the gap between mechanical and supercritical fluid extraction methods, the latter being efficient but costly (Odewole et al., 2017). Supercritical extraction is highly effective for microalgal lipids, especially long-chain unsaturated fatty acids (Li et al., 2014). Despite this, mechanical extraction has key advantages, which avoids chemical solvents, preserving Halal status and ensuring purity. It also retains nutrients and bioactive components, resulting in higher-quality oil with health benefits. Additionally, Bratfalean et al., (2006) found that mechanically pressed oil had better quality and less acidity compared to solvent-extracted oil.

To sum up, mechanical extraction is an eco-friendly and Halal-compliant technique, applies pressure to extract lipids, preserving nutrients and bioactive elements without chemical solvents. Though it yields less oil than solvent extraction, it suits various oilseed sources. Further research is needed to optimize this process for different oils and lipid sources.

2.1.2 Wet Rendering

Oils and lipids may be extracted by the rendering process from a variety of sources, such as fish oils and animal fats (Fig. 2.2). High temperatures are used in this process to melt the fat, which is subsequently separated from the other ingredients. In the food business, rendering is a typical technique for removing oils and lipids from animal by-products such bones, skin, and offal. Several studies have evaluated the efficiency and effectiveness of rendering for lipid extraction. Vechtomova et al., (2022) evaluated different methods for obtaining rendered animal fats and indicated their advantages and disadvantages.

Rendering methods are used to extract lipids from non-traditional animal raw materials, including microalgae and fish oils. Araujo et al., (2013) assessed five methods for extracting lipids from *Chlorella vulgaris*, while Fattah et al., (2020) optimized ultrasonication and Soxhlet extraction for maximum oil yield from microalgae. Bako, (2017) noted that rendering fish oils at high temperatures can increase susceptibility to thermal degradation. Despite its effectiveness, rendering's high temperatures risk degrading the oil. Fizal et al., (2022) found hydrolysis preferable to wet rendering due to reduced lipid oxidation. Thus, while effective, rendering requires careful temperature management to prevent oil degradation.

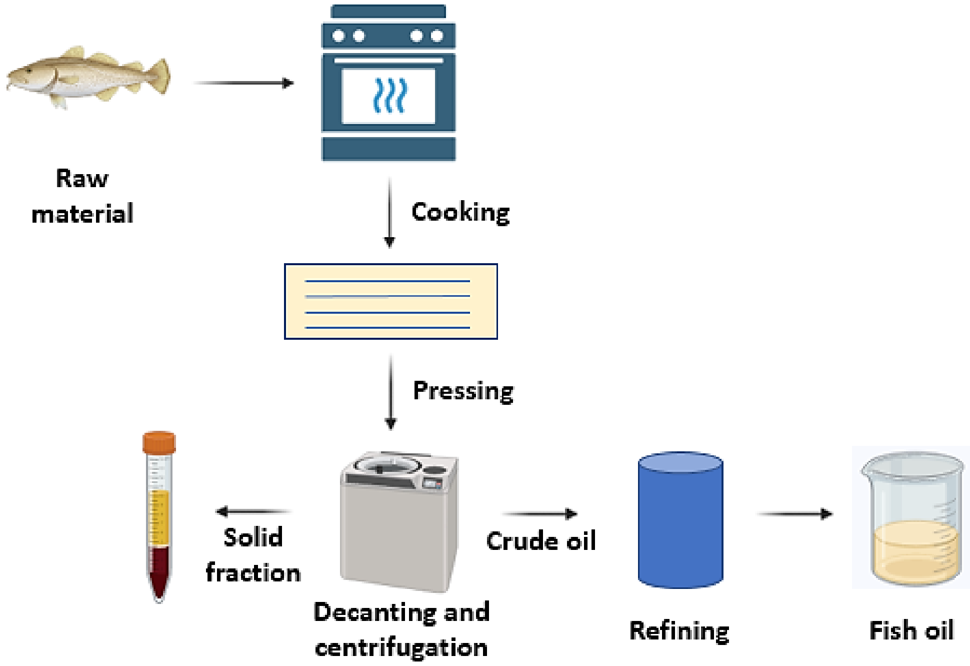

Figure 2.2: Schematic diagram of fish oil extraction using wet rendering method (Jeevitha et al., 2023)

2.2 Modern Methods for Oil and Lipid Extraction

2.2.1 Solvent Extraction

Solvent extraction, or liquid-liquid extraction (Fig. 2.3), uses solvents to dissolve lipids from source materials like plant seeds, microalgae, and spent coffee grounds (Saini et al., 2021). Common solvents used in lipid extraction include hexane, ethanol, and chloroform (Rizwanul Fattah et al., 2020). Hexane is the most widely used solvent in lipid extraction because it is inexpensive, highly oil-soluble, and simple to recover (Dias et al., 2022). Despite its effectiveness, solvent extraction requires large amounts of organic solvents, posing environmental and health risks. Hexane use raises concerns about environmental impact and potential residue, affecting Halal certification due to Toyyib requirements, which emphasize product safety.

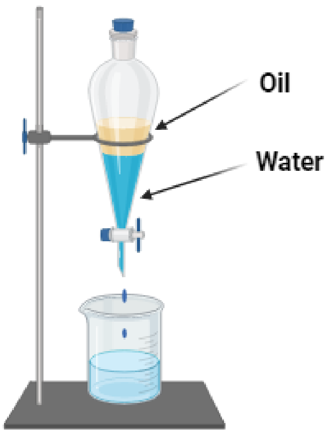

Figure 2.3: Representation of solvent extraction between oil and water

Alternatively, there is an increasing interest in adopting green solvents for lipid extraction, such as ionic liquids (ILs), deep eutectic solvents (DESs) and natural deep eutectic solvents (NADES. The efficiency of green solvents for lipid extraction has been examined in several researches. For instance, Dai et al., (2013) discovered that natural deep eutectic solvents (NADES) exhibited strong solubilization power for both polar and nonpolar molecules when extracting phenolic metabolites from *Carthamus tinctorius L.* Gu et al., (2014) extracted phenolic chemicals from model oil and discovered that DES were a great solvent for extraction.

Besides DES, ILs are gaining popularity for lipid extraction. Although green solvents are currently more expensive than fossil fuel-based solvents, costs may decrease with increased demand and production. Future research should focus on developing automated methods for high throughput lipidomic analysis to improve efficiency, quality, and reproducibility. In "Qawathi' al-Adillah fi Ushul al-Fiqh," Al-Samani puts forth the concept that actions falling under the category of 'haram' are those for which a punishment is prescribed, while halal encompasses actions that are deemed obligatory. On the other hand, 'jaiz' refers to actions that lack punishable consequences or commendable rewards. Any action that promotes well-being for the mind, body and soul falls within the realm of halal (Nafis, 2019). Consequently, the definition of halal, as derived from Quranic and Hadith sources, can be succinctly characterized. Factors influencing lipid extraction include solvent selectivity, hydrophobicity, biochemical composition, and temperature. Table 2.1 summarizes the advantages and disadvantages of these green solvents.

Table 2.1: *Green solvents for lipids and oils extraction.*

Solvent	Advantages	Disadvantages	References
Ionic Liquids	Possesses both ions and enable to design suitable solvent. Less hazardous Non-flammable	Expensive Studies on scaling-up is crucial to assess the feasibility of the process	(Greer et al., 2020; Kianfar & Mafi, 2020)
Deep Eutectic Solvent	Easy to synthesize Safe and inexpensive	Role of these solvents on lipid extraction are yet to be studied.	(Ünlü et al., 2019;Zhang et al., 2022)
Bio-derived solvent	Derived naturally Bio-based feedstocks Low toxicity, biodegradability and renewability	Feedstock supply More research on thermodynamic and kinetic performance needs to be studied	(Dejoye Tanzi et al., 2013;Li et al., 2016)
Liquid polymers	High solubility Safe to be used Amphiphilic in nature	Application in lipid extractions is yet to be studied.	(Correia et al., 2020; Wu et al., 2022)
Supramolecular solvents (SUPRAs)	Present in nature Tunability of the solvent is high Have various polarity regions suitable for extraction	Factor and extraction efficiency for lipid extraction are yet to be studied.	(Moradi et al., 2021; Rubio, 2020)

2.2.2 Supercritical Fluid Extraction (SFE)

Supercritical fluids are recognized as environmentally friendly solvents employed in the extraction of lipids (Abrahamsson et al., 2018). Numerous verses within the Qur'an, such as those found in *Taha*, *An-Nahl*, and *Al-A'raaf*, highlight the provisions bestowed by Allah s.w.t that serve to nourish and sustain our physical well-being (Elgharbawy et al., 2022). According to Nagappan et al., (2019), SFE possess the advantageous characteristic of chemical inertness. SFE uses carbon dioxide as a solvent and has the ability to extract biological components without modifying their chemical structure, which improves its efficiency (Gustinelli et al., 2018). By changing the pressure and temperature throughout the extraction process, SFE method additionally makes it possible for chemicals to be fractionated as reported by Reverchon & De Marco (2006). This enables the solubilities of compounds with desired features to be optimized. Furthermore, unlike organic solvent extraction methods, lipid extracts obtained using supercritical fluids do not contain any residual solvents. As a result, the energy consumption required for product purification is minimal. It is currently used extensively in a variety of industries, including but not limited to the food and pharmaceutical sectors. Additionally, it is widely used in a variety of fields including toxicology, chemistry, textiles, environmental research, polymers, petrochemicals and more (Ahmad et al., 2019).

The extraction process occurs within a cylindrical vessel, known as an extraction column, where finely ground raw material particles are packed to create a porous bed as illustrated in Fig. 2.4. According to Salamatin, (2020), the solvent is introduced initially into the vessel until the desired operational pressure (ranging from 40 to 70 MPa) and temperature (ranging from 40 to 70°C) are achieved. The solvent permeates the oil-containing cells of the plant material, dissolving the extractable oil components. Subsequently, the solvent is circulated through the bed at a specified flow rate, facilitated by an applied pressure gradient. Within the extraction process, the solute undergoes diffusion through the internal transport channels present within the plant particles (Salamatin, 2017). This diffusion process enables the solute to move towards the surface of the ground material. Subsequently, the solute is transported further by the fluid flow towards the outlet cross-section of the vessel (Salamatin, 2016). The concentration gradient of the oil within the individual particles serves as the driving force for the extraction process.

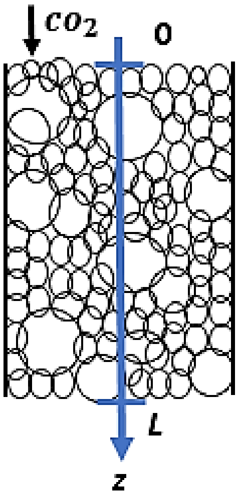

Figure 2.4: The illustration depicts a vertical cross-section of a packed bed in the form of polydisperse spherical particles comprising ground raw material. The particles are shown in two different sizes. The supercritical CO_2 flows in the z-direction, from the top to the bottom of the column while *L* represented the height of the packed bed (Salamatin, 2016).

Gustinelli et al., (2018) observed that employing supercritical fluid extraction (SFE) at 80°C for 1 h increased oil yields from black currant and cloudberry seeds. Similarly, De Souza et al., (2020) found SFE significantly enhanced grape seed oil extraction yield by up to 12.54% compared to other methods. Kuvendziev et al., (2018) evaluated SFE-CO_2 for extracting fish oil from freshwater carp, employing non-toxic solvents and achieving satisfactory extraction efficiency. Salinas et al., (2020) determined optimal SFE conditions for almond oil extraction at 60°C and 40 MPa, yielding approximately 40 ± 1% oil. Hogan et al., (2021) conducted SFE on *Amphora sp.* biomass with or without silica, noting an 8% increase in lipid yield with silica addition. Dimić et al., (2023) found SFE yielded the highest cherry seed oil content ranging from 2.50% to 13.02%, surpassing Soxhlet and cold pressing methods. In summary, SFE efficiently extracts oil and lipids from bioactive materials using carbon dioxide as a solvent, preserving biological integrity. It

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boasts high extraction efficiency and enables compound fractionation, offering high quality product and aligning with green process technologies.

2.3 Emerging Technologies for Oil and Lipid Extraction and Its Factors

Traditional lipid extraction methods like the Bligh & Dyer as well as Folch methods rely on methanol and chloroform combinations, mainly for small-scale lab use (Patel et al., 2018). Emerging technologies (Fig. 2.5) such as enzyme-assisted extraction, ultrasound-assisted extraction (UAE), pressurized liquid extraction (PLE), microwave-assisted extraction (MAE), and green solvent extraction offer sustainable, efficient, and high-quality alternatives. These advancements, summarized in Table 2.2, are transforming the industry, finding applications in pharmaceuticals, food, and cosmetics. Further research and development will optimize and expand these technologies, enhancing their utility across diverse sectors. In the subsequent sections, we will describe some of the notable techniques that have been reported in this field.

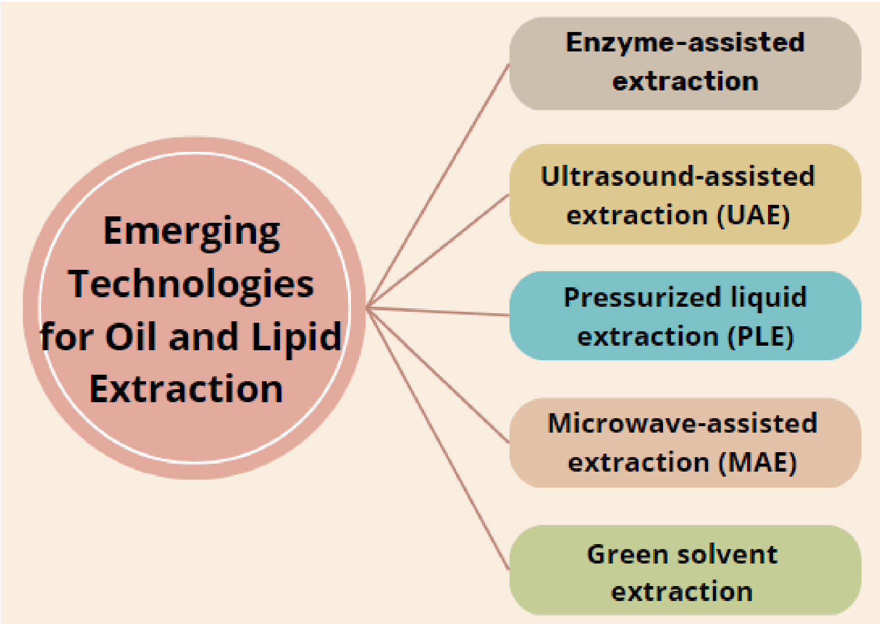

Figure 2.5: Examples of technologies for oil and lipid extraction

Table 2.2: *Comparison of conventional (Soxhlet method) and advanced extraction techniques for analytical-scale extraction.*

Extraction method						
	Soxhlet	**Enzymatic Extraction Method**	**Ultrasound-Assisted Extraction (UAE)**	**Pressurized Liquid Extraction (PLE)**	**Ionic Liquid-Based Extraction**	**Microwave-Assisted Extraction (MAE)**
Description	The sample is placed within a thimble, which is then filled incrementally with freshly condensed extractant (referring to the solvent utilized for extraction) from a distillation flask (López-Bascón-Bascon & Luque de Castro, 2019).	Involves the mechanical crushing of the material followed by the utilization of enzymes to degrade complex structures composed of lipoproteins, lipopolysaccharides, and cell walls (Thilakarathna et al., 2022).	Operates based on the principles of cavitation and oscillation induced by ultrasound waves (Dzah et al., 2020).	A technique that involves using solvents at elevated temperatures and pressures to extract desired compounds (Picot-Allain et al., 2021).	The plant material is mixed directly with ILs, and the temperature, solid-liquid ratio, and extraction time are optimized for the process (Ullah et al., 2019).	Involves the utilization of nonionizing electromagnetic waves within 300 MHz to 300 GHz to induce cell disruption or structural modifications in the sample matrix (Gomez et al., 2020).
Extraction time	3 to 48 h	1 to 12 h	10 – 60 min	5 to 30 min	30 to 60 min	3 to 30 min
Extraction temperature	37 to 80°C	40 to 55°C	45 to 60°C	50 to 200°C	80 to 140°C	60 to 80°C
Advantages	No filtration is required, easy to handle	High efficiency and mild conditions	Easy to use and can be recycle	Fast extraction and low solvent consumption	High extractant capacity and selectivity	Low solvent use, no filtration necessary, rapid extraction
Disadvantages	Longer extraction time, large amount of solvent used and not easily automated	Expensive equipment, limited-scale operations, and potential environmental pollution	Large solvent volume and filtration step is necessary	Clean-up step is needed and possible degradation of thermolabile analytes	Poor recovery and problems with reproducibility	Filtration and cooling time for vessel is necessary, low selectivity

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2.3.1 Enzymatic Extraction Method

Enzymatic extraction method involves the mechanical crushing of the material followed by the utilization of enzymes to degrade complex structures composed of lipoproteins, lipopolysaccharides and cell walls. This enzymatic process facilitates the release of oil during extraction as reported by Thilakarathna et al., (2022). During the enzymatic extraction process, non-oil components separate from the complex and partition into the extractant (Fig. 2.6), while the oil forms a separate phase according to Song et al., (2019). They discovered that by treating peony seeds with 0.15% (w/w) enzyme (pectinase) for 1 h at a temperature of 50°C were able to achieve the highest oil yield (92.06%).

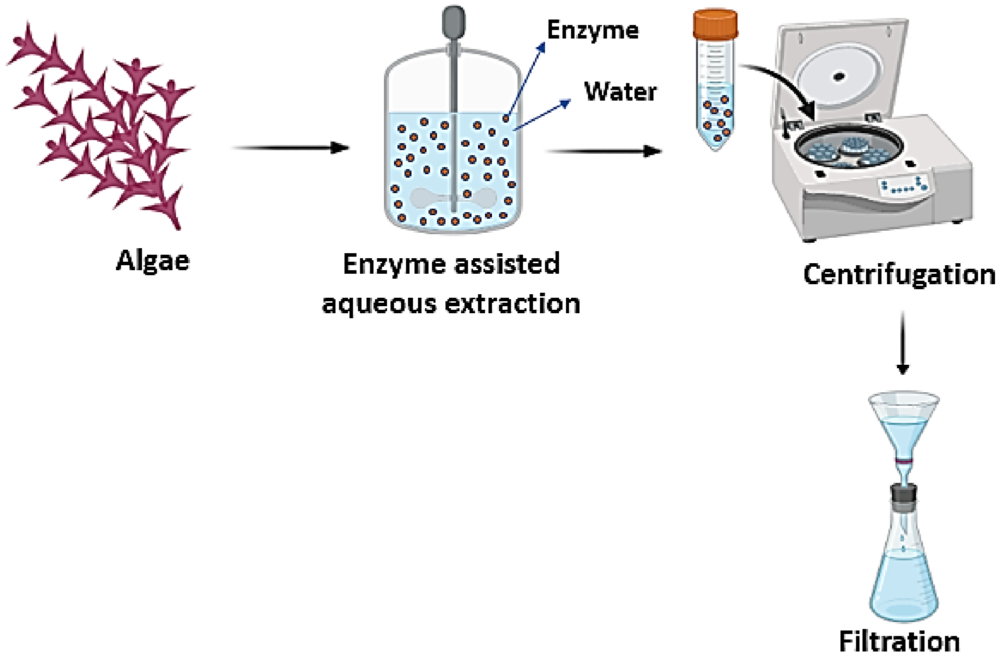

Figure 2.6: Schematic diagram of enzyme assisted aqueous extraction steps from algae

As a result, the presence of enzymes in the extraction process aids in softening and encourages the degradation of seed tissues, thereby enhancing solvent permeability. Enzymes, however, often take longer to dissolve the cell wall such as lignin, hemicellulose, cellulose and pectin compounds (Çakaloğlu et al., 2018). This time delay can be overcome by the application of ultrasound. Ultrasonic waves induce bubble cavitation, and when combined with enzymes, they can enhance the capacity of oil to be extracted. Together, enzymes and ultrasound contribute to the enhanced efficiency of oil extraction. Additionally, ultrasound is extremely important in enhancing the interaction of the enzymes with the substrate. This importance is primarily due to the disruptive effect of ultrasound on tissues, resulting in increased mass transfer of the solvent-enzyme mixture into the cellular structure (Roselló-Soto et al., 2016). Besides that, ultrasound increases the surface area of contact between the seed powder and the enzyme-aqueous solution, enhancing the interaction between the enzyme and the substrate.

According to Goula et al., (2018), ultrasonication significantly enhanced enzymatic oil recovery, boosting pomegranate seed oil extraction by 18.4%. Similarly, Liu et al., (2019) found a combination of neutral cellulase, protease, and pectinase enzymes increased *Sapindus mukorossi* seed kernel oil extraction to 82.67%, albeit with prolonged incubation times of 8 to 12 hours. Su et al., (2019) introduced an enzyme-assisted extraction method for Black soldier fly larvae (BSFL) fat, with Protamex enzyme yielding a remarkable 2.2-fold increase in fat yield compared to untreated extraction. Blanco-Llamero et al., (2021) investigated simultaneous use of Celluclast, Viscozyme, and Alcalase enzymes, achieving oil yields of approximately 29% with a synergistic effect observed. Their ultrasound-assisted enzymatic pretreatment at

55°C for 6 h yielded the highest polar lipid content. Enzyme-assisted extraction yields superior-quality products suitable for human consumption, with reduced environmental impact compared to solvent methods (Mwaurah et al., 2020). It enables extraction of diverse compounds from plants, requiring a thorough understanding of oil seed or fat structure for efficacy of enzyme-assisted extraction.

2.3.2 Ultrasound-Assisted Extraction (UAE)

Ultrasound-assisted extraction (UAE) is gaining prominence for seed oil extraction, offering an alternative to conventional methods like Soxhlet extraction. Recognized for its potential in food and phyto-pharmaceutical industries (Böger et al., 2018), UAE stands out for its unique mechanism and advantages over traditional approaches. It enhances oil extractability by reducing extraction times, operating at lower temperatures, and minimizing energy and solvent usage compared to Soxhlet and mechanical extractions (Thilakarathna et al., 2023). For example, Mohammadpour et al., (2019) compared UAE with Soxhlet extraction for *Moringa peregrina* seed oil, achieving a maximum yield of 53.101% with optimal UAE parameters of 26.3 mins extraction time and 17.8 mL/g liquid-to-solid ratio.

According to Dzah et al., (2020), ultrasound-assisted extraction (UAE) is recognized as cost-effective due to its reliance on cavitation and oscillation induced by ultrasound waves. This can be achieved via direct methods using an ultrasound probe or indirect methods with a bath sonicator device. Ultrasound waves create vibrations, generating voids in the medium and transferring energy to solid particles. Cavitation bubbles form and collapse near the solid surface, influenced by factors like voltage waveform duration and processing temperature Zhang et al., (2017). Intense cavitation disrupts particles at a microscopic level, increasing diffusion rates (Roohi et al., 2019). This process ruptures cell walls, aiding the release of desired compounds trapped inside cells into the solvent medium (Mushtaq et al., 2020).

Hence, as per Misra et al., (2017) findings, the phenomenon of cavitation plays a significant role in the mechanism of UAE for oil and lipid extraction. This is by promoting mass transfer through intense mixing and streaming which involves various mechanisms such as capillary effects, diffusion, surface structural damages and transient cell membrane passage. Figure 2.7 illustrates a summary of the oil extraction mechanism using UAE.

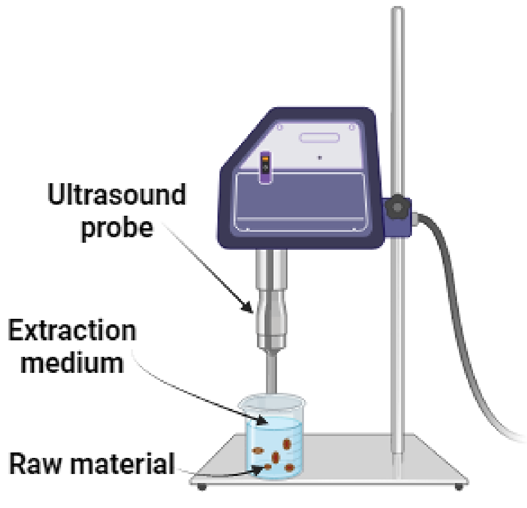

Figure 2.7: Ultrasound-assisted oil extraction method

In the study by Sanwal et al., (2022), maximum recovery of sea buckthorn seed oil (SBSO) reached approximately 6.87 g/hg, notably higher than solvent extraction. UAE reduces extraction time and operates at lower temperatures, preserving nutritional and bioactive components (Jalili et al., 2018). Study by Rezvankhah et al., (2018) demonstrated that the hempseed oil extraction yield using UAE was 33.61% w/w, lower than the yield obtained with the Soxhlet method (37.93% w/w). However, UAE had a much shorter processing time of 10 mins compared to the Soxhlet method, which required 8 h. In a study conducted by Zhang et al., (2018), they investigated the utilization of enzyme-assisted extraction for extracting lipids from the microalga *Scenedesmus sp*. They optimized the conditions and employed xylanase, cellulase, and pectinase enzymes during the extraction process which resulted in a significant improvement in lipid yield, with approximately twice the amount obtained (13.8 ± 0.4 g/100 g) compared to the control group that did not undergo enzymatic treatment.

In conclusion, UAE is a valuable method for oil and lipid extraction, offering faster times, reduced energy consumption, and improved yields. Cavitation induced by ultrasound enhances mass transfer, while lower temperatures preserve nutritional components. Further optimization and research are crucial for maximizing UAE's potential in oil extraction.

2.3.3 Pressurized Liquid Extraction (PLE)

Pressurized liquid extraction (PLE), also known as pressurized fluid extraction, involves using solvents at elevated temperatures and pressures to extract desired compounds (Picot-Allain et al., 2021). This technique alters the physicochemical properties of the solvent, enhancing mass transfer rates, reducing surface tension and viscosity, and increasing analyte solubility (Alvarez-Rivera et al., 2019). Consequently, the solvent can penetrate solid matrices more effectively, resulting in higher yields compared to traditional methods. PLE offers faster extraction processes and reduces solvent consumption for solid materials due to decreased viscosity and surface tension. Additionally, automated PLE instruments enhance reproducibility and enable less labor-intensive techniques to be developed.

In He et al., (2019) study, microalgal lipids from *Isochrysis* biomass were extracted using Soxhlet, Folch, and PLE methods, with PLE at 90% ethanol concentration yielding the highest lipid extraction efficiency (41.5 wt%) and total fatty acids recovery (92.17 wt%). De Mello et al., (2019) achieved a 49.1% yield from crambe seed oil using PLE, while their 2021 study on pressurized ethanol extraction from radish seeds resulted in a 36.6% yield at 5 MPa pressure, with no significant impact observed from further pressure increases (De Mello et al., 2021). Villanueva-Bermejo et al., (2019) demonstrated near 100% recovery from chia seeds using food-grade ethanol at 60ºC with PLE within a 10 min extraction period.

In conclusion, the results of the study on PLE indicate that this technique offers promising outcomes for the extraction of different oils or lipids from various seed or microalgal sources. When pressurized ethanol was used at particular temperatures and pressures, it worked well to provide substantial oil yields in only a few short minutes of extraction time. These findings demonstrate the possibility of PLE as a workable method for successful oils or lipids extraction.

2.3.4 Ionic Liquid-Based Extraction

Ionic liquids (ILs) have recently acquired interest for solvent extraction and have a lot of potential for extracting lipids from microalgae. It is known colloquially as "Newton's Liquid", where liquid salts are created through the combination of an organic cation and an organic or inorganic anion as shown in Figure 2.8. ILs can be categorized into two distinct types: aprotic ILs (APILs) and protic ILs (PILs). Protic ILs, unlike aprotic ILs, are formed by the transfer of protons from a Brønsted acid to a Brønsted base, resulting in the formation of molten salts (Guo et al., 2020). According to De Jesus & Maciel Filho (2022), the key distinguishing characteristic between PILs and APILs is attributed to the transfer of protons from an acid to a base, resulting in the presence of both proton donor and acceptor sites. This unique feature enables the construction of a hydrogen-bond network within PILs.

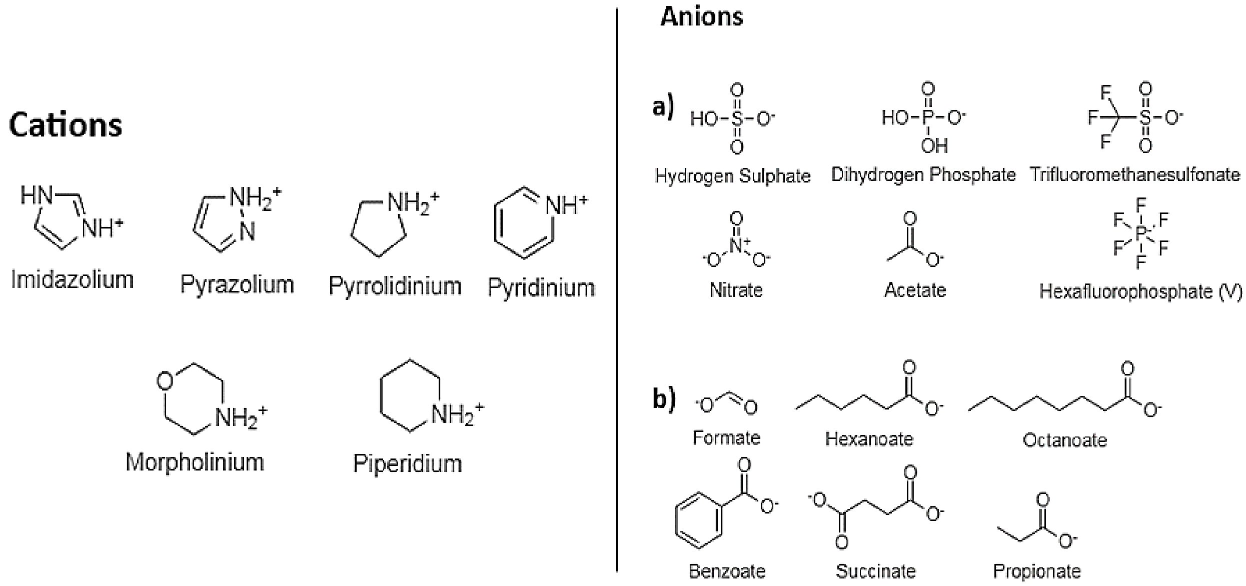

Figure 2.8: The chemical structures of cations and anions (a) for preparing aprotic ILs (APILs) and (b) protic ILs (PILs) commonly employed to form ionic liquids

Extracting oils and lipids using ILs have offers an alternative to conventional solvent extraction techniques that use hazardous solvents, require prolonged extraction times, and use a lot of energy (Motlagh et al., 2019). The use of ILs offers a more effective and eco-friendly method for extracting the lipids from microalgae. Furthermore, the previous results of the study suggest that using ILs has improved the efficiency of extracting lipids from microalgae. Even under mild temperatures ranging from 80 to 140° C, these ILs may successfully dissolve both dry and wet microalgae (Shankar et al., 2017). The adaptability of ILs in aiding lipid extraction from microalgae is further highlighted by the fact that the extraction procedure can be carried out with or without the addition of a co-solvent.

For examples, Zhang et al., (2018) explored the potential of two different ILs which namely, imidazolium 1-ethyl-3-methylimidazolium ethylsulfate [C_2mim][$EtSO_4$] IL and phosphonium (tetrabutylphosphonium propanoate [P_{4444}][Prop]) in facilitating the extraction of PUFA (polyunsaturated fatty acid) containing lipids from *Thraustochytrium sp.* (T18). They effectively observed a disruption of the cells which facilitated the efficient recovery of lipids within a relatively short duration (1 h) at room temperature. Furthermore, Egesa & Plucinski, (2022) conducted a comprehensive study that successfully demonstrated the effectiveness of utilizing ILs for the extraction of lipids from microalgae that were previously separated magnetically. They achieved an impressive extraction efficiency of 99% with ILs/hexane, whereas the use of hexane alone resulted in a mere 5% extraction efficiency. Additionally, Lozano et al., (2020) reported that the extraction process for obtaining oils from dry microalgae (*Chlorella vulgaris* or *Chlorella protothecoides*) involved incubating them in a suitable binary mixture of ILs at a temperature of 110° C.

In conclusion, ILs offer numerous advantages over conventional solvents for oil and lipid extraction, enhancing efficiency and reducing waste through recyclability. They have proven effective in extracting oils from diverse sources. Further research is needed to optimize IL selection, process conditions, and scale-up. Overall, ILs present a promising and sustainable method for oil and lipid extraction across various industries.

2.3.5 Microwave-Assisted Extraction (MAE)

Microwave-Assisted Extraction (MAE) technology offers a rapid, efficient, and safe method for lipid extraction, minimizing exposure to high temperatures and preserving structural integrity (Krishnan et al., 2020). MAE utilizes nonionizing electromagnetic waves within the 300 MHz to 300 GHz frequency range to induce cell disruption or structural modifications in the sample matrix (Gomez et al., 2020). As seen in Figure 2.9, both heat and mass gradients synergistically facilitate the extraction of high-value compounds by promoting their movement outward from the cells. Energy transfer occurs via dipole rotation and ionic conduction mechanisms, facilitating the extraction of compounds from cells (Chemat et al., 2019). Heat and mass gradients work synergistically to enhance extraction yields while reducing processing time. Ionic conduction involves ion migration through the solution, leading to homogeneous heating as the solvent resist's ionic migration under electromagnetic wave exposure.

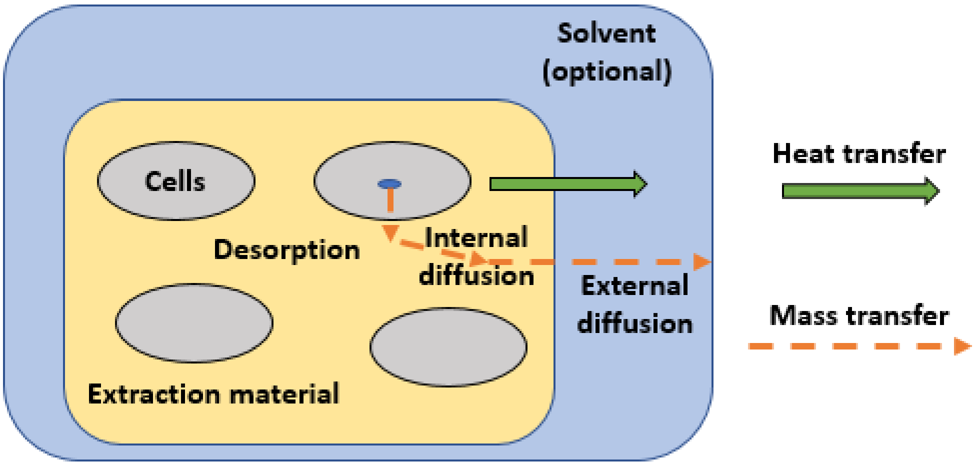

Figure 2.9: Heat and mass transfer gradients in microwave-assisted extraction (MAE) (Chemat et al., 2019)

MAE devices can be categorized based on the amount of microwave energy applied to the sample. There are two main classifications (Luque-García & Luque De Castro, 2003):

a) multimode systems, where microwave radiation is dispersed throughout a space, ensuring uniform treatment of the samples, and
b) single-mode or focused systems, which apply microwave energy specifically to the targeted zone of the sample to achieve a more efficient extraction.

In many cases, multimode systems are employed in conjunction with closed vessels, enabling the application of high pressure during the extraction process. Conversely, single-mode systems are typically associated with open vessels that operating at atmospheric pressure (Gomez et al., 2020). This technology has been positively evaluated by numerous studies and is considered highly scalable in its application.

De la Fuente et al., (2022) compared MAE with Soxhlet extraction for oil recovery from salmon, finding MAE achieved impressive recovery rates of 69% for total lipid content in backbones and heads and 92% for viscera. In another study by the same authors reported that MAE efficiently extracted over 50% of total lipid content from heads of European seabass (*Dicentrarchus labrax*) and gilthead seabream in less than 11 minutes (De la Fuente et al., 2022). Patra et al., (2022) obtained optimal extraction yields of 78.4 wt% for sea buckthorn pomace and 59.9 wt% for seeds using a microwave reactor with ethanol solvent at 80° C and 150 W. Pandey & Shrivastava (2018) investigated the effect of MAE parameters on rice bran oil recovery, achieving over 95% oil recovery in 8 to 10 minutes with varying microwave powers (200, 320, 450, and 560W) and solvent-to-bran ratios (1.6, 2.3, and 3 mL/g). These studies highlight MAE's efficiency in lipid extraction from diverse sources with reduced processing times and solvent usage / consumption.

In summary, MAE is highly effective for extracting compounds from diverse samples by leveraging nonionizing electromagnetic waves to induce structural changes. Energy transfer mechanisms such as ionic conduction and dipole rotation enhance extraction efficiency. MAE systems range from single-mode, focused setups for targeted extraction to multimode systems for uniform treatment, offering versatility across sectors. Whether operating at atmospheric pressure in open vessels or employing closed vessels for high-pressure applications, MAE consistently delivers enhanced yields and shortened processing times.

2.4 Green Extraction for Oils and Lipids

In response to growing concerns about environmental and human safety, researchers are increasingly focusing on alternative and eco-friendly extraction methods, aiming to overcome the limitations of conventional techniques (Fraterrigo Garofalo et al., 2021). 'Green chemistry' principles, introduced by Anastas et al. (2002) in the 2000s, advocate for the development of processes that minimize the use of hazardous substances. Emerging extraction technologies employ non-conventional solvents to reduce processing time and energy consumption while producing high-quality, stable products (Chemat et al., 2019). These efforts align with the push for ecologically responsible and sustainable practices in the chemistry industry, where industries are actively seeking alternatives to organic solvents to address safety, health, environmental, and regulatory concerns (Roselló-Soto et al., 2018).

The Quran conveys the idea that individuals are instructed to consume food that is both permissible and of high quality (Toyyib), while avoiding excess or surpassing reasonable limits (Fatmawati, 2019). A verse from *Al-Baqarah* (2:168) explains: "*O mankind, eat from whatever is on earth [that is] lawful and good and do not follow the footsteps of Satan. Indeed, he is to you a clear enemy*".

Regarding the concept of "Halalan Toyyiban" which was previously discussed, this notion is also referenced in a verse from *Al-Maidah* (5:88) as follows: "*And eat of what Allah has provided for you [which is] lawful and good. And fear Allah, in whom you are believers*". Within that verse, Allah instructs individuals to consume the sustenance that is both permissible (halal) and of high quality (good) which He has provided.

Innovative methods like green extraction have emerged for efficient and environmentally friendly oil and lipid recovery. Green extraction utilizes alternative solvents and renewable natural substances to produce high-quality extracts, aligning with circular bioeconomy principles. Certain green solvents, although nonpolar, possess solubility characteristics similar to conventional solvents, effectively dissolving similar molecules (Mwaurah et al., 2020). To establish and implement green extraction processes in both laboratory and industrial settings, three key solutions have been identified to ensure efficient utilization of raw materials, solvents, and energy as reported by Ivanovs & Blumberga, (2017).

a) the existing process optimization and improvement,
b) the use of non-specific facilities,
c) innovations in processes and procedures, including the discovery of alternative solvents.

Laboratory experiments have showcased various environmentally friendly solvents as viable substitutes for conventional organic solvents like n-hexane, hexane, and chloroform in lipid extraction processes (Ibrahim et al., 2019). These solvents are particularly adept at extracting lipids from oilseeds and microbial fatty acids, facilitating the production of biodiesel and oils enriched with valuable compounds like terpenes and tocopherols (De Jesus et al., 2018; Probst et al., 2017). Terpenes, comprised of isoprene units, encompass significant compounds such as *d*-limonene, *p*-cymene, and α-pinene, primarily sourced from agricultural residues. For instance, *d*-limonene is extracted from citrus fruits, while α-pinene is derived from pine sources (Jeevan Kumar et al., 2017). In a comparative study by De Jesus et al., (2019) on lipid extraction from microalgae, green solvents like cyclopentyl methyl ether (CPME) and 2-methyltetrahydrofuran (2-MeTHF) exhibited better selectivity towards fatty acid yields. Sed et al., (2018) utilized *Scenedesmus dimorphus* biomass and a green solvent composed of octanoic acid and dodecanoic acid, achieving remarkable extraction efficiencies of up to 99% for proteins, 86% for carbohydrates, and 99% for neutral lipids.

In conclusion, the rising demand for environmentally friendly technologies that cause less harm to the planet has been the primary driving force behind the support for replacing fossil-based solvents with green solvents, given the global issue at hand. This support has been largely influenced by governments and populations worldwide.

2.5 Quality of Oil or Lipid after Extraction

Oil or lipid extraction is an essential process utilized in diverse industries, including food, pharmaceuticals, cosmetics, and biofuels. The quality of the extracted oil or lipid plays a pivotal role in determining the effectiveness of the final product and ensuring consumer satisfaction. Considering the well-established knowledge, it is widely recognized that the oxidative degradation of food lipids can lead to formation of various compounds that have the potential to cause adverse health effects (Nieva-Echevarría et al., 2020). Othman et al., (2017), emphasize that the Prophet Muhammad (PBUH) always directed his followers to consistently prioritize matters concerning halal nutrition, as evidenced by a hadith narrated by Anas bin Malik, in which the Prophet stated: "It is the duty of Muslims to seek halal". This discussion, will explore the key factors influencing the quality of oil or lipid after extraction, along with the methods employed to evaluate its overall quality.

2.5.1 Factors Influencing Quality of Oil or Lipid Extraction

Ahmed et al., (2016) reported that food quality can deteriorate due to lipid or oil oxidation, which occurs through autoxidation, enzyme-catalyzed oxidation, and photo-oxidation mechanisms. Autoxidation, the primary process in lipid oxidation, involves initiation, propagation, and termination stages. In initiation, free radicals abstract hydrogen atoms from polyunsaturated fatty acids, leading to diene conjugation and stabilization (Huang & Ahn, 2019). Subsequently, these conjugated dienes transform into highly reactive peroxyl radicals (lipid radicals) in the presence of oxygen. The rate of peroxyl radical formation depends on the total number of bis-allylic carbons in a fatty acid molecule. Reactive oxygen species (ROS) are the main compounds responsible for abstracting free hydrogen atoms from lipid molecules Abeyrathne et al., (2021). Fig. 2.10 illustrates the lipid oxidation process, depicting the stages of initiation, propagation, and termination. During propagation, peroxyl radicals continually abstract hydrogen atoms from adjacent polyunsaturated fatty acids until the termination step, where unstable peroxyl radicals convert into stable non-radical compounds (Yaman & Ayhanci, 2018).

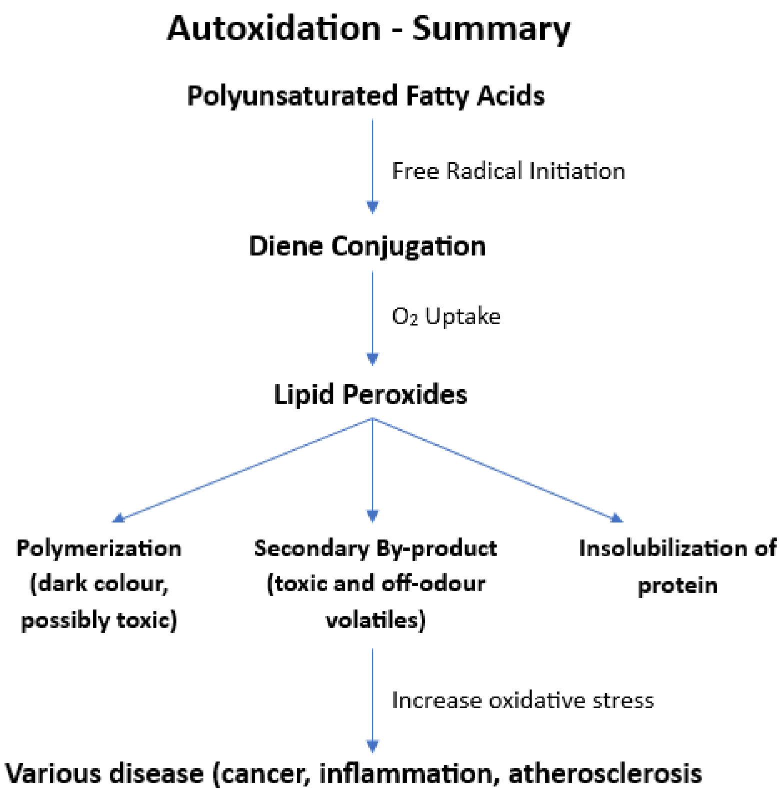

Figure 2.10: Schematic diagram depicting the pathway of lipid oxidation along with the various categories of resultant products (Abeyrathne et al., 2021)

Additionally, the quality of oil or lipid greatly depends on the source material from which it is derived (Roy et al., 2022). According to Soumya et al., (2021), different plant or animal sources possess distinct compositions of essential fatty acids, antioxidants, and other bioactive compounds, thereby impacting the overall quality of the extracted lipid. Thus, the extraction method chosen plays a pivotal role in determining the final product's quality. For instance, cold pressing, solvent extraction, and supercritical fluid extraction techniques yield oils with differing compositions, stabilities, and flavors. Maintenance of oil or lipid quality necessitates careful consideration of storage conditions for both the source material and the extracted product (Mousavi et al., 2021). Exposure to heat, light, and oxygen can induce oxidation and rancidity, diminishing nutritional value and sensory attributes (Othón-Díaz et al., 2023). Furthermore, factors like pressure, temperature, and duration during extraction significantly impact the chemical composition and overall quality of lipids (Zhou et al., 2022). Impurities or contaminants in the source material or extraction process can also compromise oil quality. To ensure oil and lipid quality aligns with halal and Toyyib principles, meticulous control of these parameters and appropriate purification measures are imperative. This aligns with the words of Allah s.w.t (Saad & Ramli, 2019):

Meaning: "*O you who believe! Eat of the clean and pure that We have provided for you, and be grateful to Allah, if it is Him you worship*".

[Surah al-Baqarah: 172]

The Quranic verse clearly suggests that it is important for food and drinks to not only be permissible (halal) but also of high quality, containing the essential minerals and vitamins that the human body needs. The mention of 'be grateful to Allah' in the verse also indicates that consuming halal and nutritious food and beverages brings not only physical advantages but also spiritual benefits. This can lead to an increase in a person's God-consciousness (*taqwa*) and gratitude (*syukur*) towards Allah s.w.t. (Yahaya & Ruzulan, 2020).

2.5.2 Methods to Evaluate Oil or Lipid Quality

Research indicates that improper practices among food handlers within the food industry are accountable for approximately 97% of cases of foodborne illnesses, resulting in the unfortunate loss of around 1.9 million lives worldwide on an annual basis as reported by Saad & Ramli, (2019). As documented in theQuran, specifically in the verses of al-Maidah (5:4), al-A'raf (7:157), al-Baqarah (2:168), al-Mu'minun (23:51), and al-Nahl (16:114), the food processing sector is required to adhere to the principles of 'Halalan Toyyiba' throughout the entire journey from agricultural origins to consumption (Saad & Ramli, 2018). The effectiveness, stability, and broad application of oils and lipids can all be influenced by their quality.

Oils and lipids are evaluated using a variety of techniques to determine whether they meet industry standards and customer expectations. For example, the fatty acid composition, peroxide value, FFAs content, and oxidative stability of the lipid are frequently evaluated using chemical tests (Pizzimenti et al., 2023). In a study conducted by Li et al., (2017), a comparison of the physicochemical characteristics of oil obtained using ultrasonic-assisted aqueous enzymatic extraction (UAAEE) with solvent extraction (SE) oil and cold pressing extraction (CPE) oil was performed. The results showed that UAAEE oil had similar refractive indices and saponification values to SE and CPE oils. However, UAAEE oil exhibited a higher iodine value, indicating a higher degree of unsaturation, and demonstrated better stability against oxidation, as evidenced by its low peroxide value. This study offers important information about the oil's purity, freshness, and oxidation susceptibility.

Besides that, the oxidation state of the oil and any potential sensory changes can be determined by analyzing the oil's colour, viscosity, and melting point. According to Delgado-Sánchez et al., (2021), fresh oils typically have distinct melting points, low viscosity and exhibit clear colours. Additionally, the organoleptic characteristics of the oil can be assessed using sensory analysis, which also includes assessments of taste, odour and overall acceptability. Sensory evaluation experts are capable to identify odd tastes, rancidity and other undesirable characteristics (Sharif et al., 2017). In addition, accelerated stability tests can expose the oil to high temperatures and assess how long it will remain stable in different conditions (Manzocco et al., 2012). This testing provides insight into the oil's stability and its duration of storage. A study by Aytaç, (2022) demonstrated the quality of coconut oil obtained through various extraction techniques, including Soxhlet, cold press, and SFE under different conditions. Based on their findings, it was determined that SFE is the superior method for obtaining coconut oil in terms of several factors, including fatty acid content, yield, and health properties although all techniques have a long shelf-life and minimal rancidity.

After extraction, several variables, such as the source material, the extraction technique, the storage conditions, the processing parameters and contaminants, can affect the quality of the oil or lipid. Its quality is extensively assessed by physical, chemical, sensory and stability tests (Fig. 2.11) to ensure that the highest-quality products are delivered to customers for a variety of uses. In conclusion, to produce healthier and more sustainable products across a range of industries, adherence to high-quality standards during the extraction process is essential. Future improvements to products will be facilitated by ongoing research and breakthroughs in analytical techniques that will further our understanding of oil and lipid quality.

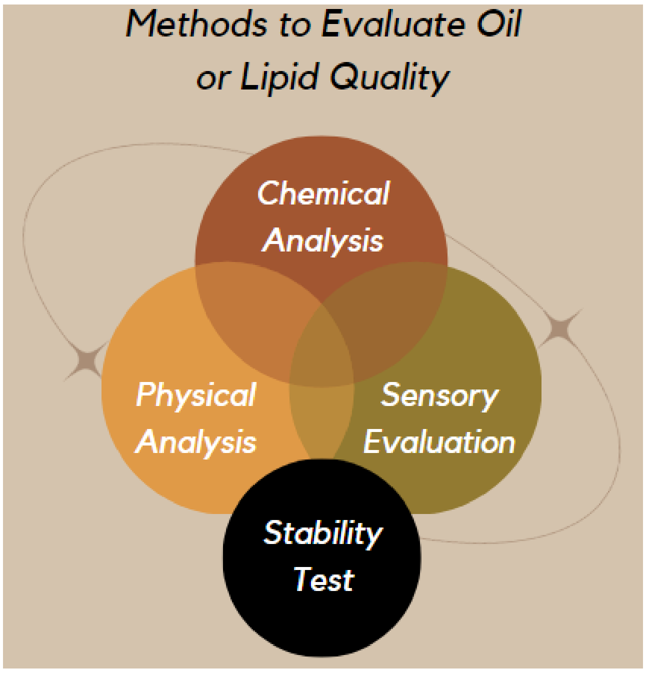

Figure 2.11: The evaluation of oil and lipid quality involves a comprehensive approach that combines chemical, physical, sensory stability test analyses

3 Conclusion

In this review, fundamental understanding regarding oil and lipid extraction was provided, and the benefits, drawbacks and operational principles of conventional and modern extraction methods were assessed. In conclusion, the extraction of fat and oil is an essential procedure that affects a variety of companies, demanding the use of suitable extraction techniques to produce high-quality products which adhere to Halal and Toyyib principles. The chosen technique must be in line with the raw materials, yield objectives, and environmental considerations whether it uses mechanical or solvent-based procedures. Through chemical, physical, and sensory investigations, rigorous quality evaluations guarantee adherence to industry standards and consumer preferences. Innovative strategies like waste utilization and green extraction techniques are becoming more popular as sustainability gains significance. This dynamic environment emphasizes the significance of effective, ecologically responsible extraction to satisfy the needs of developing industries and provide high-quality products.

4 Publisher's Note

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Author's Detail

Amal A. M. Elgharbawy[1,2*], Sharifah Shahira Syed Putra[3,4]

[1]International Institute for Halal Research and Training (INHART), International Islamic University Malaysia, P.O Box 10, 50728 Kuala Lumpur, Malaysia
[2]Bioenvironmental Engineering Research Centre (BERC), Department of Biotechnology Engineering, Faculty of Engineering, International Islamic University Malaysia (IIUM), P. O. Box 10, 50728, Kuala Lumpur, Malaysia
[3]Department of Chemical Engineering, Faculty of Engineering, Universiti Malaya, Kuala Lumpur 50603, Malaysia
[4]Faculty of Applied Sciences, Universiti Teknologi Mara (Perak), Tapah Campus, Tapah Road, 35400 Tapah, Perak, Malaysia.

*Corresponding author

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