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Recent studies on optimization of oil encapsulation process for spray drying: A comprehensive review

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Abstract

Background: Encapsulation of oils is a critical technology with widespread applications in food, pharmaceuticals, and other industries, enhancing stability, bioavailability, and controlled release of active ingredients. Spray drying is extensively used to preserve encapsulated oil in powder form. The major concern is to obtain high quality oil powders, which requires optimization of spray drying conditions. The physicochemical and microstructural characteristics of the product are influenced by several spray drying parameters, including high drying temperatures that destroy delicate bioactive components and carrier agents that protect such compounds.

Scope and approach: The review systematically examines various aspects of the oil encapsulation process, including drying technique, and optimization of spray drying conditions. Spray drying is one of the techniques used extensively for encapsulation of oil compared to other drying techniques and it is also used under optimal processing conditions to obtain powders. Several new techniques have been introduced into spray drying, helping to overcome its limitations. These techniques are posted to set new standards for the production of high-quality oil powders in industrial applications.

Key findings and conclusions: The most significant factors in spray drying are inlet air temperature and carrier agent. Successful spray drying enhances the physical properties of powdered products while preserving bioactive compounds with the help of carrier agents or their combinations. Response surface methodology is the most suitable tool for the optimization of spray drying conditions. This review summarizes current trends of the spray drying of oils, covering principles of spray drying, physicochemical and microstructural effects of spray drying conditions, optimization of spray drying conditions and new developments in spray drying.

Keywords: Oils, Encapsulation, Spray drying, Inlet/outlet air temperature, Wall material and Optimization

Introduction

The consumption of functional and healthy food products has been growing in the human diet. Vegetable oils are one of the effective and industrial substances that are used for the fortification of foods to produce functional foods. In particular, there is a growing demand for nutritious and healthy oils in the food, pharmaceutical, and cosmetic industries due to their multi-functional properties (Mohammed *et al*., 2020) [29]. However, processing may lead the ingredients to oxidise, which may result in several negative impacts like the appearance of unwanted organoleptic characteristics as such off flavours and changed texture a decrease in nutritional value, and a shorter shelf life. Several approaches have been developed to increase the stability of oil powder form as compared with its liquid counterpart throughout handling, processing and storage. The flexibility of powder form facilitates advanced formulations and opens new markets. In addition, the production of highly nutritional and regularly micro-structured oil powders may meet the requirements in the pharmaceutical and cosmetic industries.

Encapsulation is a promising approach that is widely used to overcome the above-mentioned problems by protecting the core materials from heat, light and oxygen, thus promoting stability, increasing bioavailability, flavour-masking, and controlled release, while maintaining the oils' functional properties and increasing their ease of handling (Fang *et al*., 2010). Encapsulation is the process of trapping one material inside another, resulting in particles that range in diameter from a few nm to a few mm (Mohammed *et al*., 2020) [29].

A variety of drying techniques are available for use on the industrial scale. According to (Aguiar *et al*., 2020) [3] the most successful method used for oil powder production is spray drying. Spray drying process of encapsulation ensures rapid, continuous drying, is economical and flexible, uses readily available equipment and produces powdery spherical particles of good quality (Prince, 2011). It provides a large surface area in the form of fine liquid droplets through atomization in the drying chamber, which leads to the production of regularly and spherically shaped powder particles (Fazaeli, *et al*., 2012). The quality of the final product depends on the conditions of spray drying process, which include feed concentration, inlet and outlet air temperature, feed flow rate, compressor air flow rate, drying air flow rate, type of atomizer and atomizer speed (Goula and Adamopoulos, 2010) [21]. Recently, the demand for oil powders has considerably increased due to multiple benefits of the application of these products in varieties of food formulations. Hence, it is important to know how the spray drying technique and its processing factors influence the powder properties and how to optimize the suitable ranges of processing factors. Only a few literature reviews on these studies are however available, in which the principles and complications of spray drying and the effect of spray drying factors on the properties of oil powders are discussed (Prince, 2011). Especially recent research updates about the advances of spray drying of oils are missing, which may be useful to perform further research. To obtain certain quality in powdered product, it is also a prerequisite to find out the optimum conditions of spray drying factors. Therefore, more research is needed on optimization of spray drying of oils to understand the suitable optimum conditions for specific kinds of samples. Furthermore, limitations of spray drying, such as loss of heat sensitive compounds, broad distribution of particle size and sometimes irregular microstructure have recently been addressed. Therefore, this review integrates advances in spray drying of encapsulation of oils, process optimization for suitable spray drying conditions and new trends of spray drying to overcome the limitations of conventional spray drying, which might be more beneficial for further research in the field of spray drying of encapsulated oils products.

1. Oil encapsulation process

The principle of microencapsulation is to create a physical barrier between the active compounds, adverse environmental conditions and the food matrix. In addition, this technique can enable the controlled release of the encapsulated core when certain conditions are met. Moreover, encapsulation is widely used in the food industry to incorporate oil aromas in a spray-dried form, because it is inexpensive, flexible, can be used in continuous operation, and produces particles of good quality (Fang *et al.*, 2010). The process can be conducted by changing the slurry emulsion from a liquid form into a powder in a continuously operating procedure. The basic principle of this method is to dissolve the core/wall materials in water to prepare an emulsion in liquid form, and then to feed this emulsion into a hot medium (100–300 °C) to evaporate the water. The final dried product can be collected in powder form or as agglomerated particles, depending on the nature of the materials used in the feed, the design of the dryer's operation and the operating conditions. The high temperature of the drying chamber facilitates the water evaporation from the droplets.

2. Spray drying as a process for encapsulation

Spray drying, an economical and flexible process, is the method commonly used for microencapsulation, which converts liquids into powders with easier handling, storage and transportation and makes its uniform mixing in food formulations easier. Equipment is readily available and production costs are lower than most other methods. Compared to freeze-drying the cost of spray-drying method is 30–50 times cheaper.

3.1. Importance of spray drying

Spray drying is regarded as the most economic drying technique due to its low operational cost. Also, it is a process widely used for microencapsulation of oils and flavours. Santivarangkna (2016) [38] reported that spray drying is eight times more economic than freeze drying and four times more economic than vacuum drying. Moreover, spray drying includes the benefits of relatively short drying contact time (5-100 s), and therefore tends to preserve sensitive quality attributes, for example nutrients, colors and flavors. The spray-dried product is highly stable, because of its low moisture content and water activity. It results in powders with good quality, low water activity, easier handling and storage and also protects the active material against undesirable reactions. Under these conditions, the powdered products are rather resistant to microbiological, oxidative degradation and other enzymatic activities. Hence, the technique is extensively applied to produce oil powders in food and even in pharmaceutical industries.

Limitation of spray drying

Even though, spray drying has a short drying contact time, it involves relatively high drying temperatures, typically 150-220°C inlet air temperature and 50-80 °C outlet air temperature (Phisut, 2012) [33]. The products to be spray dried can be categorized into two major groups: non-sticky and sticky products. Sticky products are generally difficult to use in the spray-drying process. During the drying process, they may remain as syrup, stick on the dryer wall or form unwanted agglomerates in the dryer chamber and conveying system, which causes lower product yields and operating problems (Sonone *et al.*, 2016) [43]. The main limitation of the spray drying technique in microencapsulation is the limited number of wall materials available and that must have a good solubility in water (Gharsallaoui and Roudaut, 2007) [20].

3.3. Unit operation of spray drying

Spray drying consists of three steps: atomization of feed sample, drying of liquid droplets and powder recovery. During atomization, liquid feed is passed through an atomizer to the drying chamber and distributed into tiny liquid particles in a large volume. The atomization maximizes the surface volume area of liquid feed for effective and efficient drying. The properties of the final product depend on the atomizer design and its performance (Phisut, 2012) [33]. The most common commercial atomizers are rotary atomizers, pressure nozzles and twin-fluid nozzles used in industrial application. Pressure or twin-fluid nozzles create tiny droplets with larger volume distribution than rotary atomizers. Hence, these are preferred in applications aiming at a small particle size. The atomized droplets and hot air interact in a drying chamber. The hot air increases droplet's temperature, which leads to the increase of water evaporation of tiny droplets. A dry layer develops on the droplet surface, while the moisture content of the droplet reaches the critical point. The resulting powdered particles might be spherical or oval shaped with smooth or rough crust depending on product characteristics and drying conditions (Phisut, 2012 [33]; Tonon *et al.*, 2008) [46]. Hot air blow in the drying chamber can flow in several directions, such as co-current, counter-current and mixed flow direction. The co-current direction is preferable for heat sensitive compounds, in which feed is passed through atomization following the route of drying air (150 °C-220 °C) flow and the final powder is exposed to moderate temperature of 50°C-80°C, which limits the thermal degradation (Gharsallaoui and Roudaut, 2007) [20]. After the drying is finished, the dried particles are detached from the humid air through cyclone and recovered at the end of the cyclone equipped with collection bottle.

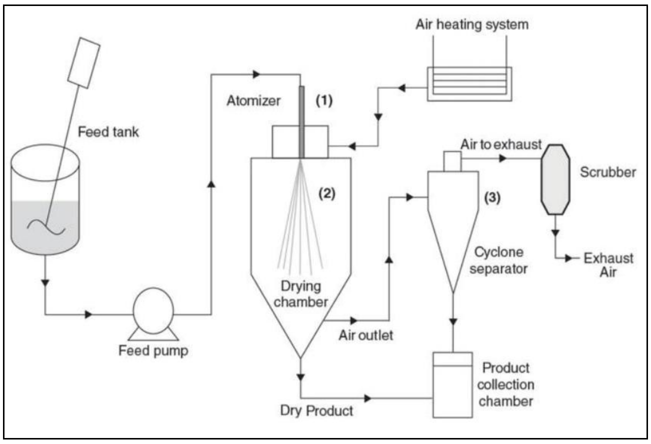

Fig 1: Schematic representation of spray-drying mechanism

3. Spray drying parameters of encapsulated oil powder

The quality of encapsulated oil powders depends on processing factors of spray drying. The most important factors of spray drying are drying temperature, drying air flow rate, feed flow rate, speed of atomizer, sort of carrier agent and concentration of carrier agent (Phisut, 2012; Tonon *et al*., 2008) [46].

Inlet and outlet temperature

The temperature of spray drying chamber is designated inlet air or drying temperature. Drying temperature is the most important factor affecting physicochemical properties of spray-dried powder. To obtain a final product with a high yield and degree of encapsulation, the inlet and outlet air temperatures should be optimized, and the feed emulsion should be stable throughout the processing time. In general, the inlet air temperature is in the range of 150–220℃ and evaporation occurs instantaneously. The low air inlet temperature results in a low evaporation rate, which leads to microcapsules with high-density membranes, high moisture content, low fluidity and ease of agglomeration. Therefore, the particles will easily stick to the internal wall of the drying chamber, resulting in a low yield. However, too high an inlet temperature results in extreme vaporization and membrane cracks may occur, and subsequently premature release and degradation or loss of encapsulated cores (Gharsallaoui and Roudaut, 2007) [20]. According to Andrea (2018) [5] moisture increased and hygroscopicity decreased when higher feed flow and lower temperature were used. The effect of inlet air temperature on powder morphology, when the inlet air temperature was low (130°C), most of the particles showed a shrivelled surface, while increasing drying temperatures (202°C) resulted in a larger number of particles with rigid smooth surface and lower shrinkage. Also, air inlet temperature is directly proportional to the microcapsule drying rate and the final water content. When the inlet air temperature is low, the low evaporation rate causes the formation of microcapsules with high density membranes, high water content, poor fluidity, and easiness of agglomeration. However, a high inlet air temperature causes an excessive evaporation and results in cracks in the membrane inducing subsequent premature release and a degradation of encapsulated ingredient or also a loss of volatiles. The inlet air temperature is usually determined by two factors: the temperature which can safely be used without damaging the product or creating operating hazards and the comparative cost of heat sources (Wang *et al*., 2018).

The temperature at the end of the drying zone, also called in literature as exhaust temperature or air outlet temperature, obtained under given conditions can be considered as the control index of the dryer. It is quite difficult to predict this outlet temperature in advance for a given product, since it depends on the drying characteristics of the material. Contrary to the air inlet temperature, the air outlet one cannot be directly controlled since it depends on the inlet air temperature and the ideal air outlet temperature for the microencapsulation of food ingredients such as flavors has been reported to be 50–80℃ (Gharsallaoui and Roudaut, 2007) [20].

Table 1: Recently optimized condition of inlet and outlet air temperature for the encapsulation of some different oil by spray-drying

Sr. No	Core material	Wall material	Inlet/Outlet temperature	References
1.	Palm fibre	Gum Arabic	130–202℃	Samsu *et al.*, 2020 [37]
2.	Sour cherry oil	Maltodextrin + gum Arabic	120–220 ℃	*Basyigit et al.*, 2020 [9]
3.	Walnut oil	SMP + Tween 80	180 ℃	Shamaei *et al.*, 2017 [39]
4.	Almond oil	Isolated starch	145 ℃	*Hoyos et al.*, 2019 [25]
5.	Palm Fibre Oil	Gum Arabic	166 ℃	Carmona et *al.*, 2018 [11]
6.	Citronella oil	Gum Arabic	136–203 ℃	Yingngam *et al.*,2019 [50]
7.	Pomegranate Seed Oil	Xanthan gum + gum Arabic	170℃/85℃	Yekdane *et al.*, 2019 [49]
8.	Lavender oil	Maltodextrin + gum Arabic	140℃/95 ℃	Burhan *et al.*, 2019 [10]
9.	Sesame oil	MG, MD: DE 10	120, 140 and 160 ℃	Onsaard *et al.*, 2018 [32]
10.	Sunflower seed oil	MD	180 ℃	Roccia *et al.*, 2014 [36]
11.	Fish oil	WP	160 ℃	Aquilani *et al.*, 2018 [6]
12.	Soya oil	Sodium caseinate, carbohydrate	180℃, 95℃	Hogan *et al.*, 2001 [24]
13.	Shrimp oil	WPC	180 ℃/90 ℃	Benjakul *et al., 2017*
14.	Annatto seed oil	WPI, modified starch	170 ℃	Silva *et al.*, 2016 [41]
15.	Garden cress seed oil	WPC	165℃	Yenge, 2018
16.	Coffee oil	Gum Arabic	166℃	Frascareli *et al.*, 2012 [17]
17.	Ginger oil	Inulin + whey protein isolate	140 ℃,155 ℃ and 170 ℃	Fernandes *et al.*,2017 [16]
18.	Rapeseed oil	Soy protein isolate + Maltodextrin	140–220 ℃	Linke *et al.*, 2019
19.	Rice bran oil	Jackfruit seed starch + whey protein isolate	140, 150 and 160℃	Murali *et al.*, 2017 [30]
20.	Gac peel oil	Whey protein + gum Arabic	160℃	Chuyen *et al.*, 2019 [13]
21.	Echium oil	Gum Arabic	150 ℃	Comunian *et al.*, 2019 [14]
22.	Nigella sativa oil	Maltodextrin + gum Arabic	160 ℃/88℃	Edris *et al.*, 2016 [15]

Selection of wall material

Spray drying of oil has challenging issues, such as stickiness, wall deposition and low yield. These issues are attributed to the presence of sugars and organic acids rich compounds. The choice of wall material for encapsulation by spray-drying is very important for encapsulation efficiency and microcapsule stability. The wall system is designed to protect core material from factors that may cause its deterioration, to prevent a premature interaction between the core material and other ingredients, to limit volatile losses, and also to allow controlled or sustained release under desired conditions (Shahidi and Han, 1993). Different types of wall materials used in spray drying of oils such as carbohydrates, gums and proteins.

Table 2: Wall materials commonly used for encapsulation in the spray drying process

Sr. No.	Wall Material	Properties	Reference
1.	Maltodextrin (DE < 20)	Film forming	Mohammed *et al., 2020* [29]
2.	Gum Arabic	Emulsifier, Good emulsifier	
3.	Whey protein	Good emulsifier	
4.	Modified starch	Very good emulsifier	

Carbohydrate is considered as good encapsulating agents because they exhibit low viscosities at high solids contents and good solubility, but most of them lack the interfacial properties required for high microencapsulation efficiency and are generally associated with other encapsulating materials such as proteins or gums. Gums are used in microencapsulation for both their film forming and emulsion stabilization properties. Gum arabic is usually preferred because it produces stable emulsions with most oil over a wide pH range, and it also forms a visible film at the oil interface. Because of this emulsifying efficiency, gum arabic has been usually used to encapsulate lipids. The excellent functional properties of proteins allow them to be a good coating material for the microencapsulation by spray-drying. In addition, proteins possess high binding properties for the flavor compounds (Mohammed *et al.*, *2020)* [29]

Table 3: Effect of different wall material on quality properties of encapsulated oils powder

Sr. No	Sample	Wall material	Effect of wall material on powder		References
			Increase	Decrease	
1.	Palm fibre oil	Maltodextrin	Bulk density, Tapped density	Moisture content, Solubility, Yield	Samsu *et al., 2020* [37]
2.	Fish oil	Whey protein, Maltodextrin and Gum arabic	Encapsulation efficiency, Moisture content	Peroxide value, Surface oil	Aquilani *et al., 2018* [6]
3.	Flaxseed oil	Maltodextrin	Encapsulation efficiency, oxidation stability	Lipid oxidation, Moisture content, Water activity	Sharif *et al., 2017* [40]
4.	Chia seed oil	Sodium caseinate, lactose, or Maltodextrin	Dispersibility, Encapsulation efficiency, oxidation stability	Moisture content, Water activity	Medina *et al., 2018* [28]
5.	Sunflower oil	Maltodextrin	Yield	Peroxide value, Surface oil, Moisture content	Roccia *et al*.,2014 [36]
6.	camellia seed oil	Whey protein, Maltodextrin	Encapsulation efficiency, oxidation stability	Surface oil	Song *et al*., 2022 [42]
7.	Buriti oil	Chickpea protein (CP) and high-methoxyl pectin (HMP)	Encapsulation efficiency	Moisture content	Poliana *et al., 2019* [34]
8.	Virgin Coconut oil	Maltodextrin: Sodium caseinate	Encapsulation efficiency, Bulk density, Surface oil, antioxidant activity	Peroxide value, Moisture content	Hee *et al*., 2015 [22]
9.	Nigella sativa oil	Maltodextrin + Gum arabic and Sodium caseinate +Whey Protein	Viscosity, large droplet size, best solubility	Moisture content	Mohammed *et al.*, 2021
10.	Pumpkin Seed Oil	Maltodextrin+Gum Arabic+ Whey Protein	Encapsulation efficiency	Total oil content, Surface oil, Moisture content, Bulk density	Ogrodowska, *et al.*, 2017 [31]

1. Selection of suitable conditions for spray drying of oils

Lots of researches have been performed in order to find out the significant factors and suitable ranges of the significant factors in spray drying of oil. To meet the desired quality of a product, it is essential to find out the optimum condition, which will ensure process efficiency and product quality. Extensive research efforts have been devoted to the optimization of drying conditions, as the following section describes.

2. Optimization process

Optimization aims at improving performance of a process by adjusting its parameters. In past, optimization was performed by observing the effect of alteration of a single factor on an experimental response, while all other factors remained fixed, known as one-variable-at-a-time optimization. The drawback of this method was that a huge number of tests were required; interactive effects among the parameters were not studied and the effects of the parameters on the response were not fully described. In order to overcome these limitations, the optimization can be carried out by using multivariate statistic techniques, such as full factorial design, D-optimal design, response surface methodology and combined design. Response surface methodology (RSM) is a multivariate technique, which is mostly used in optimization process involving two or more variables for statistical observation and analysis (Chelladurai *et al.,* 2021) [12].

6.1 Response surface methodology (RSM) and its application on spray drying of oils

Response surface methodology (RSM) is a collection of statistical techniques for facilitating efficient optimization of processes that depend on several variables and elucidation of interactions among these variables (Chelladurai *et al.,* 2021) [12]. The foremost benefit of the RSM is that the amount of data required for assessment, analysis and optimization considerably decreases the total number of tests needed and explores the response surface with equal precision in an efficient way. Central Composite Design (CCD) and Box Behnken Design (BBD) are the most recommended choice to researchers among RSM designs. RSM is commonly applied in the optimization of spray drying conditions for the production of oil powder. The most popular method of RSM in the optimization of spray drying conditions is central composite design (CCD). Inlet temperature, concentration of carrier agent and feed flow rate are the most important parameters. In case of spray drying of different oil, the most common range of inlet temperature is 120-180℃.The optimization of these parameters improves physicochemical characteristics of encapsulated oil powder.

Recent studies in spray drying

Spray drying is extensively used in food industry due to good physical properties of the products, short drying time and economic potential for scale-up. Spray drying is a process by which a liquid will be turned into dried particles by a hot drying gas medium. The spray-drying process reduces water activity of products, retards bacterial degradation and extends the product's shelf life. Nowadays, spray drying is widely used in pharmaceutical, food and chemical industries.

Table 4: Recent studies of encapsulated different oil using the spray drying technique in several functional food products

Sr. no.	Encapsulated oil	Product Name	Results	References
1.	Chia seed	Cookies	The highest sensory assessment scores were obtained when margarine was partially replaced with 15% weight percentage of microencapsulated chia seed oil.	Venturini *et al.,2019* [48]
2.	Garden cress seed	Biscuits	Biscuits (90 percent maida and 10 percent garden cress seed flour) and (90 percent wheat flour and 10 percent garden cress seed flour) with constant levels of other ingredients stored at ambient temperature had better acceptability till 90th day.	Gaikwad *et al., 2019* [18]
3.	Flaxseed oil	Bread	Breads fortified with microencapsulated flaxseed oil showed a reduced peroxide index, a greater value of α-linolenic acid, and helped preserve sensory attributes.	Helena *et al., 2012* [23]
4.	Rapeseed oil	Yogurt	Yoghurt matrix containing microcapsules had a 30-day stability period and a highly acceptable appearance.	Moura *et al., 2019*
5.	Fish oil	Soup powder	The acceptability of the microencapsulated fish oil improved soup powder has been shown by its high sensory acceptance score.	Kumar *et al.*, 2019 [26]
6.	Shrimp oil	Biscuits	Biscuits supplemented with 6% microencapsulated shrimp oil were stored in the dark to maintain their oxidative stability.	Takeungwongtrakul *et al.*, 2017 [45]
7.	Nigella sativa oil	Yoghurt	High thymoquinone stability as well as suitable chemical and sensory characteristics for yoghurt containing oil microcapsules from Nigella sativa seeds.	Abedi *et al., 2016* [1]
8.	Walnut oil	Bread	Compared to control, addition of encapsulated Walnut oil increased final product quality by lowering oil oxidation during storage period.	Akhtar *et al.*, 2022 [4]
9.	Nutmeg oil	biscuits	The encapsulated garden cress seed oil (GCO) was incorporated in biscuits to provide n-3 fatty acid rich biscuits.	Prince *et al.*, 2011 [35]
10	linseed oil	Bread	A formulation which can minimize α- Linolenic acid (ALA) loss after to bread preparation, quality assessment and sensory tests will be performed.	Gallardo *et al., 2013* [19]
11	Pomegranate seeds	pomegranate juice	Encapsulation with pomegranate juice was successful in improving the oxidative stability of pomegranate seed oil.	Yekdane *et al., 2019* [49]
12	Pomegranate seeds	Bread	Studies on the rheological qualities of the dough and breads made with 10% level of 0.15mm pomegranate seed powder (PSP) showed a slight decrease in volume, crumb hardness, and sensory scores as compared to the control group.	Aruna *et al.,2018* [7]
13	Walnut oil	Yoghurt	yoghurts with the addition of oil show a more favourable share of the examined groups of fatty acids (SFA, MUFA, and PUFA) and lower values of the analysed ones' fat quality indices.	Turek *et al.*, 2023 [47]

Conclusion

Spray drying is a well-established and extensively used technique in many products. Inlet air temperature of 120-180 ℃ and wall material concentration of 7-20% are the most common spray drying conditions for the production of oil powder. The products possessed excellent quality, i.e. low moisture, low hygroscopicity and small particle size; and high bulk density and high encapsulation efficiency. The protection of the bioactive compounds was improved by using carrier agents, such as maltodextrin, whey protein and gum arabic. In general, higher drying inlet temperature increased yield, solubility, hygroscopicity and particle size and reduced the moisture content, water activity and bioactive compounds. In contrast, addition of carrier agent enhanced protection of bioactive compounds, yield, solubility, bulk density with reduced moisture and hygroscopicity. Through meticulous experimentation, researchers have identified key parameters and methodologies to improve the encapsulation process, leading to advancements in various industries including food, pharmaceuticals, and cosmetics. The findings underscore the significance of factors such as emulsion stability, drying methods, and encapsulating agents in achieving optimal encapsulation efficiency and product quality. Overall, this comprehensive review illuminates the current state of knowledge in the field and sets the stage for future research directions aimed at unlocking the full potential of oil encapsulation for diverse applications. By continuing to refine techniques and explore novel approaches, researchers can contribute to the development of more efficient and sustainable encapsulation processes, ultimately benefiting industries and consumers alike.

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