

## EVOLUTIONS IN VIRGIN COCONUT OIL: LIQUID TO POWDER

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### Article history

Received

1 November 2020

Received in revised form

22 March 2021

Accepted

30 March 2021

Published online

22 April 2021

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### Graphical abstract



### Abstract

Awareness of virgin coconut oil (VCO) as a functional food supplement has grown enormously. The treatment of raw material and processing parameters is crucial as they affect the overall yield and quality of the oil. Meanwhile, the microencapsulation technology offers better handling, good oxidative stability, and longer shelf life of the VCO powder. Although good quality liquid and powdered oils can be obtained through sophisticated methods, the process involved is expensive and required high maintenance. By utilising dry kernel and coconut milk-based extractions methods, high-grade quality of VCO can be achieved. During microencapsulation, the spray drying method can preserve the phenolic and antioxidant compounds and maintain the low rancidity of the VCO. This paper evaluates the recent findings of the oil extraction methods and the development of VCO microencapsulation. These methods offer good quality, reproducible and economic viability.

Keywords: Virgin coconut oil, extraction method, microencapsulation, spray drying

### Abstrak

Kesedaran terhadap minyak kelapa dara (VCO) sebagai makanan tambahan berfungsi telah berkembang pesat. Perawatan bahan mentah dan parameter pemprosesan adalah penting dalam mempengaruhi hasil keseluruhan dan kualiti minyak. Sementara itu, teknologi mikroenkapsulasi menawarkan pengendalian yang lebih baik, kestabilan pengoksidaan yang baik dan jangka hayat serbuk VCO yang lebih lama. Walaupun kualiti minyak dalam bentuk cecair dan serbuk boleh diperolehi melalui kaedah-kaedah yang canggih, proses yang terlibat kebiasaanya lebih mahal dan memerlukan penyelenggaraan yang lebih tinggi. Dengan menggunakan kaedah pengekstrakan berasaskan isirung kering dan santan kelapa, VCO dengan kualiti yang baik akan diperolehi. Semasa mikroenkapsulasi, kaedah semburan kering mampu memelihara sebatian fenolik dan antioksidan dan mengekalkan paras ketengikan VCO yang rendah. Kajian ini menilai penemuan terbaharu mengenai kaedah pengekstrakan minyak dan perkembangan mikroenkapsulasi dalam VCO. Kaedah-kaedah ini memberikan kualiti yang baik, boleh dihasilkan semula dan ekonomi

Kata kunci: Minyak kelapa dara, kaedah pengekstrakan, mikroenkapsulasi, pengeringan sembur

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## 1.0 INTRODUCTION

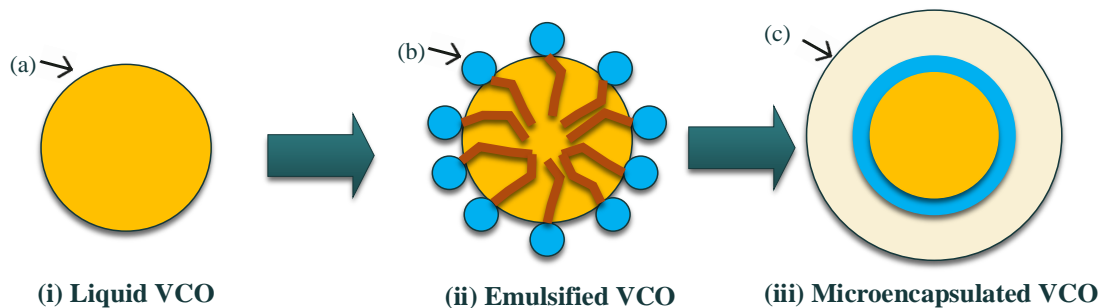
The trends for healthy food ingredients and products have increased due to people's awareness. As society becomes more affluent, people become more health-conscious and concerned about what they consume and the benefits they can derive from it. Coconut oil consistently flourishes in the pharmaceutical, nutraceutical, and cosmeceutical industries. Due to its wide range of applications, the global demand for coconut oil is growing significantly, and the situation has positively impacted economic value [1]. Since the introduction of VCO to local and international markets, the demands for coconuts increase exponentially. The global market for VCO stood at \$2.7 billion (2018) and is expected to expand at a CAGR of over 9%, reaching \$4.7 billion (2024) [2].

As VCO consists of high saturated fatty acids, its role in human health has always been misrepresented. It is noteworthy that not all saturated fats are associated with the risk of diet-related non - communicable diseases. The saturated fats are differentiated according to their biochemical, physicochemical, and physiological characteristics [3]. The high level of medium-chain saturated fatty acid (MCFA) present in VCO showed significant improvements in human health, such as the availability of dietary supplements for patients suffering from fat-absorption disease, anti-obesity treatment, and the reduction of total blood cholesterol [4].

The coconut oil derived from copra offers high recovery. However, it must be refined, bleached, and deodorised (RBD) to be suitably used in the market due to its unfavourable aroma and taste. However, several coconut producing countries emphasised that the RBD stage is unnecessary in VCO production [5]. This basis is because VCO must be processed from a fresh and mature kernel of coconut, technically by processing using natural means with or without the application of heat, without undergoing chemical refining, and so that it does not lead to the alteration of the natural state of the oil [6]. Several other extraction methods have been developed to produce a high degree of recovery with good quality VCO. The VCO processing is initiated either from coconut kernel or coconut milk. The dry method utilises the coconut kernel in the processing,

whereas coconut milk is used in the wet, integrated wet, cold-pressed, hot-processing, fermentation, and enzymatic processes [7]. All these methods contribute to different yield and quality of the oil produced.

In its original state, the oil-based form of VCO is insoluble in water and creates limitations in food application. On the other hand, the liquid form of VCO is also messier, less convenient, more likely to spill, and more difficult to transport than the powdered form of VCO. Furthermore, in the presence of moisture, oxygen, heat, and light, the oxidation of liquid oil during storage, the oil will turn rancid. This rancidity will reduce the presence of high free fatty acid and peroxide values and alter the oil to an unpleasant aroma and 'sour' taste. Coconut oil powder-based products are also available in the market, namely coconut creamer, medium-chain triglycerides (MCT) powder, and many others. The lingering question is whether all these products have the same characteristic as virgin coconut oil? Therefore, microencapsulated VCO in the powder form will mitigate these hurdles and offer a broader application in the product formulation than the liquid form VCO. Microencapsulation technology facilitates the challenge of providing unspoiled healthy ingredients into food without compromising its bioavailability and functionality, improving the shelf life of an encapsulated product, and increasing its thermostability and oxidative stability [8], [9]. The drying methods are crucial to obtain good powder characteristics, including spray-drying, spray-chilling, freeze-drying (lyophilisation), and fluidised bed coating [10], [11]. Figure 1 shows the development of VCO from liquid to powder. The active ingredient is diffused through the wall material [9]. Subsequently, the mixture of the active ingredient and the wall material is converted into an emulsion. The preparation of the emulsion preparation and dispersion homogeneity are essential in the oil microencapsulation technology, as the adsorption of liquid-liquid interfaces has a pronounced effect on the stability of the emulsion [8], [12], [13]. Spray-drying is a microencapsulation technique widely used in the food industry as it provides low cost and availability of the equipment [14]. This process yields VCO powdered form with low water activity, high quality, and good storage capability [15].



**Figure 1** Form of VCO. Symbol indicated: (a) oil drop, (b) emulsifying agent, and (c) wall material

This paper emphasises on:

- (1) The general definition of the processing methods involved in the production of VCO.
- (2) The general discussion on the yield and quality of the VCO.
- (3) The parameters involved during the microencapsulation process and the quality of the powder produced.

## 2.0 COCONUT OIL OBTENTION

Typically, coconut has a maximum of 75% oil content [16]. A production with a high oil recovery without compromising its quality is a significant concern in methodological strategy. By employing the drying method, 65% of oil can be extracted from the copra [7]. However, the copra needs a few months of drying (sun drying) or using high temperatures (smoke drying). Consequently, this method struggles with oxidative rancidity and aflatoxin contamination, eliminating its natural colour and aroma [17]. As a result, the RBD process is introduced to overcome this problem, resulting in a tasteless, odourless, and clear appearance [1]. The RBD process is not a practical approach in producing high-quality functional oils such as VCO, as it may degrade some of the nutrients [17], [18]. The RBD oil is not considered as VCO due to the usage of chemicals and high temperatures in the process [7].

### 2.1 Dry Method

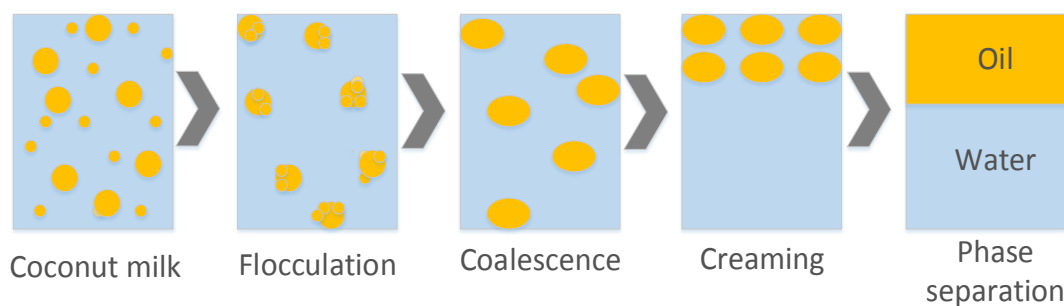
The dry method is when the coconut kernel was dried at 60°C before extraction and further dried at 65°C. The VCO yield obtained from this method was 25% which was relatively lower than RBD oil. However, it could preserve the minor compounds such as  $\alpha$ -tocopherol and polyphenols, reflecting the increased activity of antioxidant enzymes and inhibited lipid peroxidation [19], [20]. This outcome was expected when a lower temperature was used, and instant drying may preserve

the minor compounds in VCO compared to copra oil [21].

### 2.2 Wet Method

Another extraction method of VCO is the wet method which utilises coconut milk during its extraction process. The coconut meat is processed into coconut slurry and pressed using a cloth to obtain the coconut milk. The coconut milk is then refrigerated and heated to obtain the VCO [19]. As coconut milk produces stable emulsion, an external force is needed to break it. The emulsion stability was due to coconut proteins such as albumins, globulins, and phospholipids. These proteins are partially present in the aqueous process of coconut milk, which interacts with the fat globules and functions as an emulsifier by coating their surfaces [22].

Coconut milk can be destabilised using several mechanisms, such as cremation, flocculation, and coalescence. Figure 2 shows the destabilising mechanism of the emulsion. Creaming (or settling) is a separation phenomenon that occurred due to the differences in density between two phases due to the force of gravity [23]. The oil phase travels as clusters but does not separate the oil globules, and the arrangement of the particles remains intact. The weak attractive forces between the colloids produce flocculation behaviour [24]. Eventually, the coalescence-movement takes place slowly, and the interfacial oil globules are ruptured, allowing the globules to merge [25]. By cooling the emulsion at an appropriate temperature that exceeds the energy barrier, the emulsion continues to destabilise as fat crystals from the oil droplets behave as the nucleus, continually penetrates the neighbourhood droplet, causing partial coalescence. As a result, the emulsion will be destabilised [26]. Partial coalescence alters the oil-in-water phase (O/W) to the phase separation during the churning process [27]. The wet method also exhibited good preservation of the minor compounds [19]. However, the oil yield is relatively lower than the dry method since the oil/water separation efficiency contributes to the higher oil recovery [28].



**Figure 2** The de-emulsification mechanism of coconut milk

### 2.3 Integrated Wet Process

The integrated wet process combines cooling, freezing, thawing, and centrifugation procedures. Firstly, coconut milk will undergo the cooling process to break

down the oil/water emulsion at a temperature between 5–10°C [24], [29]–[31]. Better recovery of coconut oil is achieved at a lower cooling temperature. It is, therefore, essential to determine the correct cooling temperature for the processing of coconut oil [22].

Additionally, mechanical aid, such as agitation during the churning process, accelerates the de-emulsification of coconut milk [31]. The thawing process also destabilises coconut emulsion as it deformed the protein when heated [26]. During this process, the fat crystal coalesces and turns into the water-in-oil emulsion [32]. The subsequent centrifugation process is required to remove unwanted residues in coconut oil [33]. The quantity of oil yield from the integrated wet process was 85% higher than the fermentation method (without adding any bacteria or enzymes) but relatively lower than the dry method. The churning process of converting the coconut milk to butter has increased the water removal from coconut milk and may retain its nutty flavour and aroma [34].

## 2.4 Cold Process

The cold extraction involves cooling the coconut milk at low temperatures (2-8°C). The oil-water phase is then separated by the centrifugation and filtration of the oil [7]. Another method is to heat the coconut milk at 100–120°C to evaporate the water, followed by a decantation cycle to extract the oil. The VCO recovery in the hot condition is lower than the cold condition [22].

## 2.5 Fermentation Process

The fermentation method could be done by naturally separating the oil from water by fermenting the coconut milk, and, after some time, it will break into an emulsion [20], [35]. Microbial fermentation can separate the oil and other coconut milk emulsion elements such as protein [36]. In this method, the selection of bacteria plays a vital role in obtaining a high coconut oil recovery level. For instance, the *Lactobacillus planatarum* showed better emulsion breaking capacity than *Lactobacillus delbrueckii* due to its ability to multiply faster in coconut milk during the fermentation process. *Saccharomyces cerevisiae* also had been successfully used to extract the coconut oil. The bacteria release lipase and protease enzymes to break the coconut milk emulsion [37]. The maximum

yields of 21-37% were also recorded by the fermentation method [36],[38]. The enzymatic extraction is performed by adding enzymes in the coconut milk emulsion to expedite sedimentation/fermentation in the aqueous system. Using Viscozyme L and Neutralse 1.5 MG, 83% of the protein-oil emulsion was successfully achieved [39]. The use of  $\alpha$ -amylase, cellulase, protease, and polygalacturonase enzymes also contributed to quite a significant amount of oil yield (42%) [40]. The  $\alpha$ -amylase helped to hydrolyse the  $\alpha$ -linkages to liquefy the starch compound and produce maltose. Simultaneously, polygalacturonase hydrolyses the  $\alpha$ -linkages of polygalacturonic acid compounds from the end of the polymer, and the function of the protease enzyme is to hydrolyse the plant protein [41].

## 2.6 Fatty Acid and Triglyceride Composition in Virgin Coconut Oil

Saturated fatty acids can be differentiated based on physicochemical, physiological, and biochemical interpretive [3], [42]. Naturally, the medium-chain fatty acids (MCFAs) in VCO are bond together as medium-chain triglycerides (MCT) [43]. The MCT principally consists of caprylic acid/hexanoic acid (C6), caprylic acid/octanoic acid (C8), capric acid/decanoic acid (C10) and lauric acid/dodecanoic acid (C12) [44]–[48]. As the MCT is the primary triglyceride component, the MCFA terminology focuses on its physiological and metabolic functions. Proportionally, the MCFA comprises fatty acids from C6 to C12 [49]–[51]. In comparison, C14 (myristic acid/tetradecanoic acid) and above are known as long-chain fatty acids (LCFA) [44], [52]. The coconut oil consisting of more than 65% MCFA is also known as MCFA oil [3]. Thus, all studies [17], [20], [53]–[59] showed a presence of at least 55% of MCFA (Table 1), and this can be considered as MCFA oil. The variability of fatty acid content in coconut oil was due to the coconut variety, the sample preparation, the state of processing, and the processing method.

Table 1 Fatty acid composition in coconut oil

Method	C6:0	C8:0	C10:0	C12:0	C14:0	C16:0	C18:0	C18:1	C18:2
RBD [17]	0.63	8.24	6.53	47.42	18.26	9.33	2.68	5.25	1.57
RBD [53]	NM	7.24	5.25	50.90	21.38	9.22	0.38	4.81	0.81
Dry [54]	0.48	7.21	6.24	48.96	18.28	7.70	Total 6.56		3.26
Dry [55]	0.11	5.45	5.42	51.35	19.74	8.09	2.49	5.62	1.11
Cold-pressed [20]	0.70	6.67	5.58	48.18	20.40	8.78	3.71	5.75	1.10
Integrated wet [60]	0.40	6.05	5.77	50.93	19.40	8.17	2.82	5.23	1.10
Enzymatic [56]	0.52	6.63	5.49	46.36	19.54	9.94	3.37	6.50	1.63
Enzymatic [57]	NM	6.3	5.4	48.8	20.0	9.8	3.6	5.4	0.6
Fermentation [56]	0.57	7.21	6.07	48.42	18.75	9.06	3.15	6.35	1.36
Fermentation [58]	NM	9.7	6.4	49.2	19.5	7.0	2.3	4.7	1.1



## 2.7 Phenolic Compound in Virgin Coconut Oil

The presence of phenolic acid shows its antioxidant potential, allowing it to be used in the food and nutraceutical industries [61], [62]. The minor compound of phenolic acid exhibits antioxidant properties of antimutagenic, antiproliferative, and anticarcinogenic, benefiting human beings. In VCO, the phenolic compounds identified are ferulic, p-coumaric, sinapic, and caffeic acids [17], [63]. These phenolic compounds have exhibited strong antioxidant capability towards free radicals [64], [65]. The VCO produced by wet methods contains higher biologically active compounds such as polyphenol (84 mg GAE/100g oil), which reflected an increased antioxidant activity and inhibited the peroxidation of lipids compared to copra oil (64.4 mg/100g oil) [19]. The cold-pressing process indicated  $\alpha$ -tocopherol, phenolic compound, and antioxidant capacity (5.4 mg/100g, 57.8 mg GAE/100g, 0.48 mg GAE/ml).

Meanwhile, the fermentation process showed a lower concentration of compounds (4.7 mg/100g, 53.7 mg GAE/100g, 0.81 mg GAE/ml). The lower tocopherol suggested that a longer fermentation process caused the coconut oil oxidation during the process. Meanwhile, the lower phenolic compound was due to the phenolic polarity causing it to dissolve at the aqueous layer of the oil/water phase during the fermentation period, which lost during oil collection [20]. The phenolic compound concentration and antioxidant capacity were also higher in hot conditions at 100-120°C (44.9 mg GAE/100g, 0.05 mg GAE/ml) (44.9 mg GAE/100g, 0.05 mg GAE/ml) than in cold conditions at 10°C (6.6 mg GAE/100g, 0.03 mg GAE/ml oil). This circumstance could be due to the evaporation of water in the coconut milk during the process, which increased the phenolic concentration in the coconut oil [25], [66]. The integrated wet process showed the least amount of phenolic compound (20mg GAE/ 100g) but the highest antioxidant content (0.005 mg GAE/ml) [60].

From all the studies, the high antioxidant properties were not reflected by the high phenolic content in VCO, as it is subjected to the functional group in the phenolic compound. Therefore, the phenolics' concentration and consistency are essential for determining the antioxidant capacity in VCO [66]. Instead of variations in the type of processing, the differences in tocopherol, phenolic compounds, and antioxidants can be related to different processing parameters, preparation of samples, coconut variety used, storage time, and condition [60], [67], [68].

## 2.8 Oil Rancidity

Several parameters, such as peroxide value, acid value, ester value, and saponification value, are vital in

determining the oil's physicochemical properties [69]. The moisture content showed the amount of water remaining in the coconut oil. In compliance with the Malaysian Standards for virgin coconut oil, coconut oil's moisture content shall not exceed 0.15% [70]. Hydrolytic rancidity decreases the pleasant aroma and flavour of coconut oil and can be tested with a high free fatty acid content. The Malaysia Standards emphasised that free fatty acids must not exceed 0.5% [70]. The peroxide value shows the early stage of oxidation in the oil by reacting with oxygen in the atmosphere and producing hydroperoxide. The oxidation of unsaturated fatty acids (C18:1 and C18:2) is due to high temperature or long storage duration. The low amount of peroxide will ensure the freshness of the oil produced. From the Malaysia Standards, the peroxide value must be less than 3 meq/kg [70]. The iodine value indicates the degree of the unsaturation of the oil. High iodine value is undesirable due to the high rancidity of oil [71]. The Malaysia Standards also stated that the amount of 5.5-10.6 is acceptable for iodine value [70]. Saponification values stated the amount of short-chain fatty acids in the oil [53]. It is useful to determine the mean weight of acid, quantity, and type of glycerides and adulteration in oil [72]. The saponification value of 248-265 mgKOH/g oil is set for virgin coconut oil under Malaysia Standards [70].

From Table 2, the moisture content of all the methods is acceptable except for the centrifugation and fermentation methods. The speed of centrifugation and fermentation was suspected for the increasing oil moisture [40]. In the meantime, high levels of free fatty acid have been detected in RBD and copra oils. The high amount of free fatty acid may be caused by inadequate copra storage, high temperature, and high oil moisture content [53], [76]. The high moisture content due to the fermentation method also suggested a high free fatty acid value. This condition was influenced by changes in acidity due to the longer fermentation time [40]. Low peroxide and iodine values were also seen in all coconut oil production methods compared to the Malaysia Standards. The low peroxide does not help any oxidative degradation of the oil. Variation of findings for the same coconut oil production methods can also be observed, for example, in fermentation and enzymatic processes, the different uses of bacteria, enzymes, their concentrations resulting from the differences of hydrolysis reactions to discharge the water [75]. All the methods discussed also showed that the saponification values fell within Malaysia Standards, indicating good oil purity [72].

**Table 2** The physicochemical properties of coconut oil

Method	Moisture content (%)	Free fatty Acid (%)	Peroxide value (mequiv. oxygen/kg)	Iodine value	Saponification value (mg KOH/g oil)
RBD [53]	-	0.530	0.000	6.00	255.90
RBD [71]	0.01-0.10	0.01-0.08	0.27-0.8	6.81-8.91	-
Copra [71]	0.08-0.14	0.66-2.50	0.72-2.77	6.61-7.31	-
Copra [56]	0.040	0.46	-	4.18	258.42
Chilling and thawing [40]	0.15	0.08	0.43	-	261.00
Centrifugation [40]	0.34	0.17	0.34	-	250.00
Fermentation [73]	-	0.15	0.78	7.26	262.77
Fermentation [74]	0.30	0.22	2.540	-	-
Fermentation [35]	0.013	0.04	2.550	3.10	-
Enzymatic [41]	0.110	0.05	0.017	8.30	261.00
Enzymatic [57]	0.110	0.23	0.200	4.13	258.30
Enzymatic [75]	0.06-0.12	0.34-0.40	0.01	2.41-2.44	-
Enzymatic [40]	0.39	3.28	0.43	-	259

### 3.0 MICROENCAPSULATION OF VCO

Generally, the technique for making microcapsules involves four main steps. (i) Selecting the best encapsulating material. The materials used for encapsulation should be food-grade quality, has good emulsifying properties, low viscosity, and good film creator [77]. (ii) The homogenisation of the emulsion consists of the target component and the supporting materials of the wall materials. Therefore, the optimisation of the emulsion parameters is necessary to obtain adequate and stable droplets of oil [78]. (iii) Hot air was inserted into the atomised particle through the nozzle or spinning wheels. The atomisation is crucial as it significantly affects the chemical composition of the particle's surface in the spray drying process. (iv) Collection and air-tightening the produced powder [79]. Such steps are beneficial during the encapsulation of sensitive ingredients such as oil, as they contain a high amount of nutritionally valuable components, including antioxidants, bioactive phenolics, and essential fatty acids. Note that the enhancement of encapsulation efficiency is another critical objective in encapsulation technology. The efficiency reflects the presence of an oil phase on the surface finished powder particles, as shown by the wall material's ability to prevent leakage of the inner oil phase due to the leaching process [80].

In the previous study, the VCO was successfully encapsulated using respective spray drying and supercritical carbon dioxide spray drying (SC-CO<sub>2</sub>) [81]–[84]. The ability to protect the antioxidant properties and provide less rancidity of the VCO, like liquid form, is also desired during the microencapsulation process. As

there were limited studies on VCO microencapsulation, a few related research are discussed here to understand VCO powder's microencapsulation better.

#### 3.1 Selection of Encapsulation Materials

The preparation of the emulsion is the initial step that must be taken before the drying process. The emulsion must have good homogeneity, provides stability and low viscosity to ensure the oil droplets is protected by the emulsifying film and can be dried correctly during spray drying. Therefore, selecting the wall materials is crucial as it alters the emulsion properties and microencapsulation performance. The wall material, also referred to as a coating material, is made of natural or modified polysaccharides, proteins, sugars, gums, lipids, and synthetic polymers [85], [86]. It is expected to protect the bioactive compounds from environmental degradation. The requirements for the wall material used must be food-grade, and the addition of wall material leads to higher stability of the oil - in - water emulsion [10]. For instance, maltodextrin, which originated from starch, is widely used as it is an inexpensive material, possessed high water-soluble and has low viscosity at high concentration [87]. Meanwhile, the addition of gum Arabic to sodium caseinate and maltodextrin mixture during emulsification can preserve better antioxidant capacity for about 80% in the liquid VCO but lower encapsulation efficiency compared to without the addition of gum Arabic. The addition of gum Arabic preserved the volatile compound, increased the emulsion viscosity, enlarged the particle size, and increased the surface oil that leads to the oxidation of the oil [81], [88]. Another option available

is the usage of agave inulin as wall materials which offers a reduction in surfactant used and provides a healthier benefit [88].

Due to its drawback of poor surface active of wall materials, the incorporation of emulsifiers such as sodium caseinate, whey protein concentrate, gelatine, or surfactant could reduce the interfacial tension, therefore, stabilised the binding properties to the oil [88]. For non-ionic surfactants such as Tweens and Spans, both can reduce the interfacial tension between the two-immiscible oil/water system and the droplets' size [89]. The surfactant is an amphiphile that has an equilibrium in lipophilic and hydrophilic states. Surfactants are molecules that can form micelles. The lipophilic portion of the surfactant connects to the interior, and the hydrophilic part is facing the aqueous solution. The hydrophilic-lipophilic balance (HLB) is a predictive approach to the emulsifying characteristics for emulsifiers except for its efficiency, ranging from 0-20, specifically for non-ionic surfactants [90]. Different surfactants originating from different chemical groups have different polarities, contributing to the different wetting capacity [91]. For example, Tween 80 (HLB 15.0) showed a better emulsifying capability compared to Tween 20 (HLB 16.7) and Tween 40 (HLB 15.6) at a ratio of oil to the water of 2:8 in the VCO emulsion [92]. Therefore, the combination of wall materials and emulsifying agents have increased the stability of an emulsion. For example, maltodextrin, 3.5% sodium caseinate, and 3.5% of gelatine showed good stability during VCO microencapsulation [81]. For other coconut-based products, the introduction of guar gum (1%) and Tween 60 (1%) was seen to improve the stability of the emulsion and thus reduced the size of the droplets when coconut milk powder was encapsulated using a spray dryer [93]. Good stability of the emulsion was also reported with the addition of Tween 20 (0.5%), which minimised the partial coalescence phenomenon in the O/W formulation of palm kernel and canola oils [94]. The positive correlation between wall materials usage and emulsifying agents with emulsion stability is also emphasised [95], [96].

Often, when excess wall material or surfactant is added to the emulsion, it can remain free in the aqueous phase and cause a decrease in the emulsion. This condition will result in the flocculation and coalescence of oil droplets. The excessive usage of surfactants can be measured by critical micelle concentration and critical coagulation concentration [97]. Excess emulsifier negatively affects micelles' development, which would later interfere with active ingredients such as phenolic, thereby altering the partitioning between the different phases of emulsion and the role of the active ingredients [88].

The encapsulation efficiencies can also be affected by the viscosity of the emulsion, which determines the flow resistance of the emulsion [88]. When an emulsion fluid moves through a surface, the heat transfer

between the hot air from the spray dryer and the emulsion creates the convective heat transfer. Therefore, the speed of the fluid is one of the essential parameters to hasten the drying of the particles. Higher viscosity reduces the speed of the emulsion movement [98].

The viscosity is significantly influenced by the concentration of an emulsion [99]. During the emulsion preparation, the total concentration of solid content in the emulsion significantly impacted the encapsulation's efficiency. It will be part of the wall structure supporting the microcapsules during the drying process [100]. Generally, the ratios of 1:1 to 1:10 of oil: wall material are required to ensure the encapsulation's efficiency [101]. For instance, the ratios of 1: 1.45 to 4.00 were used during the emulsification of VCO [81], [83]. Instead of the ratio mentioned, the emulsification must be supported with a good wall material to obtain a good microencapsulation efficiency. For example, the utilisation of gum Arabic (10-20% from total solid) increased the total solid content and yielded approximately 30% of the babassu coconut powder [102]. The increase was due to the low viscosity, high solubility, and the ability to retain volatile compounds that make gum Arabic suitable for wall material in encapsulation [103]. Conversely, the usage of maltodextrin (40-45% of total solid) in the formulation of encapsulated coconut milk yielded only 4.6% [104]. However, the ratio of each wall solid content and wall and core materials were relatively to give lesser impact during the encapsulation of VCO compared to the effect of emulsifying and spray drying processing conditions [83], [105].

### 3.2 Homogenisation

The observation of the homogenisation process is essential to determine a good encapsulation of VCO. The homogenisation process using mechanical force, such as homogeniser, was used to transform the two immiscible liquids into an emulsion. A smaller droplet size was desired to increase the emulsion's physical stability during homogenisation [106].

The type of homogeniser and homogenisation duration are crucial to obtain a monodispersed particle size [107]. Homogenizers, namely high shear disperser, rotor-stator, ultrasonic, and high-pressure, are commonly used to provide energy to deform droplets and increased the interfacial area [12]. Besides converting the liquids into an emulsion, homogenisation reduces the size of the emulsion [108]. The high-pressure usage up to 300 bar improvised the droplet size by more than 50% reduction compared to the lower pressure of 30-50 bar [12], [88]. In contrast, the homogenisation pressure (400-600 bars) was insignificant to the droplet size unless it increases the oil fraction's aroma [109]. Table 3 explained the effects of the formulation and homogenisation process on the droplet size produced.

**Table 3** Effect of homogenisation process in oil-in-water emulsion

Material	Homogenisation procedure	Formulation	HLB Values	Z-average(n m)	Average particle size (nm)	Observation
VCO [110]	Prehomogenized at 12,000 rpm, 2 minutes, followed by ultrasonication at 60% powder intensity \ for 3 minutes	2.5% consists of Tween 80 and soy lecithin	-	-47.5	608.00	Good entrapment of VCO droplets and good stability of the emulsion.
Clove oil [111]	Ultrasonication at 5 minutes, duty cycle at 0.75% and intensity at 208 W/cm <sup>2</sup>	5% consists of Tween 80 and Span 80	9	-	43.00	Good stability of nanoemulsion was achieved.
Poly(lactic acid) [112]	Double sonication to achieve double emulsion	5% polyvinyl alcohol,	-4.4	200	200	Low dispersity of nanodroplets was achieved.
Avocado oil [113]	Premix using magnetic stirring at 200 rpm for 10 minutes, followed by high-speed homogenising at 16,800 rpm for 15 minutes. Finally, sonication at 20 kHz for 20 minutes.	7.5-10% Tween 80	-	-26 to-59	103-249	Good oxidative stability of nanoemulsion was achieved.
Coconut oil [114]	Homogenised at 10,800 rpm for 5 minutes.	5% polyethylene glycol hydrogenated castor oil	-	-	162	Good stability and uniform size of nanoemulsion
VCO [115]	homogenization at 15,000 rpm for 4 minutes.	0.5-1% Tween 80 / Span 80, VCO/water ratio 80/20	8.58	-	-	Good oxidative stability of emulsion
Rice bran oil [116]	Mechanical homogenising at 12000 rpm for 3 minutes followed by high-pressure homogenising (HPH) at 1000 MPa for 3 cycles.	0.5-1.5% high methoxy pectin-zein, 20% rice bran oil	-	-	5000-6000 using mechanical homogenizer and 3000-4000 using HPH	Differences in homogeniser pressure were relatively not significant compared to rice bran oil concentration due to the high concentration of rice bran oil in the formulation. A good physical stability of emulsion was achieved.
Mixture of sunflower and olive oils [117]	Pre-mixed at 5000 rpm for 5 minutes using colloid mill (CM), followed by conventional HPH at 15 MPa or ultra-HPH at 200-300 MPa	5% sodium caseinate with mixture (75% sunflower oil, 25% olive oil)	-	-	5421-6820 (CM), 98-210 (ultra-HPH), and 572-597 (conventional HPH).	Better physical and oxidative stability in ultra-HPH compared to only colloid milling or colloid-milling with conventional HPH.
Soy protein isolate(SPI) [118]	HPH-homogenizing at 5-80 MPa	50% soy protein isolate, 50% soybean oil	-	-	-	More stable SPI gels were formed at higher pressure, which is desired to reduce the interfacial tension.
Mussel myofibrillar proteins (MMP) and lecithin [119]	Prehomogenized at 20000 rpm for 2 minutes, HPH-homogenizing at 40-120 MPa for 3 cycles	90% consist of soy lecithin and MMP, 10% soybean oil	-	10.7-37.0	440-460	The highest stability and best emulsifying activity were at 80 MPa.



### 3.3 Spray Drying Condition

The mechanism of spray drying is designed to remove moisture through a heated condition during feeding. The process involved atomisation, a transformation of droplets into particles, and the collection of the particles [120]. For example, the inlet temperature is vital to achieving good encapsulation efficiency. Increased inlet drying temperature and inlet flow rate reduced the moisture content of the encapsulated materials. This phenomenon occurred due to the higher heat and mass transfer of the inlet air, causing a higher evaporation rate of the particles [103]. The inlet temperature should be more than 140°C to obtain a good encapsulation [100]. Additionally, the maximum amount of 4% of moisture content is recommended for dried powder in the food industry [121].

The moisture of 3.19% exhibited good oxidative stability in the microencapsulation of spray-dried sesame oil [122]. The inlet temperature of 180°C, outlet temperature of 85°C, and the feed flow rate of 10 mL/min were used during the encapsulation of VCO, resulting in 2.54% of moisture content [81]. The results indicated that the parameters are substantial and adequate to produce a good VCO powder. In comparison, the treatment with 170–220°C inlet temperatures throughout the encapsulation of babassu coconut milk achieved moisture contents of 10.53–2.39% [102]. However, if the inlet temperature is too low, water evaporation becomes inefficient, resulting in wet powder and low encapsulation yield [123]. The higher moisture of 5.74% was achieved by oven drying at 50°C to produce coconut milk powder [124]. Additionally, the moisture content also impacted the powder's flowability, as the moisture content of 4% was required to produce a good flow characteristic of the coconut flour [125].

### 3.4 Powder Collection

In a spray dryer, the coarse dry particles are collected in the chamber attached to the spray dryer [120]. The remaining finer particles (less than 30%) are further conveyed to the cyclone separator or bag filter attached to the spray dryer. The cyclone separator more opts since it offers higher efficiency for powder entrapment [127].

Once the powder is collected, it must be kept under proper packaging to maintain its quality and prolong its shelf life. For instance, infant milk powder can easily oxidise, caking and browning is suitable to be packed with poly-olefin metal containers. It is better to purge the container with nitrogen or carbon dioxide gas to remove the remaining oxygen. Another method introduced is to use high gas resistant packaging, such as composite paper cans and flexible pouches. For any of these methods, it is best to keep the product under room temperature [128]. Meanwhile, the moisture content of coconut protein powder did not show a significant effect when stored at 4–38°C using metal polyester packaging. It exhibited that the polyester packaging can prevent oxygen, light and moisture

from penetrating inside the packaging [129]. Overall, it is better to keep the coconut powder at a lower temperature and under nitrogen gas treatment to avoid product deterioration [130].

### 3.5 Characterisation of Microencapsulated VCO

The characteristics that determine the quality of microencapsulated oil are solubility, bulk density, wettability, and other indicative measurements as required by a study. For VCO, the microencapsulation process's ability to preserve the minor compound and provide oxidative stability is essential to ensure the powdered VCO has a similar quality characteristic as in liquid form. During microencapsulation VCO using spray drying and SC-CO<sub>2</sub>, both methods demonstrated the ability to preserve phenolic compounds and antioxidant in VCO. The spray drying and supercritical carbon dioxide (SC-CO<sub>2</sub>) methods successfully retained the maximum of 1.13 (mmol BHT/ml oil) of phenolic content. However the SC-CO<sub>2</sub> achieved higher antioxidant capacity of (0.8–1.48 (mmol Trolox/ml oil) compared to spray drying method of 1.48 (mmol Trolox/ml) [81], [82]. Meanwhile, the peroxide value of VCO powder achieved was also relatively within the limit of Malaysia Standards of below 3meq/kg oil in the spray-drying method [81]. Comparatively, the SC-CO<sub>2</sub> system exhibited a better quality of the powder produced, but the equipment is more expensive than the spray dryer. Besides, spray-drying is more economical, versatile, and already proven in the food industry [123].

A spray-dryer application creates a layer of fat on its particle surface that can reduce its solubility and increase oxidation and stickiness, which degrades the product's quality. High powder solubility is required to ensure optimum functionality in the food system [78]. For instance, solubility lower than 65% was achieved using a spray dryer compared to a freeze-drying method that reached 80% solubility during coconut skim milk encapsulation. The variation in the powder's solubility was also shown in the different concentration of emulsion [126]. By using different concentrations of sodium caseinate and type of protein source, the different solubility was achieved in the freeze-dried creamer [127]. Other reports indicated the solubility difference from slightly soluble to very soluble resulting from different maltodextrin concentrations and skim milk during coconut powder encapsulation [128]. However, spray drying provided a better sensory effect but retained its moderately good functional properties compared to freeze-drying [126].

The characteristic of powdered oil, such as its high bulk density, significantly impacts the functional and economic assessment as it reduces the packaging and transportation costs [129]. Low bulk density is undesirable due to its positive correlation with oxidation and its stability [102]. The bulk density increased significantly by lowering the inlet drying temperature, and the aspiration rate from the outer layer of the droplet is rapidly dried. Higher bulk density can also be achieved by increasing the feed mass flow rate.

Inversely, the minimised drying temperature and increased feed flow rate reduce the powder's size and the encapsulation efficiency with a higher moisture content [130]. The 350 kg/m<sup>3</sup> bulk density of coconut protein powders was reported when the inlet temperature was 130°C and a flow rate of 75 ml/min [131]. Meanwhile, a bulk density of 850kg/m<sup>3</sup> was obtained when the inlet temperature was 160°C during Black seed oil encapsulation [132].

The low concentration of wall material also reduced the bulk density but adversely affected the spray-dried powder's particle density [133]. Various bulk densities (550-620 kg/m<sup>3</sup>) were shown when using different concentrations of sodium caseinate in the emulsion during the production of non-dairy creamer [127]. All these results suggested that adjustments in formulation and drying conditions would lead to variation in bulk density.

The interaction between bulk powder with water can be thermodynamically determined by wettability{Formatting Citation}. The dispersibility shows the quickness of the powder to dispersed in water through slow stirring [131]. Wettability is generally influenced by the size, size distribution, and particle shape of the powder [134]. The type of wall materials and the amount of total soluble solid used in the formulation also improved powder wettability. It noted that the wettability of 30-60 s is required to ensure a good dispersibility of the powder in the water [102], [135]. The wettability of 60 s with a dispersibility of 93% was achieved during microencapsulation of coconut skim milk powder. The particle size was 70 µm with irregular shapes and had no crack [131]. However, in coconut milk whey encapsulation, the wettability of (21-23s) with dispersibility (93-97%) was achieved. The particle size of 69.5-78.8 µm with no indication of the particle shape was reported [135]. Both studies indicated relatively similar particle sizes but differed in wettability. This divergence may be caused by the difference in spray drying processing parameters, affecting the final moisture content, wettability, and hygroscopicity of the powder [105]. By using different types of wall material, the whey protein isolates exhibited the highest wettability powder (420s), compared to the use of the mixture of whey protein isolate/maltodextrin (265s) and whey protein isolate/inulin (205s) during the encapsulation of novel structure lipids. The low wettability indicated that the particles are easily in contact with water, showing the ability to wet well. The whey protein isolate/inulin also had the lowest free oil value to reduce hydrophobicity and minimise particle surface blockage in contact with water [121].

#### 4.0 CONCLUSION

This overview reveals the benefit of VCO and the process involved during the extraction and microencapsulation of the oil. Copra oil practically produces a high oil recovery, but it undergoes the RBD

process, consequently eliminating some of the essential nutrients present in the coconut oil. Therefore, to alleviate this issue, several other attempts were studied to produce VCO without compromising its quality. Generally, all the methods had shown an adequate MCFA oil supply but differ in terms of the fatty acid concentration due to variation in raw material preparation, processing conditions, and methods used.

The evolution of VCO has been extended to the microencapsulation of its oil for better handling and storage capacity. The preparation of the emulsion and the processing parameters of the equipment must be considered to produce good quality microencapsulated VCO. The spray dryer's processing condition also contributes to the excellent quality of VCO powder, such as inlet temperature and flow rate.

#### Acknowledgement

The authors would like to thank all members of the Institute of Bioproduct Development (IBD), UTMLEAD and Grant Q.J130000.2851.00L39 for the encouragement and support.

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