



# Production of virgin coconut oil microcapsules from oil-in-water emulsion with supercritical carbon dioxide spray drying



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## ABSTRACT

Virgin coconut oil (VCO) has been shown to have health-promoting effects due to its high medium-chain fatty acid (MCFAs) content and antioxidant properties, however, its taste and feeling of greasiness are not acceptable to many consumers. In this study, VCO was microencapsulated to develop a new and healthy consumable food product. Spray drying VCO with supercritical carbon dioxide (SC-CO<sub>2</sub>) at pressures from 12 to 16 MPa, temperatures from 40 to 60 °C was studied for an emulsion feed flow rate that was varied from 3 to 5 mL/min. The encapsulation efficiencies ranged from 73% to 80%, the microcapsules were spherical with diameters that ranged from 27 to 72 μm and the antioxidant activities of the retained microencapsulated oil ranged from 0.6 to 1.0 mmol butylated hydroxytoluene (BHT) equivalent/ml oil and 0.8–1.3 mmol trolox equivalent/ml oil for 2,2-diphenyl-1-picrylhydrazyl (DPPH) tests and 2,2'-azino-bis (ABTS) tests, respectively. SC-CO<sub>2</sub> spray drying is an effective method to encapsulate VCO and other food oils.

## 1. Introduction

Virgin coconut oil (VCO) is obtained from the meat of coconut (*Cocos nucifera* L.) with physical or thermal methods that do not alter its nature [1]. VCO that is not refined, bleached or deodorized retains its nutritional value and distinctive aroma and taste. VCO has many potential health benefits due to its rich medium-chain fatty acids (MCFAs), vitamins and antioxidants. Lauric acid is the dominant fatty acid in VCO and contributes to its strong antiviral and antibacterial activity [2]. MCFAs of VCO can increase metabolism rates in the liver when ingested and can help prevent obesity [3]. VCO has a cardioprotective effect that is beneficial in lowering lipid components, increasing antioxidant enzymes and reducing lipid peroxide content due to its polyphenolic content [4,5]. However, many consumers do not accept the texture of VCO because it contains saturated fatty acids that are solid at room temperature. Therefore, liquid VCO is typically converted into a powder form via spray drying to give consumers a new perception of VCO as a food product [6].

The application of microencapsulation in the food industry includes providing a protective barrier to sensitive functional compounds, masking unpleasant tastes and smells and stabilizing and increasing the bioavailability of the bioactive compound [7]. Spray drying is the mostly broadly technique applied in oil microencapsulation, however,

it has several drawbacks, including the destruction of heat-sensitive compounds, large particle sizes, wide particle size distribution, low precipitation yields and difficulty in removing any solvents used [8–12].

There is lack of reported studies on the microencapsulation of VCO with SC-CO<sub>2</sub> particle formation methods. Spray drying with SC-CO<sub>2</sub> has the following advantages including mild operating temperatures (ca. 60 °C), small particle size and narrow particle size distribution, favorable yields for many products, it is safe and its removal is simple by depressurization [13]. In this work, the VCO was prepared as an oil-in-water emulsion and products were processed at 40–60 °C. The influence of operating conditions, namely, pressure, temperature and emulsion feed flow rate, on the physicochemical characteristics of the VCO microcapsules was investigated. Properties measured include encapsulation efficiency, particle size, particle size distribution, particle morphology, powder flowability, antioxidant activity and fatty acid composition.

## 2. Materials and methods

### 2.1. Materials

VCO was provided by Adirondack (M) Sdn. Bhd. (Petaling Jaya,

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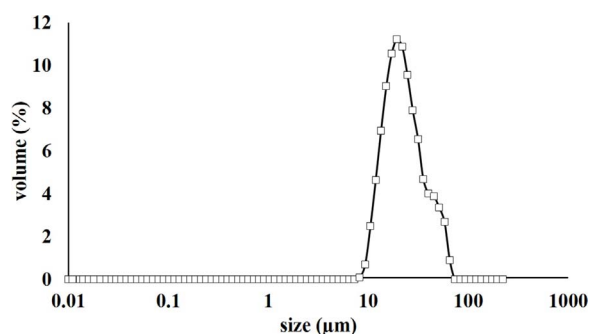
**Table 1**

Effect of operating variables on the encapsulation efficiency of virgin coconut oil microcapsules using supercritical carbon dioxide spray drying and characteristics of the resulting powder.<sup>a</sup>

Pressure (MPa)	Temperature (°C)	Emulsion feed flow rate <sup>b</sup> (mL/min)	Total oil (%)	Surface oil (%)	Encapsulation efficiency (%)	Particle size, D (4,3) (μm)	Moisture content (%)	Bulk density (g/cm <sup>3</sup> )	Compressibility index
12	50	4	36.1 ± 0.4	9.8 ± 0.2	72.7 ± 0.5	39.5 ± 13.8	4.1 ± 1.6	0.32 ± 0.04	14.5 ± 5.0
14	50	4	35.8 ± 0.6	9.4 ± 0.1	73.8 ± 0.5	59.9 ± 14.7	3.9 ± 0.6	0.32 ± 0.03	19.1 ± 3.3
16	50	4	37.5 ± 0.4	8.6 ± 0.2	77.0 ± 0.4	51.5 ± 16.2	2.9 ± 0.5	0.30 ± 0.03	14.6 ± 4.7
16	40	4	37.2 ± 0.3	7.5 ± 0.3	79.8 ± 0.8	48.3 ± 10.9	3.4 ± 0.2	0.31 ± 0.02	14.9 ± 2.0
16	60	4	34.9 ± 0.5	9.5 ± 0.2	72.9 ± 0.6	26.7 ± 10.8	4.0 ± 0.3	0.30 ± 0.01	9.9 ± 2.1
16	40	3	37.0 ± 0.4	7.3 ± 0.2	80.2 ± 0.6	66.9 ± 14.4	3.9 ± 0.4	0.31 ± 0.04	11.6 ± 1.7
16	40	5	37.0 ± 0.7	8.2 ± 0.2	77.8 ± 0.7	72.1 ± 13.8	3.7 ± 0.5	0.29 ± 0.02	12.7 ± 5.2
Spray dried VCO powder [6]			38.6	7.47	80.5	229.7	2.35	0.29	–

<sup>a</sup> Ratio of VCO to encapsulating agent was 2:3 where VCO loaded was 40% in dry weight. Data are indicated as means ± standard deviation.

<sup>b</sup> Flow rates at 27 °C and at the respective pressure.



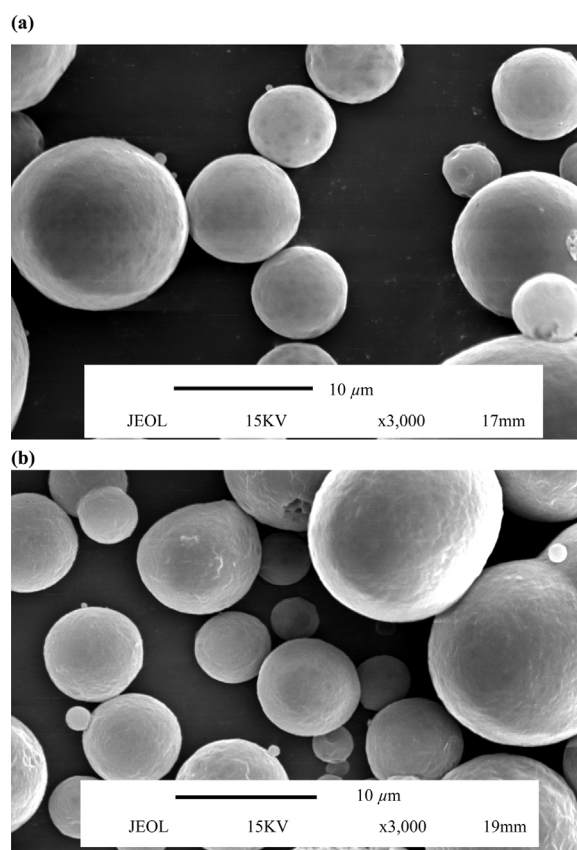
**Fig. 1.** Particle size distribution curve of virgin coconut oil microcapsules encapsulated by maltodextrin and sodium caseinate precipitated with supercritical carbon dioxide spray drying with supercritical CO<sub>2</sub> conditions at 16 MPa, 60 °C and 4 mL/min of emulsion feed flow rate at 27 °C and 16 MPa.

Malaysia). As the encapsulating materials, maltodextrin DE10 was purchased from Porrina (M) Sdn. Bhd. (Batu Caves, Malaysia), and sodium caseinate was purchased from V.I.S. Foodtech Ingredient Supplies Sdn. Bhd. (Sri Damansara, Malaysia). Soy lecithin was purchased from Se Scientific Supplies (Ampang, Malaysia). Carbon dioxide (CO<sub>2</sub>) with 99.9% purity was purchased from Mox-Linde Gases Sdn. Bhd. (Petaling Jaya, Malaysia). Reagent grade chemicals methanol (≥95%), hexane (≥95%), ammonia solution (≥95%), diethyl ether (≥95%), isopropanol (≥95%) and petroleum ether (≥95%), were purchased from Merck Sdn. Bhd. (Bandar Sunway, Malaysia) and used in the analyses. Reagents related to bioassays, 2,2-diphenyl-1-picrylhydrazyl (≥98%), butylated hydroxytoluene (≥99%), potassium persulfate (≥99%), trolox (97%), 2,2'-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) diammonium salt (≥98%) and fatty acid methyl ester (FAME) mix standards (C8-C22) were purchased from Sigma-Aldrich (M) Sdn. Bhd. (Petaling Jaya, Malaysia).

## 2.2. Particle formation with SC-CO<sub>2</sub> solvent

An oil-in-water emulsion was produced with the formulation described by Hee et al. [6]: reagents maltodextrin (13.0%), sodium caseinate (4.4%) and soy lecithin (1.0%) were dissolved in water (70.0%) and then blended with VCO (11.6%) using a blender (MX-800S, Panasonic, Petaling Jaya, Malaysia); the wall material and oil had a ratio of 60:40. The pre-homogenized emulsion was subjected to a high-pressure homogenizer (APV 1000, SPX Corporation, Albertslund, Denmark) at 18 MPa for seven cycles and was then used as feed in a SC-CO<sub>2</sub> spray unit.

The particle formation process was performed with an aqueous spraying unit (FeyeCon, Development & Implementation BV, Weesp, the Netherlands). The precipitation chamber (25 L) was set to the desired temperature; a high-pressure pump (NP16/14-210C, Speck-



**Fig. 2.** Morphology of virgin coconut oil microparticles precipitated with supercritical CO<sub>2</sub> conditions at (a) 16 MPa, 40 °C and 3 mL/min of emulsion feed flow rate (27 °C and 16 MPa) (b) 12 MPa, 50 °C and 4 mL/min of emulsion feed flow rate (27 °C and 12 MPa).

Kolbenpumpenfabrik, Geretsried, Germany) pumped the SC-CO<sub>2</sub> into the precipitation chamber through a full cone spray nozzle with an internal mix setup (PNR, Voghera, Italy) with a regulated volumetric flow rate. When the required conditions were achieved, the feed was delivered into the precipitation chamber by a syringe pump (260 D, Teledyne ISCO, Lincoln, USA) through the spray nozzle. SC-CO<sub>2</sub> was continuously fed into the vessel to sustain stable operation. Once the emulsion was depleted, the liquid pump was discontinued and CO<sub>2</sub> was allowed to flow through the chamber for 30 min to eliminate the remaining liquid, which was solubilized in SC-CO<sub>2</sub>. The gas within the precipitation chamber was slowly depressurized until it returned to atmospheric pressure. The powder was collected from a filter bag located in the precipitation vessel and immediately vacuum packed and stored at –2 °C until further characterization tests were performed.

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