Application Note
No. 302/2017

Protection of bioactive oils using encapsulation

Mini Spray Dryer B-290, Encapsulator B-390, Encapsulator B-395 Pro, Multivapor™ P-6 / P-12

1. Introduction

Bioactive oils are commonly used in food, pharmaceutical and cosmetic applications[1,2]. Those oils are usually rich in unsaturated and polyinsaturated fatty acids. The majority of fatty acids can be produced by the human body, however omega-3 and omega-6, essential in human nutrition, cannot be synthetized and must be provided by food[1]. Oils rich in unsaturatted fatty acids are therefore widely used in food preparation or for direct consumption. They are though reported to be chemically unstable and to deteriorate through mechanisms such as primary and secondary oxidation or hydrolytic rancidity when exposed to oxygen, light, temperature, moisture or transition metal ions[3]. Rancidity of edible oils and oil-based products through lipid oxidation is commonly mentionned as a serious problem in the food industry[4]. The oxidative stability of unsaturated oils is influenced by the degree of unsaturation, the position and the number of the polyunsaturated fatty acids[4]. In summary, the more double bonds and/or electron-donating groups, the easier it is for fatty acids to be oxydised[3].

The value of such bioactive oils does not only reside in their amount of unsaturatted fatty acids but also in the presence of other active compounds such as sterols, caroteinoids, xanthophylls, flavonols or monoterpenes[1]. These active compounds are reported to exhibit special properties such as anti-inflammatory, antioxidant, antibacterial, antiviral, anticancer or regenerative activity, however they are also quite fragile[1].

Encapsulation of bioactive oils in a convenient, suitable system is of potential interest. It represents a feasible and efficient approach to modulate oil release, increase physical stability, protect from oxidation reaction, enhance bioactivity, reduce toxicity, mask unpleasant taste and therefore improve patient compliance and convenience[3,5].

Several methods exist to extract oil for food industry, the most used being solvent extraction or mecanical extraction methods such as pressing or centrifugation. Regulation regarding hexane have led to new large scale design for methods such as supercritical CO_2 extraction [6,7]. Even though cold press and supercritical CO_2 extraction are possibly more suitable extraction methods to preserve omega-3, omega-6 and antioxydants, solvent extraction can still successfully be used and was chosen in this experiment for process conveniency.

In this study, two oils representing high linoleic (hemp) and high linolenic (flax) acid contents were respectively chosen for this study as they represent premium oil grown locally, together with a novel high linoleic acid containing oil extracted from chia seeds[2,4,8,9]. The objective of this work was to extract the oil from the seeds using a classical Soxhlet extraction method and to then encapsulate them to be protected from oxidation. A dry powder was produced by Spray Drying using the Mini Spray Dryer B-290 and wet particles by coextrusion technology using an Encapsulator B-395 Pro.

2. Equipment, Chemicals and Material

- Equipment
  - Retsch ZM200 Ultracentrifuge mill with 1mm sieve
  - Glass Soxhlet
  - BUCHI Chiller F-308
  - BUCHI Heating Bath B-305
  - BUCHI Multivapor™ P-6
  - Drying oven
  - BUCHI V-300 Vacuum pump with I-300 Pro controller
  - BUCHI Encapsulator B-395 Pro
  - BUCHI Mini Spray Dryer B-290

- Chemicals
  - n- Hexane, technical grade, VWR
  - Calcium chloride dihydrate, ACS reagent, 99 %, Sigma Aldrich
  - Sodium alginate low viscosity grade, BUCHI (11058394)
  - Maltodextrin DE 19
  - Gum Arabic, Sigma Aldrich

- Samples
  - Linseed, declared fat content 31 g/100g
  - Chia seed, declared fat content 33 g/100g
  - Hemp seed, declared fat content 32 g/100g

3. Experimental

The seeds were first ground into fine powder using an ultracentrifuge mill with the appropriate sieve (Retsch ZM200). After milling, the oil was extracted with a Soxhlet glassware using hexane (technical grade, VWR) at 68°C for 3 h. The oil was then recovered by evaporating the solvent on a Multivapor™ P-6 parallel evaporator acording to the method in Table 1, at 50°C with a rotation speed of 5.

Table 1: Evaporation method using the Multivapor™ P-6

Initial pressure	Final pressure	Time
Atmospheric pressure	350 mbar	As fast as possible
350 mbar	350 mbar	1 min
350 mbar	250 mbar	3 min
250 mbar	250 mbar	2 min
250 mbar	180 mbar	3 min
180 mbar	180 mbar	15 min
180 mbar	120 mbar	1 min
120 mbar	120 mbar	5 min

The residual solvent was evaporated in an oven at 100°C for 60 min after the extraction process.

After extraction the oil was encapsulated into a dry powder using the Mini Spray Dryer B-290. 10 g of Maltodextrin, 5 g of oil and 5 g of Arabic gum were mixed with 100 mL of water using a high sheer mixer in order to produce an emulsion. The emulsion was then fed to the spray dryer using the following parameters (Table 2).

Table 2: Spray drying parameters of the extracted oil.

Parameters	Value
Inlet temperature	150°C
Outlet temperature	80°C
Feed rate	15-25 %
Spray gas flow	35-40 mm
Aspirator	100 %
Set up	Open mode with 0.7/1.5 mm nozzle

For the production of wet particles, the Encapsulator B-395 Pro was used with the parameters in Table 3.

Table 3: Parameters for the encapsulation of the extracted oils with the concentric nozzle system.

Alginate concentration	2 %
Calcium chloride concentration	0.1 M
Shell nozzle	400 µm
Core nozzle	300 µm
Pressure	500 mbar
Frequency	680-720 Hz
Electrode	2500 V
Oil flow rate (syringe pump)	4-6 mL/min

4. Result and discussion

Oil was successfully extracted from linseed, chia seeds and hemp seeds using a Soxhlet and a Multivapor P-12 to evaporate the extraction solvent. Yields above 85 % were obtained compared to the theoretical declared fat content. Linseed oil yielded over 100 %, however, in a natural product, the real oil value can differ from theoretical value, which would influence yield results. The presence of residual solvent could also be considered to explain this high yield, however, it is quite unlikely due to further evaporation step in the oven.

Table 4: Oil extraction results

Oil	Theoretical oil amount[a] [g_{oil}/100 g_{seeds}]	Recovered oil amount [g_{oil}/100 g_{seeds}]	Yield [%]
Linseed	31	39.2	126
Hemp	32	28.8	87
Chia	33	30.8	93

[a]according to the declared value of the food producer

The extracted oils were successfully encapsulated using both the Mini Spray Dryer B-290 and the Encapsulator B-395 Pro. Alginate core-shell capsules were successfully obtained using the parameters in Table 3. Capsules of approximately 800 µm were obtained with an oil loading of roughly 50 % (Figure 1). With Spray Drying, a dry powder composed of particles with sizes between 1 µm and 15 µm was obtained (Figure 2). The oil loading of the dry particles was 25%.

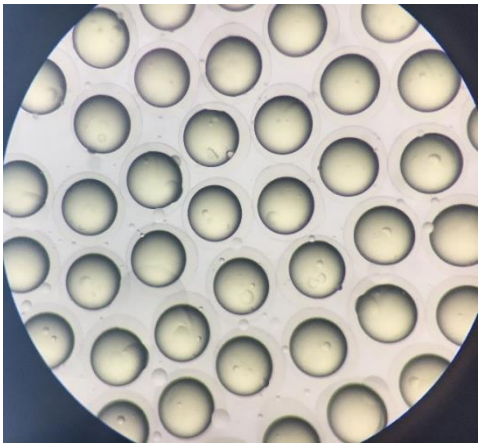

Figure 2: Linseed oil core capsules with oil loading of approximately 50 %.

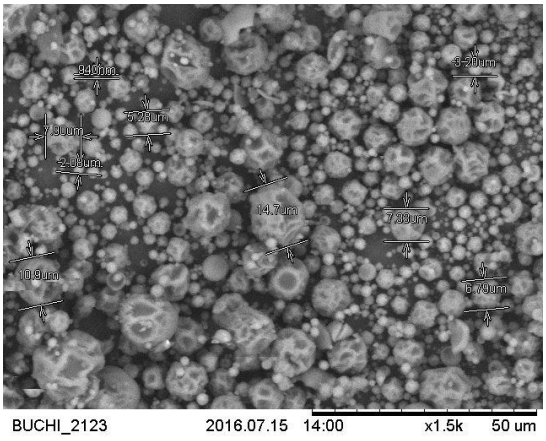

Figure 2 : Powder obtained after spray drying an emulsion of oil, maltodextrin and gum arabic (1:2:1)

5. Conclusion

The combination of BUCHI instruments allowed the successful extraction and encapsulation of bioactive oil. The Multivapor™ enabled a quick and efficient evaporation of the solvent after the Soxhlet extraction process while the Mini Spray Dryer B-290 and the Encapsulator B-395 Pro allowed a mild encapsulation process of the extracted oil. Conventional, classical polymers have been used for both Encapsulator and Spray Dryer processes, however one could imagine to adapt encapsulation process using other carbohydrate or protein based polymers; oil core capsules could be produced in a similar way with the Encapsulator using other hydrophilic polymers such as agar or gelatin for example.
For oil determination and quality control BUCHI also offers several analytical extraction solutions; amongst them the Extraction Unit E-816 SOX or the SpeedExtractor E-916. These instruments could be used to extract oil in order to determine fat content and fat composition such as the content of omega fatty acids from seeds, intermediate and final products.
More information about fat determination from seeds and BUCHI extraction unit for fat determination can be found in the application note (308/2017).

6. References

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