biology and life sciences forum

MDPI

Proceeding Paper

Chia Oil Microcapsules Obtained by Different Drying Methods †

Claudia N. Copado [1], Vanesa Y. Ixtaina [1,2] and Mabel C. Tomás [1,*]

[1] Centro de Investigación y Desarrollo en Criotecnología de Alimentos (CIDCA), CCT La Plata (CONICET), Facultad de Ciencias Exactas (FCE), Universidad Nacional de La Plata (UNLP), 47 y 116, La Plata 1900, Argentina; claudiacopado16@gmail.com (C.N.C.); vanesaix@hotmail.com (V.Y.I.)
[2] Facultad de Ciencias Agrarias y Forestales (FCAyF, UNLP), 60 y 119, La Plata 1900, Argentina
* Correspondence: mabtom@hotmail.com
† Presented at the III Conference la ValSe-Food and VI Symposium Chia-Link Network, Online, 15–17 November 2021.

Abstract: A technology used to protect chia oil from lipid oxidation during processing and storage is microencapsulation. Thus, microcapsules containing chia oil could be applied as an ingredient to develop enriched foods with ω-3 fatty acids. The objective of this technology is to achieve high microencapsulation efficiency and provide greater oxidative stability to the chia oil. This work compares microcapsules obtained by different methods such as spray-drying and freeze-drying. To establish relationships between the microencapsulated chia oil using both methodologies and some of the characterization parameters studied, a multivariate analysis was carried out considering the microcapsules obtained from the parental emulsions with 10 or 15% *w*/*w* of chia oil, 10% *w*/*w* of lactose, and 10% *w*/*w* of sodium caseinate, whose aqueous phases were or not heat-treated at 60 or 100 °C, 30 min. The results show that the main components 1 (CP1) and 2 (CP2) explain 46.7 and 38.1% of the observed variability, respectively, totaling around 85%. The CP1 allowed separation of the microcapsules obtained by spray-drying from the freeze-drying ones, while the CP2 permitted to discriminate within the chia oil microencapsulated by freeze-drying, the systems whose aqueous phases were treated or not at 100 °C, 30 min from the rest of the microcapsules. The multivariate analysis made it possible to differentiate the microcapsules obtained by spray-drying and the freeze-drying ones. The former being associated with greater luminosity and microencapsulation efficiency, as well as a lower level of moisture content, water activity, and b* (blue-yellow component of the CIELab system) values.

Keywords: chia oil; freeze-dryer; microcapsules; spray-dryer

Citation: Copado, C.N.; Ixtaina, V.Y.; Tomás, M.C. Chia Oil Microcapsules Obtained by Different Drying Methods. *Biol. Life Sci. Forum* **2021**, *8*, 10. https://doi.org/10.3390/blsf2021008010

Academic Editors: Loreto Muñoz and Claudia M. Haros

Published: 25 January 2022

1. Introduction

The growing knowledge available about the relationship between food and health has prompted various actions in order to achieve an improvement in the composition of the diet through the incorporation of functional compounds. This reality has led the food industry to concentrate more and more on the development of functional foods [1].

The consumption of polyunsaturated fatty acids (PUFAs) ω-3 offers various health benefits; for example, protection against coronary heart disease, inflammatory disorders, asthma, and retinal diseases, as well as an improvement in brain function. Thus, the incorporation of these compounds into the diet is very important [2].

Oils from vegetable sources are good sources of essential fatty acids and other fatty acids derived from them. Regarding the lipid fraction of green leafy plants and some seeds, a relevant component is α-linolenic acid (ALA) [3]. Specifically, the oil content of chia seeds is about 25–40% *w*/*w*, with ALA (~65%) and linoleic acid (LA) (~20%) as the most abundant fatty acids, and a low percentage of saturated fatty acids (SFA) [4]. The high content of ω-3 fatty acids gives chia oil a high nutritional value. However, the unsaturation

degree of PUFAs is responsible for the great susceptibility of this oil to lipid oxidation [5]. It is, therefore, essential to use some strategies such as microencapsulation to guard this oil during storage and/or processing. Among the different microencapsulation processes, spray-drying and freeze-drying have been applied to microencapsulate different types of oils. The features of the obtained microparticles depend on the microencapsulation process, the wall material, the wall/core ratio, and the characteristics of the parent emulsions before the drying process.

The Maillard reaction is a series of complex chemical reactions between the reducing end of a carbohydrate and the free amino group of a protein, accelerated under heat treatments [6]. Different studies found that Maillard reaction products (MRPs) offer the proteins a better emulsifying capability, foaming, heat stability, and solubility, than the native form. Additionally, MRPs have antioxidant activity, mainly due to the compounds formed from the Amadori rearrangement [6,7]. Thus, MRPs can be used as wall material to microencapsulate chia seed oil.

The objective of this study was to characterize the chia oil microcapsules obtained by spray drying and freeze-drying, utilizing MRPs generated from heat treatment of sodium caseinate and lactose as wall material, and to analyze the data by a multivariate statistical method verifying its ability to distinguish groups of microparticles.

2. Materials and Methods

2.1. Experimental Design and Preparation of Microcapsules

The experimental design proposed was selected for evaluating the influence of the oil content (10 or 15 g/100 g emulsion) and the aqueous phase heat treatment to stimulate the MRPs production. Different O/W emulsions were formulated with sodium caseinate (NaCas) (10% *w*/*w*), lactose (10% *w*/*w*) and two chia oil concentrations (10 and 15% *w*/*w*) (Table 1). The NaCas was mixed in distilled water (50 °C) under magnetic agitation. Afterward, the lactose was incorporated in the aqueous phase. The protein–carbohydrate mixture was heated (60 or 100 °C, 30 min) in a water bath. Nisine (0.0012 g/100 g) and potassium sorbate (0.1 g/100 g) were used for microbial growth inhibition. The emulsion was prepared in two stages. The pre-emulsification was carried out by Ultra Turrax T 25 equipment (IKA Labortechnik, Germany), using a S 25 N - 18 G dispersing tool (1 min, 9500 rpm). Then, the pre-emulsion was homogenized with a Panda 2K high-pressure valve homogenizer (GEA Niro Soavi, Parma, Italy) at 600 bars, for 4 cycles. The microcapsules were obtained by drying the emulsions in a Mini Spray Dryer B-290 (Büchi, Switzerland), using 170 °C for the inlet air and 75 °C for the outlet one, or in a freeze-dryer (L-A-B4-C, Rificor, Buenos Aires, Argentina). For this last process, the O/W emulsions (~100 g) were placed in 125 mm × 160 mm plastic trays, forming a ~1 cm thick layer and frozen at −20 ± 1 °C, 48 h, and then were transferred to −80 ± 1 °C for 24 h. Microcapsules were then obtained from the frozen emulsions by freeze-drying in laboratory-scale equipment using 1 atm of vacuum press for 48 h.

2.2. Moisture Content

This parameter was measured gravimetrically in a vacuum oven (Instrumentación Científica S.A., Buenos Aires, Argentina) according to Baik et al. (2004) [8].

2.3. Water Activity (a_w)

The a_w was determined using an AquaLab Water Activity Meter CX2 model (Decagon Devices Inc., Pullman, WA, USA) at 25 ± 0.5 °C.

Table 1. Experimental design of the microencapsulation of chia seed oil using two types of drying (spray-drying and freeze-drying).

Microencapsulation Process	System Code	% Oil (g/100 g of Parent Emulsion)	Oil Content (g/100 g of Microcapsules)	Lactose Content (%*w*/*w*)	NaCas Content (%*w*/*w*)	Heat Treatment
Spray-drying	STT-10 SD	10	33.0	10	10	-
	STT-15 SD	15	42.9			
	TT60-10 SD	10	33.0	10	10	60 °C, 30 min
	TT60-15 SD	15	42.9			
	TT100-10 SD	10	33.0	10	10	100 °C, 30 min
	TT100-15 SD	15	42.9			
Freeze-drying	STT-10 FD	10	33.0	10	10	-
	STT-15 FD	15	42.9			
	TT60-10 FD	10	33.0	10	10	60 °C, 30 min
	TT60-15 FD	15	42.9			
	TT100-10 FD	10	33.0	10	10	100 °C, 30 min
	TT100-15 SD	15	42.9			

NaCas, sodium caseinate.

2.4. Microencapsulation Efficiency (ME)

The free oil was quantified according to the method proposed by Augustin et al. [9] with some modifications. Approximately 1 g of powder was used, which was washed with hexane through a filter paper (Whatman N° 4). The difference of weight in the samples allowed calculation of the free oil concentration. The total oil of the microcapsules was considered equal to the initial oil. ME was calculated according to Equation (1).

$$\mathrm{EM}(\%) = \frac{(\text{total oil} - \text{free oil})}{\text{total oil}} \times 100 \quad (1)$$

2.5. Color

The surface color of the microcapsules was measured using a Minolta color analyzer (CR-400, Konica Minolta Sensing Inc., Japan). This colorimeter was calibrated with a white standard, and color was evaluated using the CieLab system (L*, a*, and b*) [10].

2.6. Statistical Analysis

A multivariate analysis (PCA principal component analysis) was performed considering the microcapsules obtained by both drying methods from systems with the same formulation. This analysis was carried out using the Software MultiVariateStatistical Package (MVSP) version 3.1 (Kovach Computing Services, Wales, UK).

3. Results and Discussion

In order to establish relationships between the microencapsulated chia oil using both methodologies and some of the characterization parameters studied, a multivariate analysis was carried out considering the microcapsules obtained from the parental emulsions with 10 or 15% *w*/*w* of chia oil, 10% *w*/*w* of lactose whose aqueous phases were or not thermally treated at 60 or 100 °C, 30 min. The results obtained for each parameter studied are shown in the Tables 2 and 3, and Figure 1 represents the result of principal component analysis (PCA).

Table 2. Parameters of the microcapsules obtained by spray-drying.

Microcapsules	H (% d.b.)	a_w (25 °C)	ME (%)	Color		
				L*	a*	b*
STT-10	1.34	0.52	83.96	93.52	−1.01	12.81
STT-15	0.74	0.48	74.67	92.21	−1.01	14.06
TT60-10	1.44	0.48	77.21	91.57	−1.01	15.05
TT60-15	2.23	0.49	72.58	93.42	−0.98	14.02
TT100-10	4.55	0.41	98.68	87.09	0.94	18.16
TT100-15	2.06	0.30	97.49	84.95	1.35	19.54

H% (d.b.) moisture content; a_w water activity at 25 °C; ME microencapsulation efficiency. Mean values (n = 2). The coefficients of variation were less than 10%.

Table 3. Parameters of the microcapsules obtained by freeze–drying.

Microcapsules	H (% d.b.)	a_w (25 °C)	ME (%)	Color		
				L*	a*	b*
STT-10	0.02	0.37×10^{-3}	99.38	97.96	−0.67	6.59
STT-15	1.33	0.41×10^{-3}	99.03	97.96	−0.57	7.86
TT60-10	0.09	0.29×10^{-3}	99.04	98.47	−0.62	5.58
TT60-15	3.00	0.47×10^{-3}	99.59	97.92	−0.40	7.31
TT100-10	0.04	0.24×10^{-3}	99.06	97.67	−0.08	7.40
TT100-15	0.03	0.33×10^{-3}	98.79	96.44	−0.49	11.32

H% (d.b.) moisture content; a_w water activity at 25 °C; ME microencapsulation efficiency. Mean values (n = 2). The coefficients of variation were less than 10%.

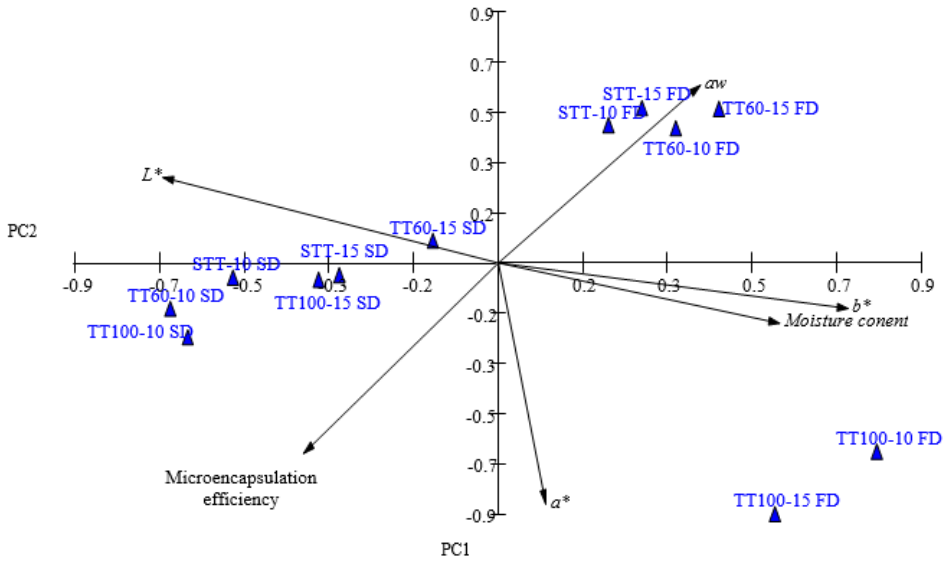

Figure 1. Principal component analysis (PCA) of chia oil microcapsules obtained from spray–drying or freeze–drying (see system codes in Table 1).

The principal components (PC) 1 and 2 explain 46.7 and 38.1% of the observed variability, respectively, totaling around 85%. As can be seen, PC1 allowed separation of the microcapsules obtained by spray-drying from those freeze-dried, while PC2 allowed discrimination within the microencapsulated chia oil by freeze-drying, the systems whose aqueous phases were treated at 100 °C, 30 min of the rest of the microcapsules. Thus, taking PC1 into account, the powders obtained by spray-drying appeared associated with the highest microencapsulation efficiencies and greatest luminosity. On the other hand, microencapsulated oils by freeze-drying were associated with higher moisture levels, water activity, and the color parameter b* (more yellowish). Regarding PC2, the microcapsules obtained by freeze-drying from parental emulsions whose aqueous phases received the most

intense heat treatment were associated with a higher a* value, a higher microencapsulation efficiency, and a lower level of a_w.

4. Conclusions

The multivariate analysis made it possible to differentiate the microcapsules obtained by spray-drying from the freeze-drying ones, presenting the former ones greater luminosity and microencapsulation efficiency, and lower moisture content, aqueous activity, and b* values.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Acknowledgments: This work was supported by the grants Ia ValSe-Food-CYTED (119RT0567), Spain, Universidad Nacional de La Plata (UNLP) (11/X907), and the Agencia Nacional de Promoción Científica y Tecnológica (PICT 2016-0323), Argentina.

References

1. Eratte, D.; Dowling, K.; Barrow, C.J.; Adhikari, B. Recent advances in the microencapsulation of omega-3 oil and probiotic bacteria through complex coacervation: A review. *Trends Food Sci. Technol.* **2018**, *71*, 121–131. [CrossRef]
2. Kaushik, P.; Dowling, K.; Barrow, C.J.; Adhikari, B. Microencapsulation of omega-3 fatty acids: A review of microencapsulation and characterization methods. *J. Funct. Foods* **2015**, *19*, 868–881. [CrossRef]
3. Lane, K.E.; Derbyshire, E.J. Omega-3 fatty acids—A review of existing and innovative delivery methods. *Crit. Rev. Food Sci. Nutr.* **2017**, *58*, 62–69. [CrossRef]
4. Ixtaina, V.Y.; Martínez, M.L.; Spotorno, V.; Mateo, C.M.; Maestri, D.M.; Diehl, B.W.K.; Nolasco, S.M.; Tomás, M.C. Characterization of chia seed oils obtained by pressing and solvent extraction. *J. Food Compos. Anal.* **2011**, *24*, 166–174. [CrossRef]
5. Drusch, S.; Serfert, Y.; Scampicchio, M.; Schmidt-Hansberg, B.; Schwarz, K. Impact of Physicochemical Characteristics on the Oxidative Stability of Fish Oil Microencapsulated by Spray-Drying. *J. Agric. Food Chem.* **2007**, *55*, 11044–11051. [CrossRef]
6. Lee, Y.Y.; Tang, T.-K.; Phuah, E.-T.; Alitheen, N.B.M.; Tan, C.P.; Lai, O.-M. New functionalities of Maillard reaction products as emulsifiers and encapsulating agents, and the processing parameters: a brief review. *J. Sci. Food Agric.* **2016**, *97*, 1379–1385. [CrossRef]
7. Waller, G.; Feather, M. *The Maillard Reaction in Foods and Nutrition*; Proc. ACS Symposium Series; American Chemical Society: Las Vegas, NV, USA, 1983; ISBN 08-412-07690.
8. Baik, M.-Y.; Suhendro, E.L.; Nawar, W.W.; McClements, D.; Decker, E.; Chinachoti, P. Effects of antioxidants and humidity on the oxidative stability of microencapsulated fish oil. *J. Am. Oil Chem. Soc.* **2004**, *81*, 355–360. [CrossRef]
9. Augustin, M.; Bhail, S.; Cheng, L.; Shen, Z.; Øiseth, S.; Sanguansri, L. Use of whole buttermilk for microencapsulation of omega-3 oils. *J. Funct. Foods* **2015**, *19*, 859–867. [CrossRef]
10. Augustin, M.A.; Sanguansri, L.; Bode, O. Maillard Reaction Products as Encapsulants for Fish Oil Powders. *J. Food Sci.* **2006**, *71*, E25–E32. [CrossRef]