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EVALUATION OF FEED FLOW RATE ON THE PHYSICOCHEMICAL PROPERTIES OF FISH OIL MICROCAPSULES

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Abstract

Fish oil is one of the sources of fatty acids and plays a significant role in maintaining a healthy lifestyle. Regular intake of fish oil can prevent cardiovascular-related disease and improve the development of infants' and young children's brain functions. However, fish oil is highly prone to oxidative deterioration, leading to higher shelf-stability reduction. Microencapsulation by spray drying technology offers a better solution by protecting the oil from further oxidation, enabling it to be delivered to food products without affecting its properties. The present study focuses on the physicochemical properties of fish oil microcapsules by spray drying techniques with different feed flow rates. The feed flow rate studied was 280 mL/h to 444 mL/h, combining maltodextrin and whey protein isolates as biopolymers. The physicochemical properties evaluated were moisture content, particle size distribution, free fatty acid, acid value and encapsulation efficiency. This work aimed to identify the most suitable feed flow rate based on the evaluated physicochemical properties. It was observed that the moisture content and particle size distribution were increased from 3.15 ± 0.01 % to 3.54 ± 0.06 % and 27.621 ± 0.320 µm to 50.636 ± 1.321 µm from the feed flow rate of 280 mL/h to 444 mL/h, respectively. The fish oil microcapsules produced using a feed flow rate of 280 mL/h recorded the highest free fatty acid, acid value and encapsulation efficiency of 5.11 ± 0.101 %, 2.82 ± 0.004 mg (KOH/g) and 80.89 ± 0.231 %. Thus, it can be suggested that spray drying with a low feed flow rate of 280 mL/h can produce fish oil microcapsules with a lower moisture content, particle size distribution, free fatty acid, and acid value with higher encapsulation efficiency.

Keywords: Feed flow rate, Fish oil, Oxidative stability, Spray drying.

1. Introduction

Fish oil acquired from oily fish tissues such as salmon, eel, anchovies, cod liver and mackerel are typically rich in essential fatty acids vital to human health [1]. For the past few decades, there has been a massive clinical interest and growing recognition of fish oil's capabilities towards supporting human health and preventing chronic diseases such as heart attack, hypertension and atherosclerosis [2]. It is not only limited to preventing chronic diseases but is also significantly beneficial in treating skin conditions such as psoriasis [3] and occupational dermatitis [4]. Due to its extensive prophylactic and therapeutic ability, there was a growing demand towards fish oil-based products worldwide. Consequently, commercialising stable and good fish oil-based products remains a key challenge. Fish oil is known for its low stability and solubility, mainly in most food classifications and is critically prone to oxidation. According to Tonon et al. [5], fatty acid oxidation deteriorates the quality of the fish oil end products and significantly leads to unpleasant tastes and odours, thus reducing shelf-life.

Microencapsulation has been practised in countless industries, such as pharmaceutical, cosmetics and most prominently in food industries. This technique is extensively applied to stabilise the bioactive contents and enable product delivery without impacting its taste and shelf-life [2]. In addition, microencapsulation aids in protecting, isolating, releasing, and transporting the constituted bioactive materials [6, 7]. Microencapsulation methods include emulsification, spray drying, freeze-drying, coacervation and extrusion [8, 9]. Among all available techniques, spray drying is one of the most practised methods in microencapsulating fatty-acids-rich oil due to its simplicity, reproducibility, economics and ease of scaling up compared to other methods [10-13]. The spray drying technique has been widely implemented in microencapsulating numerous oil such as European eel [14], anchovy [15], tuna [16], swamp eel [17], menhaden fish [12] and other commercialised fish oil [18-21].

Although microencapsulation provides adequate protection, selecting suitable wall materials and operating conditions based on the core materials' properties influenced the success rate of the encapsulation process. A wide range of wall materials available are based on the applications of desired products and play a significant role in limiting the microcapsules' volatile losses, flowability and encapsulation efficiency [14, 22, 23]. Whey proteins have been reported to be an excellent wall material for fatty-acid oils due to their amphiphilic properties. Whey protein also possesses high diffusivity compared to other wall materials, contributing to efficient core surface coverage [24-26]. Maltodextrins consisting of D-glucose is commonly used as polymers due to their potential to prevent oxidation and contribute to low production cost [27]. According to Mohammed et al. [28], a blend-based emulsion provides better protection towards core materials as protein-based materials act as emulsifier while carbohydrate performs as matrix-forming materials.

Over the past decade, various findings on the microencapsulation of fish oil have been reported, including the effect of emulsion formulation and spray drying operating parameters [6, 13, 21, 29, 30]. However, to date, no scientific report on the effect of various feed flow rates during the spray drying process on the physicochemical properties of menhaden fish oil powder has been reported. Therefore, this study highlighted the influence of feed flow rate range 280 mL/h to 444 mL/h in spray drying. The combination of maltodextrin (MD) and whey protein isolate (WPI) was used as carrier agents. The powdered microencapsulated menhaden fish oil obtained was analysed for its moisture content, peroxide value, particle size distribution, free fatty acid value, acid value, encapsulation efficiency and surface morphological images.

2. Materials and Methods

2.1. Chemical and materials

Commercial menhaden fish oil CAS 8002-50-4 (Merck, Germany) was used as encapsulated materials. Whey protein isolate (WPI) and Maltodextrin C.P. were supplied by Myprotein (Malaysia) and R&M Chemical (Malaysia), respectively. The analytical grade ethanol, methanol and hexane were supplied by Merck (Germany). The emulsions were prepared using deionised water from Milli-Q Millipore (Merck, Germany). All chemicals and materials were used without further purification process.

2.2. Preparations of emulsion and spray drying

The preparation of emulsion formulation before the spray drying process was based on Tirgar et al. [30] with slight modification. First, the aqueous emulsion was prepared by dispersing 15 wt% maltodextrin (wall material) in 300 mL deionised water at 70 ˚C. Next, about 20 wt% WPI (emulsifier) was added and stirred at 700 rpm for 20 h at room temperature. After completion of the hydration process, 10 wt% of menhaden fish oil (core material) was gradually added into the emulsion formulation while mixing. Finally, homogenised emulsification was achieved using a high-speed homogenizer (IKA Homogeniser, Germany) for 5 min at 20,000 rpm. Each batch of homogenised emulsion had a volume of 400 mL and was used immediately to produce menhaden fish oil microcapsules.

The formulated emulsion was processed into a fine powder using a laboratory scale spray dryer with an inside dimension of 1110 mm height, 825 mm width and 600 mm diameter (Lab Plant SD - 06A, UK). The temperature, air dry flow rate and feed flow rate were set at 210 ºC, 3.5 m/s and 280 mL/h, respectively. The microencapsulated menhaden fish oil was collected in a well-closed glass chamber and stored in an airtight brown bottle at room temperature. The same procedures were repeated for spray drying with a feed flow rate of 321, 362, 403 and 444 mL/h.

2.3. Measurement of moisture content

MS-70 moisture analyser (A&D, Japan) with a readability of ± 0.0001 % was used to measure the moisture content (MC) of menhaden fish oil powder. About 2 g of menhaden fish oil powder was placed on the analyser and the sample was heated at 105 ºC. The MC in % was displayed once a constant weight was obtained. The same procedure was repeated for all samples and measured in triplicates.

2.4. Powder particle size

Malvern 2000 Mastersizer Analyzer (Malvern Instruments Co., Worcestershire, UK) was employed to measure menhaden fish oil powder particle size distribution (PSD). It was fortified with an automated Scirocco 2000 unit that controls sample distribution into the unit. The method described by Abdul Mudalip et al. [31] was implemented. The PSD was measured by volume-weighted mean method and triplicates.

2.5. Free fatty acid value and acid value analysis

The method from Afolabi et al. [32] with slight modifications was adopted. Briefly, 2 g of menhaden fish oil powder was mixed with 30 ml ethanol and stirred. A Metrohm 785 DMP Titrino (Metrohm, Herisau, Switzerland) with a potentiometric sensor was used to measure the FFA and AV. The data of FFA and AV were obtained from the curve of titration measured. All samples were subjected to the same protocols. Each measurement was conducted three times.

2.6. Powder encapsulation efficiency

Determination of menhaden fish oil powder encapsulation efficiency (EE) followed Charles et al. [16] method. About 2 g (total oil) of the menhaden fish oil microcapsule was diluted with 15 mL hexane and vortexed for 3 min to de-encapsulate the wall material from the core material. Then, the mixture was centrifuged at 3500 rpm for 60 min to separate the wall material with supernatant (hexane and menhaden fish oil). Surface fish oil was achieved by separating hexane and menhaden fish oil by evaporating the supernatant in a fume hood and was determined gravimetrically. The same procedures were repeated for all samples and measured in triplicates. The collected menhaden fish oil was quantitatively calculated using Eq. (1).

$$EE\ (\%) = \left(\frac{Total\ oil\ (g) - Surface\ fish\ oil\ \ (g)}{Total\ oil\ (g)}\right)\ X\ \ 100\% \qquad (1)$$

2.7. Fatty acid composition

The fatty acids content identification for the best feed flow rate of menhaden fish oil powder was employed by GC-MS Agilent 6890 (Agilent Technologies, USA) equipped with polyethene glycols DB-Wax column (30 m × 0.250 mm × 0.250 µm). Briefly, 100 mg menhaden fish oil powder was mixed with 100 µL 2N NaOH and added into methanol. The mixture was then vortexed for 30 s and centrifuged for 10 min at 1000 rpm. The retrieved supernatant was placed in an auto-sample vial prior to analysis. The oven temperature, inlet temperature and final temperature were set at 35 ºC, 250 ºC and 280 ºC, respectively. Finally, about 1 µL of the prepared sample was injected and fatty acid was identified by comparing the retention time with NIST 05a library database.

2.8. Powder surface morphology

TM3030 Plus SEM (Hitachi, Japan) was implied to investigate the morphological images of powder with lower and higher feed flow rates. In addition, the powdered sample was sputter coated with gold (Quorum Technologies Q300TD, UK) to inhibit charging and reduce thermal damage. The gold-coated powdered samples were analysed at a magnification of 1000x with an accelerating voltage of 10.0 kV.

3. Results and Discussion

3.1. Moisture content

The microencapsulated powder absorbs moisture when continuously exposed to any environment with elevated relative humidity. One of the critical factors in the encapsulation process by spray drying is moisture content (MC), as high-water activity enhances lipid oxidation [16]. Thus, MC was used to determine the shelf life and quality of the microencapsulated powder, as it is closely interrelated to stability [12]. The effect of feed flow rate towards MC of menhaden fish oil powder is illustrated in Fig. 1. As depicted in Fig. 1, the MC of menhaden fish oil powder range from 3.15 ± 0.01 % at 280 mL/h to 3.54 ± 0.06 % at 444 mL/h. Based on the results, the MC increased with feed flow rates. Thus, it negatively affected the microencapsulated menhaden fish oil. Based on Abdul Mudalip et al. [31], it can be seen that all of the MC obtained was within the acceptable range for food-based products. This is because the safe MC for microencapsulated powder from microbial activity and storage should be less than 5 %.

These findings aligned with the previous result, where an increment in feed flow rate will reduce the contact time between drying air and emulsion formulation, making the moisture more difficult to diffuse. The higher feed flow rate results in less efficient heat transfer, thus leading to a lesser amount of water evaporation [33]. According to Lejaniya and Pui [34], a higher feed flow rate during the microencapsulation process influences the evaporating intensity and, thus, increases the percentage of water content in the microencapsulated powder. Similar trends also can be observed in the spray drying application on Jujube, where MC was significantly increased when the feed flow rate increased [35]. Therefore, it can be suggested that 280 mL/h was the best operating feed flow rate as a lower MC of 3.15 ± 0.01 % recorded, which indicates sufficient contact time between fish oil emulsion and drying air, resulting in efficient heat transfer and an increased rate of water evaporation.

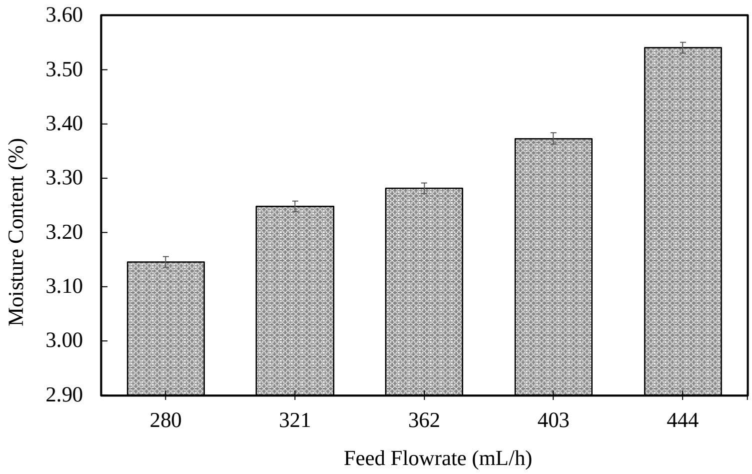

Fig. 1. The effect of feed flow rate towards MC of menhaden fish oil powder. Results are expressed as % ± standard deviations.

3.2. Particle size distribution

The particle size of microencapsulated powder significantly influences its final goods' sensory and textural properties [6]. Figure 2 illustrates the particle size distribution (PSD) of microencapsulated menhaden fish oil produced at different feed flow rates. The particle shows a bimodal distribution trend comprising two distinct peaks, each predominant representing size [33]. The range of PSD for the feed flow rate of 280 to 444 mL/h was between 27.621 ± 0.320 µm and 50.636 ± 1.321 µm. The mean diameter and volume-weighted mean of the microencapsulated menhaden fish oil are summarized in Table 1. There was a significant difference in particle size as the feed flow rate increased and the highest volume weight distribution of 405.769 µm was recorded at the feed flow rate of 362 mL/h. Volume weight distribution data is crucial, specifically from a commercial perspective, as the distribution represents the sample's composition in terms of volume or mass.

The findings were comparable with Goyal et al. [36], who reported a PSD range of 0.5 – 70 µm with bimodal distribution for flax seed oil. According to Aghbashlo et al. [37], the microcapsule size directly varies with the feed flow rate at the constant inlet air temperature and dry air flow rate. These findings agreed with Chegini and Ghobadian [38] who reports that increasing the feed flow rate resulted in bigger emulsion droplets and powder particle size. Similar behaviour was published by Carmona et al. [22], who reported the microencapsulation of orange essential oil. This is because the powder with higher MC produced at a higher feed flow rate may tend to stick together, thus leading to a larger particle form. Therefore, it can be inferred that a lower feed flow rate of 280 mL/h is more desirable as it produces a smaller microcapsule with lower MC and no visible fissures or cracks on the surface of the microcapsules (Fig. 4).

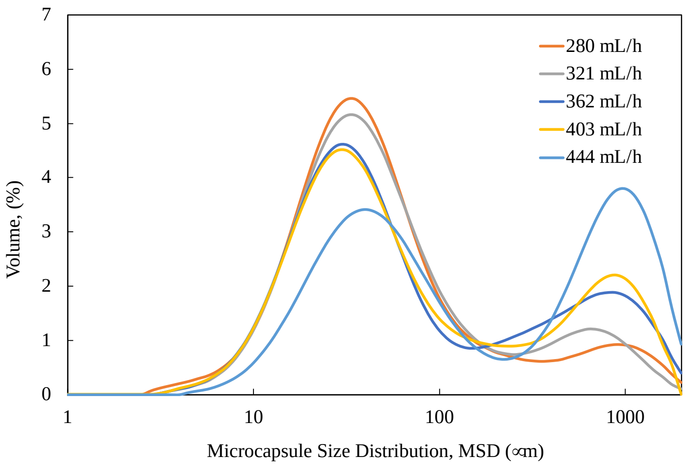

Fig. 2. PSD of menhaden fish oil powder at different feed flow rates.

Table 1. Mean diameter and volume weighted mean of menhaden fish oil powder at different feed flow rates.

Feed Flow Rate, mL/h	Mean Diameter, µm	Volumed Weighted Mean, µm
280	27.621 ± 0.320	231.161
321	32.145 ± 1.001	141.075
362	39.444 ± 0.231	405.769
403	45.801 ± 1.189	137.626
444	50.636 ± 1.321	260.216

3.3. Acid value and free fatty acid value

It was reported that most fish oil products currently available in the market used acid value (AV) as one of the indicators to determine hydrolytic rancidity. Hydrolytic rancidity defines by the production of free fatty acid (FFA) due to slow oil hydrolysis, mainly by enzymatic activities, microbial activities, or heat. According to Charles et al. [16], hydrolytic rancidity is directly proportional to the hydrolysation of triglycerides, thus leading to lipid oxidation. Commonly, hydrolytic rancidity affects the end products' nutritive values, taste, and odour during storage time. Based on the United States Pharmacopeia and Codex Standard for Fish Oils Rep 15/FO Appendix III (CAC, 2017), the safe AV of fats or oils recommended for daily human consumption should be less than 3 mg KOH/g. Lower AV indicates better production of oil quality.

Table 2 shows the AV and FFA of microencapsulated menhaden fish oil at different feed flow rates. Based on this study, the feed flow rate of 280 mL/h and 321 mL/h recorded AV lower than 3 mg (KOH/g), while the feed flow rate of 362 mL/h, 403 mL/h and 444 mL/h exceeded the Codex Standard of fish oil. The AV of menhaden fish oil microcapsule powder at a feed flow rate of 280 mL/h and 321 mL/h are below the acceptable limit, thus indicating that the feed flowrate and combination of the wall materials used in this study significantly delayed or inhibited the hydrolytic rancidity of menhaden fish oil powder. FFA depicted the free fatty acid content in percentage for the microcapsules. A similar pattern can be observed as the feed flow rate increased, percentage of FFA also increased, indicating the hydrolysis of triglycerides. It can be inferred that such a phenomenon occurred due to higher moisture content as the feed flow rate increased. As seen in Table 2, the feed flow rate of 280 mL/h showed the AV and FFA values and thus can be regarded as the best feed flow rate.

Table 2. AV and FFA of menhaden fish oil at different feed flowrate. Results are expressed as mg(KOH/g) or % ± standard deviations.

Feed Flow Rate, mL/h	Acid Value, mg (KOH/g)	Free Fatty Acid, %
280	2.82 ± 0.004	5.11 ± 0.101
321	2.91 ± 0.087	5.39 ± 1.107
362	3.24 ± 0.001	5.51 ± 0.012
403	3.32 ± 0.056	6.01 ± 0.784
444	3.88 ± 0.321	6.23 ± 0.171
Menhaden fish oil	2.74 ± 0.003	4.48 ± 0.013

3.4. Encapsulation efficiency

The critical factor in evaluating fish oil's potential range of feed flow rate is encapsulation efficiency (EE). The EE defines the quantity of fish oil entrapped inside the powder [6] and can be used to determine whether a specific functionality of the encapsulated components is delivered into the food matrix [8]. Figure 3 shows the EE of menhaden fish oil powder at various feed flow rates.

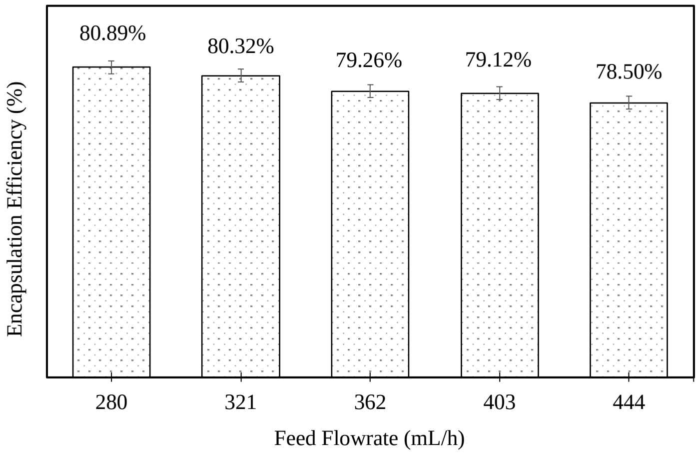

Fig. 3. Encapsulation efficiency of menhaden fish oil powder at various feed flow rates. Results are expressed as % ± standard deviations.

The range of EE obtained was a minimum of 78.50 ± 0.134 % to a maximum of 80.89 ± 0.231 %. The feed flow rate of 280 mL/h recorded the highest EE of 80.89 ± 0.231 %, while 444 mL/h recorded the lowest EE of 78.50 ± 0.134 %. Similar findings by Aghbashlo et al. [29] reported that commercialising fish oil at different feed flow rates produced microcapsules that exhibit 60 % to 82.38 % EE. An increment in the feed flow rate led to a decrement in the EE of the menhaden fish oil powder as the larger particle size was obtained. The larger droplet size of microencapsulate affects the oil-controlled release mechanism as the layer of the emulsion or coating materials is too thick and impenetrable, thus leading to lower encapsulation efficiency. The higher range of EE value proposed that studied feed flow rate range behaved as cryoprotectant up to a certain level [16] where it helped to protect and stabilize the core materials against impulsive oxidation, thus increasing the shelf life [39].

3.5. Fatty acid composition

The powder obtained at the best feed flow rate of 280 mL/h was analysed for its fatty acid compositions. Table 3 shows the fatty acid profile tabulated by peak area in percentage (%) for both microencapsulated and crude menhaden fish oil. In this study, palmitic acid (C16:0), the most predominant saturated fatty acid (SFA) in most diets, recorded the highest peak area of 17.95 % and 29.90 % in both microencapsulated and crude menhaden fish oil, respectively. A study by Taktak et al. [14] also reported the presence of C16:0 in microencapsulated European eel. However, only a tiny percentage of polyunsaturated fatty acid (PUFA), which were DHA and EPA composition, was detected for both samples with the same trend of reduction in percentage before and after microencapsulation. Fatty acids degrade when exposed to higher temperatures. In this study, it can be inferred that spray drying helped to retain the maximum number of fatty acids even though there was a slight reduction in percentage before and after microencapsulation. During emulsion preparation and encapsulation, temperature and physical stresses could affect fatty acid compositions in both samples, thus reducing the percentage of their composition.

Table 3. Percentage of fatty acid composition in microencapsulated menhaden fish oil and crude menhaden fish oil.

Compounds	Microencapsulated Menhaden Fish Oil	Crude Menhaden Fish Oil
Capric Acid (C10:0)	0.01	0.02
Lauric Acid (C15:0)	0.41	0.17
Myristic Acid (C14:0)	0.59	0.24
Palmitic Acid (C16:0)	17.95	29.90
Pentadecyclic Acid (C15:0)	1.03	1.11
Stearic Acid (C18:0)	1.61	1.83
Margaric Acid (C17:0)	0.39	0.76
Eicosapentaenoic Acid (C20:1)	0.21	7.59
Docosahexaenoic Acid (C22:6)	0.31	1.03

***** Results are expressed as a percentage of total compounds detected.

3.6. Particle size morphology

Scanning Electron Microscopy (SEM) was adapted to examine the surface appearance and structure of menhaden fish oil powders obtained using spray drying at different feed flow rates. SEM analysis showed the highest and lowest feed flow rate of 280 mL/h and 444 mL/h for its micrograph images. Figure 4(a) depicts the morphological images for menhaden fish oil powders at a feed flow rate of 280 mL/h at 2000x magnification. There was an absence of visible fissures and cracks on the microcapsules' surface, implying lower permeability to gases and efficiently shielding core material against oxidation [6]. Figure 4(b) illustrates the morphological images for menhaden fish oil powders at a feed flow rate of 444 mL/h at 2000x magnification. Based on the micrograph images obtained, the presence of visible dent and irregular microsphere shapes negatively affect the powder's flow properties and lead to oxidation due to the increase of surface area [29]. Results noticeably displayed significant differences in the shape and size of the microsphere, which is typical for spray-dried particles. Consequently, the contact time between drying air and wall materials is also reduced, leading to less effective heat transfer. According to Venugopalan et al. [20], the size of microcapsules influences the oxidative stability of microencapsulated products as it determines the percentage of surface area that is exposed to the environment air. The shrivelled particles for both images indicate slow coating formation at atomised droplets' drying process, which leads to the shrinkage of microcapsules during the finishing phases of the drying and cooling process [40].

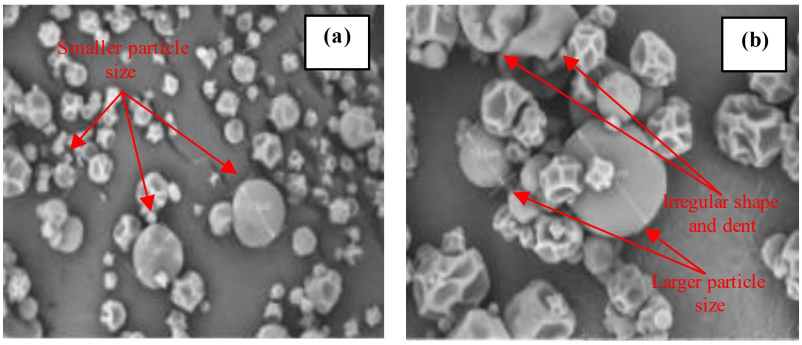

Fig. 4. Morphological images for menhaden fish oil powders at a feed flow rate of (a) 280 mL/h at 2000x magnification; (b) 444 mL/h at 2000x magnification

4. Conclusion

In conclusion, the influence of feed flow rate during the spray drying process towards the encapsulation of commercial menhaden fish oil with maltodextrin and whey protein was successfully determined. It can be inferred that the feed flow rate significantly influenced the physicochemical of the microcapsule and the feed flow rate of 280 mL/h exhibited better overall microcapsules' physicochemical properties with the lowest moisture content of 3.15 ± 0.01% and particle size of 27.621 ± 0.320 μm. Although the microcapsules produced showed the highest free fatty acid value of 5.11 ± 0.101 %, it can be considered to have better stability since it showed the lowest acid value, which was 2.82 ± 0.004 mg (KOH/g) and the highest encapsulation efficiency of 80.89 ± 0.231 %. Besides, the morphological images of the spray-dried microcapsules showed smooth particle surfaces due to the lower water contents and higher encapsulation efficiency. The results of this study can be further interpreted to understand the role of operating parameter specificity feed flow rate in the spray drying process. Other process parameters, such as air-dry flow rate and emulsion ratios, should be included in future research to give a comprehensive insight into the microencapsulation process. Future research can also be directed toward using potential industrial waste as a wall material, significantly reducing the production cost and enhancing the microcapsules' quality.

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Abbreviations

AV	Acid Value
EE	Encapsulation Efficiency
FFA	Free Fatty Acid
MC	Moisture Content
MUFA	Monosaturated Fatty Acid
PSD	Particle Size Distribution
PUFA	Poly-unsaturated Fatty Acid
SFA	Saturated Fatty Acid

References

1. De Boer, A.A.; Ismail, A.; Marshall, K.; Bannenberg, G.; Yan, K.L.; and Rowe, W.J. (2018). Examination of marine and vegetable oil oxidation data from a multi-year, third-party database. *Food Chemistry*, 254, 249-255.
2. Barrow, C.J.; Wang, B.; Adhikari, B.; and Liu, H. (2013). Spray drying and encapsulation of omega-3 oils. *Food Enrichment with Omega-3 Fatty Acids*, 194-225.
3. Chen, X.; Hong, S.; Sun, X.; Xu, W.; Li, H.; Ma, T.; Zheng, Q.; Zhao, H.; Zhou, Y.; Qiang, Y.; Li, B.; and Li, X. (2020). Efficacy of fish oil and its

components in the management of psoriasis: a systematic review of 18 randomized controlled trials. *Nutrition Reviews,* 78(10), 827-840.

4. Amin, M.; Earlia, N.; Annisa, Y.; Amin, A.Z.; Prakoeswa, C.R.S.; Idroes, R.; Amin, I.F.; and Lukiati, B. (2018). Eicosapentaenoic Acid (EPA) from fish oil and margarine as bioactive compound for anti-inflammation in occupational dermatitis. *KnE Life Sciences,* 4(5), 593-698.
5. Tonon, R.V.; Pedro, R.B.; Grosso, C.R.F.; and Hubinger, M.D. (2012). Microencapsulation of flaxseed oil by spray drying: Effect of oil load and type of wall material. *Drying Technology,* 30(13), 1491-1501.
6. Lavanya, M.N.; Kathiravan, T.; Moses, J.A.; and Anandharamakrishnan, C. (2019). Influence of spray-drying conditions on microencapsulation of fish oil and chia oil. *Drying Technology,* 38(3), 279-292.
7. Bakry, A.M.; Abbas, S.; Ali, B.; Majeed, H.; Abouelwafa, M.Y.; Mousa, A.; and Liang, L. (2016). Microencapsulation of oils: A comprehensive review of benefits, techniques, and applications. *Comprehensive Reviews in Food Science and Food Safety,* 15(1), 143-182.
8. Choudhury, N.; Meghwal, M.; and Das, K. (2021). Microencapsulation: An overview on concepts, methods, properties and applications in foods. *Food Frontiers,* 2(4), 426-442.
9. Hashim, N.A.; Abdul Mudalip, S.K.; Sulaiman, S.Z.; and Md Shaarani, S. (2020). Nutritional values and microencapsulation techniques of fish oil from different sources: A mini review. *Materials Today: Proceedings,* 42, 222-228.
10. Ramos, F.M.; Silveira Junior, V.; and Prata, A. S. (2021). Impact of vacuum spray drying on encapsulation of fish oil: Oxidative stability and encapsulation efficiency. *Food Research International,* 143, 1-10.
11. Aprilia, I.; Pujiastuti, D.Y.; and Klaypradit, W. (2021). Microencapsulated fish oil powder by spray drying using combination of wall materials in Kasetsart University, Bangkok. *IOP Conference Series: Earth and Environmental Science,* 679, 1-5.
12. Mehrad, B.; Shabanpour, B.; Jafari, S.M.; and Pourashouri, P. (2015). Characterization of dried fish oil from menhaden encapsulated by spray drying. *International Journal of the Bioflux Society,* 8(1), 57-69.
13. Encina, C.; Marquez-Ruiz, G.; Holgado, F.; Gimenez, B.; Vergara, C; and Robert, P. (2018). Effect of spray-drying with organic solvents on the encapsulation, release and stability of fish oil. *Food Chemisty,* 263, 283-291.
14. Taktak, W., Nasri, R., Lopez-Rubio, A.; Hamdi, M.; Gomez-Mascaraque, L. G.; Amor, N.B.; Kabadou, A.; Li, S.; Nasri, M.; and Karra-chaabouni, M. (2019). Improved antioxidant activity and oxidative stability of spray dried European eel (*Anguilla anguilla*) oil microcapsules: Effect of emulsification process and eel protein isolate concentration. *Materials Science & Engineering,* 104, 1-10.
15. Tatar, F.; and Kahyaoglu, T. (2015). Microencapsulation of anchovy (*Engraulis encrasicolus L.*) oil: Emulsion characterization and optimization by response surface methodology. *Journal of Food Processing and Preservation,* 39(6), 624-633.
16. Charles, A.L.; Abdillah, A.A.; Saraswati, Y.R.; Sridhar, K.; Balderamos, C.; Masithah, E.D.; Alamsjah, M.A. (2021). Characterization of freeze-dried

microencapsulation tuna fish oil with arrowroot starch and maltodextrin. *Food Hydrocolloids*, 112, 1-8.

17. Sri Priatni, S.P.; Harimadi, K.; Buana, E.; Kosasih, W.; and Rohmatussolihat, R. (2020). Production and Characterization of Spray-Dried of Swamp Eel (*Monopterus Albus*) Protein Hydrolysate Prepared by Papain. *Sains Malaysiana*, 49(3), 545-552.
18. Espinosa-Andrews, H.; Morales-Hernández, N.; García-Márquez, E.; and Rodríguez-Rodríguez, R. (2022). Development of fish oil microcapsules by spray drying using mesquite gum and chitosan as wall materials: physicochemical properties, microstructure, and lipid hydroperoxide concentration. *International Journal of Polymeric Materials and Polymeric Biomaterials*, 1-10.
19. Selim, K.A.; Alharthi, S.S.; Abu El-Hassan, A.M.; Elneairy, N.A.; Rabee, L.A.; and Abdel-Razek, A.G. (2021). The effect of wall material type on the encapsulation efficiency and oxidative stability of fish oils. *Molecules*, 26(20), 1-17.
20. Venugopalan, V.K.; Gopakumar, L.R.; Kumaran, A.K.; Chatterjee, N.S.; Soman, V.; Peeralil, S.; Mathew, S.; McClements, D.J.; Nagarajarao, R.C. (2021). Encapsulation and protection of omega-3-rich fish oils using food-grade delivery systems. *Foods*, 10(7), 1-20.
21. Łozińska, N.; Głowacz-Różyńska, A.; Artichowicz, W.; Lu, Y.; and Jungnickel, C. (2020). Microencapsulation of fish oil – determination of optimal wall material and encapsulation methodology. *Journal of Food Engineering*, 268, 2020.
22. Carmona, P.A.O.; Tonon, R.V.; da Cunha, R.L.; and Hubinger, M.D. (2013). Influence of emulsion properties on the microencapsulation of orange essential oil by spray drying. *Journal of Colloid Science and Biotechnology*, 2(2), 130-139.
23. Chang, C.; Varankovich, N.; and Nickerson, M.T. (2016). Microencapsulation of canola oil by lentil protein isolate-based wall materials. *Food Chemisty*, 212, 264-73.
24. Vahidmoghadam, F.; Pourahmad, R.; Mortazavi, A.; Davoodi, D.; and Azizinezhad, R. (2019). Characteristics of freeze-dried nanoencapsulated fish oil with whey protein concentrate and gum arabic as wall materials. *Food Science and Technology*, 39(2), 475-481.
25. Hundre, S.Y.; Karthik, P.; and Anandharamakrishnan, C. (2015). Effect of whey protein isolate and beta-cyclodextrin wall systems on stability of microencapsulated vanillin by spray-freeze drying method. *Food Chemisty*, 174, 16-24.
26. Horincar, G.; Aprodu, I.; Barbu, V.; Rapeanu, G.; Bahrim, G.E.; and Stanciuc, N. (2019). Interactions of flavonoids from yellow onion skins with whey proteins: Mechanisms of binding and microencapsulation with different combinations of polymers. *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, 215, 158-167.
27. Yakdhane, A.; Labidi, S.; Chaabane, D.; Tolnay, A.; Nath, A.; Koris, A.; and Vatai, G. (2021). Microencapsulation of flaxseed oil - state of art. *Processes*, 9(2), 1-16.

28. Mohammed, N.K.; Tan, C.P.; Manap, Y.A.; Muhialdin, B.J.; and Hussin, A.S.M. (2020). Spray drying for the encapsulation of oils - A review. *Molecules*, 25(17).
29. Aghbashlo, M.; Mobli, H.; Madadlou, A.; and Rafiee, S. (2012). Influence of wall material and inlet drying air temperature on the microencapsulation of fish oil by spray drying. *Food and Bioprocess Technology*, 6(6), 1561-1569.
30. Tirgar, M.; Jinap, S.; Zaidul, I.S.; and Mirhosseini, H. (2015). Suitable coating material for microencapsulation of spray-dried fish oil. *Journal of Food Science and Technology*, 52(7), 4441-9.
31. Abdul Mudalip, S.K.; Kathiman, M.N.; and Gimbun, J. (2022). Effect of feed flowrates on the physical properties and antioxidant of Mahkota Dewa (*Phaleria Macrocarpa*) encapsulated powder. *IIUM Engineering Journal*, 23(2), 1-9.
32. Afolabi, H.K.; Mudalip, S.K.A.; and Alara, O.R. (2018). Microwave-assisted extraction and characterization of fatty acid from eel fish (*Monopterus albus*). *Beni-Suef University Journal of Basic and Applied Sciences*, 7(4), 465-470.
33. Tonon, R.V.; Brabet, C.; and Hubinger, M.D. (2008). Influence of process conditions on the physicochemical properties of açai (*Euterpe oleraceae* Mart.) powder produced by spray drying. *Journal of Food Engineering*, 88(3), 411-418.
34. Lejaniya A.K.S.; and Pui, L.P. (2022). Effects of spray-drying parameters on physicochemical properties of powdered fruits. *Foods and Raw Materials*, 235-251.
35. Chen, Q.; Bi, J.; Zhou, Y.; Liu, X.; Wu, X.; and Chen, R. (2013). Multi-objective optimization of spray drying of jujube (*Zizyphus jujuba Miller*) powder using response surface methodology. *Food and Bioprocess Technology*, 7(6), 1807-1818.
36. Goyal, A.; Sharma, V.; Sihag, M.K.; Tomar, S.K.; Arora, S.; Sabikhi, L.; and Singh, A.K. (2015). Development and physico-chemical characterization of microencapsulated flaxseed oil powder: A functional ingredient for omega-3 fortification. *Powder Technology*, 286, 527-537.
37. Aghbashlo, M.; Mobli, H.; Madadlou, A.; and Rafiee, S. (2013). Fish oil microencapsulation as influenced by spray dryer operational variables. *International Journal of Food Science & Technology*, 48(8), 1707-1713.
38. Chegini G.R.; and Ghobadian, B. (2005). Effect of spray-drying conditions on physical properties of orange juice powder. *Drying Technology*, 23(3), 657-668.
39. Anwar, S.H.; and Kunz, B. (2011). The influence of drying methods on the stabilization of fish oil microcapsules: Comparison of spray granulation, spray drying, and freeze drying. *Journal of Food Engineering*, 105(2), 367-378.
40. BÖGer, B.R.; Georgetti, S.R.; and Kurozawa, L.E. (2018). Microencapsulation of grape seed oil by spray drying. *Food Science and Technology*, 38(2), 263-270.