Characteristics of the Encapsulated Unsaturated Fatty Acid Concentrate of Catfish Oil (*Pangasius* sp.)

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Abstract. Concentrated unsaturated fatty acids are processed from refined catfish oil extracted from belly meat which is the byproduct of the industry processing pangasius fillets. This belly part contains high enough fat which can be used as a source of essential fatty acids. This essential fatty acid is useful for human health and can reduce the risk of cancer and cardiovascular disease. The purpose of this study was to evaluate the physical and chemical characteristics of the encapsulated concentrated unsaturated fatty acids that have been produced. This study used a combination of coatings (maltodextrin and sodium caseinate; 9:1, 8:2, 7:3) with a ratio of unsaturated fatty acid concentrate with coating 1:5. The physicochemical characteristics analyzed were microencapsulation efficiency, dried encapsulated yield, emulsion viscosity, wettability, water solubility, morphology (SEM) and hygroscopicity. Based on the results of the study show that the ratio of 9:1 is the most optimal comparison in this study which have the lowest emulsion viscosity 12.60 cP with the efficiency of microencapsulation of 40.91% and the dried encapsulated yield of 47.82%.

1. Introduction
Fish oil obtained from the byproducts of the pangasius fillets still contains many essential fatty acids which are needed by the human body. Several studies related to the production and characterization of fish oil from side products of the fish processing industry have been carried out quite a lot. For example Sathivel et al. [1] conducted research by producing oil from the viscera contents of catfish (*Ictalurus punctatus*). Hastarini [2] succeeded in characterizing fish oil from the by-products of processing Siame catfish fillets and Jambal catfish. Arifianto [3] also uses the byproducts of Siamese catfish to be characterized and optimized for extracts from the oil. The fat content of the body parts of catfish is quite varied, with the lowest content of the fillet portion is 2.72% while the highest content of the belly meat is 36.21% [12]. The high fat content of the meat belly of catfish is very potential to be utilized, by being extracted into fish oil as a source of essential fatty acids. This underlies the importance of using fish oil especially those containing high unsaturated fatty acids so
that they can be utilized optimally and have benefits for the body. Some components of high
unsaturated fatty acids are important in fish oil, namely omega-3 fatty acids, omega-6 and omega-9.
Omega 3 fatty acids (EPA and DHA) can help the brain's growth process (intelligence), the
development of the sense of sight, and the immune system of a toddler. EPA and DHA fatty acids
are also useful in healing the symptoms of keloids [4], reducing cholesterol in the blood, especially
LDL, anti platelet aggregation and anti-inflammation [5].

Fish oil which specifically contains omega-3 or omega-6 fatty acids is more developed as a
supplement product or other pharmaceutical products. Utilization as a functional food is also done
quite a lot with fortification methods on dairy foods such as milk, cheese and yogurt [6]. The high
content of unsaturated fatty acids in fish oil makes fish oil easily oxidized. Oxidation products,
especially the secondary ones, can cause rancid flavors and aromas that are easily detected by the
human senses, even at very low levels [7]. One technology that can overcome the problem of
rancidity that often occurs in fish oil is microencapsulation technology.

Microencapsulation is defined as the technology of coating solids, gases and liquids by small
capsules. The capsule will release the material encapsulated at a controlled rate and certain
conditions. The challenge of the application of microencapsulation technology lies in the selection
of coating materials and the right combination.

Spray drying technique is a microencapsulation technique that has been widely used for
commercial production and is best used for heat sensitive materials, due to relatively short drying
times [8]. Microencapsulation with spray drying consists of four stages, namely (1) preparation of
emulsion solution (2) homogenization (3) emulsion atomization through the dryer nozzle and (4)
evaporation of water in the emulsion droplet [9]. Studies of microencapsulation of fatty acids from
various types of fish oil from marine fish have been reported previously. However, studies on
microencapsulation of concentrates of unsaturated fatty acids in catfish oil have not been reported.

The choice of coating material greatly influences the quality of the microcapsules produced,
so it needs to be adapted to the filling material and the microencapsulation technique used. Maltodextrin is one type of coating that is often used in the spray drying process. Maltodextrin is a
modified starch obtained from the hydrolysis of partially with acids or enzymes. This material is
often used because it has many advantages such as being able to provide low viscosity at a
concentration high enough in the emulsion. Besides that, it is also easy to get and cheap. However,
maltodextrin in the oil microencapsulation process by spray dryer causes low emulsion and oil
retention stability, but encapsulated oil has resistance to oxidation [10]. To improve the
encapsulation ability of maltodextrin, it can be combined with other types of coating material to
increase the emulsion stability [11, 12].

Sodium caseinate is one of the coatings that is often used in microencapsulation processes.
This material is easy to obtain, stable to heat and easily forms films from aqueous solutions because
naturally the molecules are random, extensively capable of forming intermolecular hydrogen bonds,
electrostatic bonds and hydrophobic bonds [13]. The disadvantage of sodium caseinate is that its
performance is limited to certain viscosities and sometimes gives a browning effect to the product.

This study used a combination of maltodextrin and sodium caseinate coating material to obtain
good quality microcapsules. The purpose of this study was to evaluate the physical and chemical
characteristics of the encapsulated concentrated unsaturated fatty acids that have been produced.

2. Material and Methods
2.1. Material
The core material used is unsaturated fatty acids concentrated from catfish oil obtained from the part
of belly meat side by side with the fillet processing industry (based on our previous research by
Khamidah et al. [14]. The coating material used is Maltodextrin and Sodium Caseinate. Other supporting materials include tween 80 emulsifiers and technical NaCl.

2.2. Methods
2.2.1 Unsaturated Microencapsulation of Fatty Acid

Microencapsulation of unsaturated fatty acid concentrates was carried out based on modifications from Yuliasari [15]. The sample is prepared by conducting a coating comparison between maltodextrin and sodium caseinate. The comparison between unsaturated fatty acid concentrates with coatings is 1:5, and the following formula is tested (Table 1).

| Code | Comparison | Unsaturated FA (g) | MD (g) | NaK (g) | Aquades (g) | Tween (g) |
|------|------------|--------------------|--------|---------|-------------|-----------|
| A    | 9/1        | 15                 | 67.5   | 7.5     | 225         | 9.45      |
| B    | 8/2        | 15                 | 60     | 15      | 225         | 9.45      |
| C    | 7/3        | 15                 | 52.5   | 22.5    | 225         | 9.45      |

The use of aquades is 2.5 times the amount of unsaturated fatty acid concentrates and coatings, while the number of tween 80 used is 3% of the mixture of unsaturated fatty acid concentrates, coatings and distilled water. The emulsion formed is then homogenized using a homogenizer (Ultra-Turrax model, Armfield) with a speed of 11000 rpm for 5 minutes. Furthermore, each sample was microencapsulated using a spray drying technique with an inlet temperature of 180°C, an outlet temperature of 100°C and a feed rate of 10 mL/minute. The following is the formula used to calculate the yield of encapsulate (DY) dry weight and microencapsulation efficiency (EM).

\[
DY = \frac{\text{Total Dry Weight in Product}}{\text{Total Dry Weight in Emulsification}} \times 100\%
\]

\[
EM = \frac{\text{Total Oil Content} - \text{Surface Oil Content}}{\text{Total Content}} \times 100\%
\]

2.2.2 Viscosity Emulsion (VE)

For viscosity emulsion, we follow Hastarini et al. [2]. The emulsion sample was put in a 300 mL measuring cup and then measured its viscosity using the Brookfield digital viscometer at a speed of 30 rpm. The unit used is centi Poise (cP).

2.2.3 Wettability

Wettability is determined by the method of Fuchs et al [16]. As much as 1 gram of encapsulated concentrated unsaturated fatty acids of catfish oil were dispersed on the surface of 100 mL of distilled water without stirring. The time taken is recorded until the sample is completely wetted completely or disappears from the aquades surface. Unit wettability is expressed in seconds per gram of encapsulate.

2.2.4 Solubility of encapsulates in water

Solubility was determined by a method modified by Cano-Chauca et al. [17]. About 1 gram of encapsulate sample was weighed (a), and 100 mL of distilled water was added, then filtered with a vacuum filter. Filter paper before use is dried in an oven at 105°C for about 30 minutes then weighed (b). After the screening process, filter paper along with the material residue was dried again in an
oven at 105ºC for approximately three hours, cooled in a desiccator for 15 minutes then weighed (c).

\[
\text{Solubility} = (1 - \frac{c-b}{100-%ka}) \times 100\%
\]

2.2.5 Hygroscopycity
Hygroscopycity is determined by the method of Cai and Corke [18]. As much as 1 gram of encapsulated sample is put into the desiccator with saturated NaCl salt solution (RH 75.2%) at 25ºC for 1 week. Hygroscopycity is determined as the weight of water absorbed by the encapsulate sample per 100 g of dry solids (g / 100 g).

2.2.6 Morphology
For morphology, we follow Yuliasari [15]. The surface morphology of MIP encapsulates was analyzed by Scanning Electron Microscope (SEM) EVO MA 10, Zeiss. The MIP encapsulate sample was prepared in the specimen holder and coated with gold. SEM observations were carried out at a voltage of 20 kV with a magnification of 5000 times. Analysis is done computationally. The analysis results obtained are in the form of particle display in the form of three-dimensional images according to magnifications and certain scales.

2.2.7 Statistics analysis
This study uses a completely randomized design (CRD). The difference in mean values was analyzed by analysis of variance (ANOVA) and continued with the Duncan Multiple Range Test (DMRT) test using IBM SPSS Statistics 22 software.

3. Results and Discussion
3.1. Effect of coating material on DY, EM and VE
The combination of the ratio between maltodextrin and sodium caseinate has different effects on each yield of encapsulate (DY) dry weight, microencapsulation efficiency (EM) and emulsion viscosity (VE). This can be explained through (Table 2) as follows.

| Parameter | 9/1          | 8/2          | 7/3          |
|-----------|--------------|--------------|--------------|
| DY        | 47.818±0.647a | 41.202±0.078b | 33.494±0.227c |
| EM        | 40.909±3.079a | 26.475±1.425b | 25.360±2.773b |
| VE        | 12.600±0.000b | 18.4±0.566b   | 36.8±3.394a   |

Based on Table 2, EM experienced a significant decline from 9/1 to 8/2, and was not significant from 8/2 to 7/3. The highest DY value was obtained from the 9/1 coating ratio treatment which was equal to 47.818% (Figure 1).
Figure 1. DY value of encapsulated concentrates of unsaturated fatty acids Catfish oil with a combination treatment of coating material Maltodextrin and Sodium Caseinate (9/1; 8/2; 7/3).

The DY value is influenced by the concentration of sodium caseinate in the emulsion. The higher the concentration of sodium caseinate in the emulsion mixture, the indirect the DY value will be lower. This is caused by an increase in emulsion viscosity (VE) which occurs as the concentration of sodium caseinate increases in the emulsion (Figure 2).

Figure 2. Viscosity Value of Emulsion Encapsulate Concentrated Unsaturated Fatty Acid Catfish Oil with a Combination Treatment of Maltodextrin and Sodium Caseinate Coating Materials (9/1; 8/2; 7/3).

This study is in line with Quynh et al. [19]. In this study the maximum DY parameter falls in the ratio 1 / 0.6. Emulsion that is too thick (high viscosity) can inhibit the spray drying process because it is difficult to suck into the tool. In addition, the high viscosity of the emulsion can also inhibit drying in the inlet tube. So that the resulting encapsulate has a high water content attached to the tube.

In the microencapsulation efficiency parameters, the value has decreased with increasing concentration of sodium caseinate in the emulsion (Figure 3). At a ratio of 9/1, the EM value
reaches 40.91%, then decreases significantly to 26.48% at a ratio of 8/2 and decreases again not significantly to 25.36% in the ratio 7/3. The results of the study are not in line with the results of the study of Quynh et al. [19] which states that an increase in the ratio of maltodextrin / sodium caseinate from 1/0.2 - 1/0.6 can increase EM values from 48.05 - 66%.

Figure 3. Microencapsulation Efficiency Value of Encapsulates Concentrated Unsaturated Fatty Acid Patin Oil with a Combination Treatment of Maltodextrin and Sodium Caseinate Coating Materials (9/1; 8/2; 7/3).

3.2. Effect of coating on wettability and solubility of encapsulates
Comparison of maltodextrin and sodium caseinate has significant effect on MTT parameters, wettability and solubility. This solubility value has decreased successively with a statistically significant difference. Each comparison of values is significantly different. This is probably due to the nature of maltodextrin which is very easy to dissolve in water, so the more the concentration decreases in the emulsion, the lower the value of the soluble.

Table 3. Wettability and solubility in water

| Treatment | Wettability (sec) | Solubility (%) |
|-----------|------------------|----------------|
| 9/1       | 875.7±36.062a    | 97.695±1.337ab |
| 8/2       | 667.5±14.849b    | 95.70±0.348b   |
| 7/3       | 501.9±13.152c    | 99.05±0.104a   |

Wettability is the time required for the encapsulate to be completely wetted or disappear from the surface of aquades [20]. A low wettability value indicates the faster the encapsulation falls below the surface and dissolves into the water. The results of the study (Table 3) show a decrease in the value of wettability as the ratio of Maltodextrin/Sodium Caseinate coatings increases.

Water solubility is a parameter related to the release of active ingredients during encapsulate applications [15]. Based on research (Table 3), it is seen that the value of solubility of encapsulation is very high but tends to fluctuate. The values range from 95.70 - 99.05% (Figure 4).
Very high values are probably caused by the size of microcapsules that are very small and smooth, namely the micro-form. Estiasih [7] mentions that microcapsules produced by spray drying techniques are of a fine size of around 100 mm so they are very soluble. The results of this study are not in line with the results of the study [15], where the use of sodium casein tends to reduce solubility. However, the range of solubility values obtained was not significantly different because the three values were more than 95%.

3.3. Hygroscopicity
The combination of the ratio of maltodextrin and sodium caseinate coating material has a significant effect on the hygroscopicity. The results obtained vary depending on the comparison. Based on Figure 8, the results showed that the comparison of MD/NK 9/1 coatings had the highest level of hygroscopicity followed by 8/2 and 7/3. The use of sodium caseinate in a mixture of coating materials can reduce encapsulate hygroscopicity. Water absorption is a critical factor in the shelf life of encapsulates because water can play a role in the oxidation process [21].

Dewanti et al [22] examined the hygroscopicity of cereal flour, he said that increasing the addition of maltodextrin in cereal pulp flour caused an increase in hygroscopicity. Maltodextrin has high hygroscopicity compared to other coating materials. So that the absorption of water vapor has also experienced an increase in instant cereal flour. Yuliasari [15] said that the use of arabic gum in the capsule mixture increased hygroscopicity of encapsulates, but the increase was not significant (p> 0.05)
3.4. Morphology
The morphology of the surface of the encapsulated unsaturated fatty acid concentrates illustrates the encapsulate characteristics such as the rate of release of active ingredients and uncapsulated oil.

Figure 6 shows the surface morphology of the encapsulated shoot by the Scanning Electron Microscope (SEM) instrument. Comparison of coating materials in the form of variations in malodextrin and sodium caseinate significantly affected SEM images. In the 9/1 ratio treatment, it appears that it is a whole sturdy circle and does not appear to be wrinkled with small spheres attached to the surface. Comparison 8/2 in (b) shows several deflating circles. This is presumably
because the encapsulate is not strong enough to withstand the pressure in the encapsulate, the wall will break and the encapsulate will deflate this event called ballooning. Ballooning can cause the loss of active ingredients from encapsulates so that the unencapsulated oil on the surface of the encapsulated wall becomes high. In ratio treatment 7/3 (c), the results show that spheres tended to clump, this was because the addition of sodium caseinate made the drying process more difficult due to the high viscosity of the emulsion solution.

4. Conclusion
The combination treatment of coating material in the form of maltodextrin/sodium caseinate affects the characteristics of the chemical physics of encapsulated concentrates of unsaturated fatty acids in catfish oil. Based on the results it can be concluded that the best ratio of coating material (maltodextrin / sodium caseinate) in this study is a comparison of 9/1 which have the lowest emulsion viscosity 12.60 cP with the efficiency of microencapsulation of 40.91% and the dried encapsulated yield of 47.82%.

References
[1] Sathviel, S., Prinyawiwatkul, W., King, J.M., Grimm, C.C. and Lloyd, S. (2003). Oil production from catfish viscera. J Am Oil Chem Soc, 80 (4):277-382.
[2] Hastarini, E. (2012). Karakteristik minyak ikan dari limbah pengolahan filet ikan Patin Siam (Pangasius hypophthalmus) dan Ikan Patin Jambal (Pangasius djambal). Bogor (ID): Institut Pertanian Bogor, Tesis.
[3] Arifianto, T.B. (2014). Karakterisasi bahan dan optimasi ekstraksi minyak ikan dari produk patin (Pangasius hypophthalmus). Bogor (ID): Institut Pertanian Bogor, Tesis.
[4] Olaitan, B.P., Chen, I-Ping., J. Norris., R. Feinn., O.M. Oluwatosin. and E.J Reichenberger. (2011). Inhibitory activities of omega-3 fatty acids and traditional african remedies on keloid fibroblasts. Wound, 23(4):97–105.
[5] Haris, W.S. (2004). Review: fish oil supplementation : evidence for health benefits, Cleveland Clin J Med, 71(3):208-219.
[6] Martini, S., J.E Thurgood., C. Brothersen., R. Ward. and DJ Mc Mahon. (2009). Fortification of reduced-fat cheddar cheese with n-3 fatty acids: effect on off-flavor generation. J Dairy Sci, 92:1876-1884
[7] Estiasih, T. (2009). Minyak Ikan: Teknologi dan Penerapannya untuk Pangan dan Kesehatan. Edisi Pertama. Yogyakarta: Graha Ilmu.
[8] Liu, X.D., Atarashi T., Furuta T., Yoshii H., Aishima S., Ohkawara M. and Linko P. (2001). Microencapsulation of emulsified hydrophobic flavors by spray drying. Drying Technol, 19:1361-1374.
[9] Shahidi, F. and Han X. (1993). Encapsulation of food ingredients. Crit Rev in Food Sci And Nutr, 33: 501-547.
[10] Madene, A., Jacquot, M., Scher. J. and Desobry S. (2006). Review flavour encapsulation and controlled release – a review. Int J Food Sci and Technol, 41: 1–21
[11] Soottitantawat, A., Bigeard, F., Yoshii, H., Furuta, T., Ohkawara, M. and Linko, P. (2005). Influence of emulsion and powder size on the stability of encapsulated limonene by spray drying. Innov Food Sci and Emerging Technol, 6:107–114.
[12] Loksuwan, J. (2007). Characteristics of microencapsulated β-carotene formed by spray drying with modified tapioca starch, native tapioca starch and maltodextrin. Food Hyd, 21: 928–935.
[13] Mc Hugh and Krochta (1994). Milk-protein-based edible films and coatings. Food Technol, 48(1), 97-103
[14] Khamidah, S.Z. (2018). Karakteristik mikrokapsul konsentrat asam lemah tak jenuh minyak ikan patin (Pangasius sp.). Bogor (ID): Institut Pertanian Bogor, Tesis.
[15] Yuliasari, S., Fardiaz, D., Andarwulan, N., and Yuliani, S. (2016). Karakteristik Enkapsulat Minyak Sawit Merah dengan Penambahan Beta Karoten. Informatika Pertanian, 25: 107-116.
[16] Fuchs M, Turchiuli C, Bohin M, Cuvelier ME, Ordonnaud C, Peyrat-Maillard MN, Dumoulin E. 2006. Encapsulation of oil in powder using spray drying fluidized bed agglomeration. *J Food Eng.*, **75**: 27–35.

[17] Cano-Chauca, M., Stringheta, P.C., Ramos, A.M. and Cal-Vidal, J. (2005). Effect of the carriers on the microstructure of mango powder obtained by spray drying and its functional characterization. *Innovative Food Sci Emerg Technol*, **6**: 420-428.

[18] Cai, Y.Z. and Corke, H. (2000). Production and properties of spray-dried Amaranthus betacyanin pigments. *J Food Sci*, **65**: 1248-1252.

[19] Quynh, N.T.N., Hai, T.C., Man, P.V. and Thanh, L.T. (2016). Effect of wall material on the property of gac oil spray-dried powder. *J Nutr Food Sci*, **6**:5.

[20] Yuliasari, S. (2015). Sifat fungsional enkapsulat nanoemulsi minyak sawit merah dengan pengayaan beta karoten sebagai ingredien pangan. Bogor : Institut Pertanian Bogor, Disertasi.

[21] Botrel, D.A., Fernandez, R.V.D., Borges, S.V. and Yoshida M.I. (2014). Influence of wall matrix systems on the properties of spray dried microparticles containing fish oil. *Food Res*, **62**: 344-352.

[22] Dewanti, T., Harijono, and Nurma. (2015). Tepung bubur sereal instan metode ekstrusi dari sorgum dan kecambah kacang tunggak (kajian proporsi bahan dan penambahan maltodekstrin). *J Teknologi Pertanian*, **3**(1) : 35-44.