Fish oil extraction as a by-product of Tilapia (*Oreochromis* sp.) fish processing with dry rendering method

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Abstract. Tilapia (*Oreochromis* sp.) is the raw material for the fish fillet industry. The fish fillet process has a by-product in the form of fish viscera which is used for tilapia fish oil production. The quality of tilapia oil in Indonesia is low and only used as an additive to animal feed. The oxidation and free radicals in fish oil can be affected by high temperature and period of extraction. The purpose of this study is determine the best temperature and period of extraction with the dry rendering method which can produce fish oil with quality characteristics according to International Fish Oil Standards (IFOS). Not all of the combinations of temperature and extraction time were suitable, but in the treatment of tilapia fish oil with a temperature of 50°C, 1 hour, the FFA value was 1.18%, PV 7.51mEq / kg, p-AV 5.57mEq / kg, and TOTOX 20.59mEq / kg. The quality characteristics of tilapia fish oil from bleaching magnesium silicate have met the IFOS requirements.

1. Introduction

*Oreochromis* sp. also known as tilapia is a freshwater fishery commodity with a production rate of 1.15 million tons in 2017. This value has increased by 3.6% compared to 2016 [1]. Tilapia is commonly used for raw material of the fillet industry and it produces by-products such as head, tail, skin, and viscera. The by-product of fish fillet production can reach a total of 70%. Based on this amount, with the tilapia fish production data in 2017, the by-product of tilapia fillets 770 500 tons. This by-product has contains fat with omega-6 and omega-9 fatty acids. Suseno [2] stated that tilapia fish oil contains 22.24% monounsaturated fatty acids (MUFA) and 18.15% polyunsaturated fatty acids (PUFA). This fatty acid content is potential for fish oil production.

Estiasih [3] reported that fish oil is a fatty component found in fish body tissue, which is extracted in the form of oil. Fish oil is a high source of fatty acids in omega-3 and omega-6 [4]. The potential benefits of fish oil are anti-inflammatory, anti-thrombotic, improving cognitive function, prevention and treatment of cardiovascular disease, and the effect of lipid metabolism [5]. Quality fish oil based on the International Fish Oil Standards (IFOS) [6] has a free fatty acid (FFA) ≤ 1.50%, peroxide value (PV) ≤ 5.00 mEq / kg, p-anisidin value (p-AV) ≤ 20.00 mEq / kg, and total oxidation (totox) ≤ 19.50 mEq / kg. The production of tilapia fish oil in Indonesia does not meet the food standard. The extraction process is carried out by using the wet rendering method, the heating process of the condenser machine which is connected through pipes in the storage container. The temperature in the heat pipe is 105°C. The oil extracted by this method contains a peroxide value, free fatty acid, and total oxidation that does not meet the IFOS [7]. The fish oil production process using high temperatures (> 70°C) is still a major problem because it can reduce the quality of fish oil which is unstable to heat.
Estiashih [3] states that the use of high temperatures and inaccurate extraction times can trigger the formation of free radicals and secondary oxidation of the decomposition of hydroperoxide compound. The fish oil production process includes extraction and refining. The extraction process using low temperatures followed by refining is one solution to improve the fish oil produced quality. Richard [8] state that low temperature is a temperature of less than or equal to 158°F or 70°C. Extraction of tilapia (Oreochromis niloticus) fish oil as a by-product of the fish fillet industry has been carried out by [2] with the wet rendering extraction method the temperature 25-90°C for 15-45 minutes. However, the quality characteristics of tilapia fish oil produced do not meet IFOS standards. Therefore, an oil extraction research was carried out from tilapia (Oreochromis sp.) by-product of the fish fillet industry using the dry rendering method with a temperature treatment of 40, 50 and 60°C for 1, 2, and 3 hours. The purpose of this study is to determine the best extraction temperature and period with the dry rendering method which can produce fish oil with quality characteristics according to IFOS [6].

2. Material and methods

2.1. Material and equipment

Tilapia viscera by-product of the fillet industry of PT Kurnia Mitra Makmur Purwakarta and magnesium silicate (MgO.nSiO₂.xH₂O) are the main materials. Other materials were chemicals for the analysis of quality characteristics fish oil such as potassium iodide (KI), 95% ethanol, distilled water, phenolphthalein indicator (PP indicator), chloroform (CHCl₃), 0.1 N potassium hydroxide (KOH), 1% starch indicator, glacial acetic acid (CH₃COOH), sodium thiosulfate (Na₂S₂O₃) 0.1 N, and isoctane. The equipment used were a knife, container, blender (Miyako BL-152 PF/ AP), heating drying oven (DHG-9053A model), micropipette (Gilson), Erlenmeyer flask (Iwaki), test tube, spatula, volumetric pipette, Mohr pipette, burette (Pyrex, Germany), bulb, beaker glass, digital scale (Chq, Taiwan, 0.01 gram), aluminum foil, electric stove (Maspion, Japan), thermometer (Pyrex, Germany), measuring cup, spectrophotometer device (Shimadzu UV spectrophotometer Pharmaspec 1700), and gas chromatography (Shimadzu, Japan, Model GC2010 with cyanopropyl methyl sil (capillary column) column, column dimensions p = 60 m, Ø = 0.25 mm, 0.25 m film thickness), centrifugation (PLC series), and freezer.

2.2. Methods

The tilapia viscera was prepared and analyzed for chemical composition (AOAC [9]) followed by extraction. Extractions of viscera tilapia oil refer to [10]. The extraction process was carried out by modifying [10] using an oven with different temperature treatments, namely 50, 60, and 70°C for 1, 2 and 3 hours. After dry rendering extraction treatments, the sample was continued by pressing. The pressed process to obtain a liquid fraction. The liquid fraction was centrifuged at a speed of 11200 xg for 10 minutes at 25°C to separate oil fractions and water. The centrifuged oil was stored in a light-tight bottle and stored in a freezer at -18°C. Extracted oil was then analyzed for yield value and quality characteristics which include the value of free fatty acids [11], peroxide number [12], p-anisidin number [13], and total oxidation [14] according to IFOS [6] to determine the best treatment. The best-extracted oil was bleached with magnesium silicate, which refers to [15] at 5% (w/w).

3. Result and discussion

3.1. Tilapia viscera chemical composition

| Parameter | Oreochromis sp | O. niloticusᵃ | O. niloticusᵇ |
|-----------|----------------|---------------|---------------|
| Moisture (%) | 72.54 ± 0.74 | 64.58 | 72.01 |
| Ash (%) | 1.10 ± 0.05 | 3.01 | - |
| Lipid (%) | 14.91 ± 0.16 | 12.98 | 1.01 |
| Protein (%) | 6.90 ± 0.08 | 13.24 | 15.36 |

ᵃSeseno et al. (2015), ᵇDewi and Ibrahim (2008)
The chemical composition analysis results showed that tilapia viscera contains 14.91 ± 0.16% of fat, 72.54 ± 0.74% of moisture, 1.10 ± 0.05% of ash, and 6.90 ± 0.08% of protein (Table 1). The content of tilapia viscera fat is higher than Oreochromis niloticus meat and by-product fillet industry, 1.01% [16] and 12.98% [2], respectively. The difference in the chemical composition of the same fish species can be influenced by several factors, such as environment, harvest age, and diet [17].

3.2. Tilapia Oil Yield

The tilapia oil extraction results showed that the fish oil yield increased with the increasing temperature and period of extraction. Wu and Peter [18] stated that temperature can affect the yield produced. Nugroho et al., [19] stated that heating cause protein denaturation and makes it easier for oil to be extracted. Besides, the longer the period extraction, the more oil is produced [3]. The extracted tilapia oil yield was in the range of 3.31% to 7.25% (Figure 1). This result is higher than the tilapia (O. niloticus) oil by-product of fillets extracted by the wet rendering method and steam jacket method, namely 1.49-6.44% [2] and 25.3% [19], respectively. The resulting difference in yield can be influenced by several factors, namely stage fish, environment, fish species, feed, and extraction method [17]. Treatment of extraction temperature and period can affect protein denaturation so that oil is extracted maximally [20].

![Figure 1. Yield of tilapia oil](image)

3.3. Tilapia oil quality characteristics

3.3.1. Tilapia Free fatty acids (FFA)

Fish oil extracted then carried out quality characteristics according to IFOS [6]. Free fatty acids (FFA) are fatty acids that are in the free acid group and are not bound to triglycerides [21]. Free fatty acids are a parameter to determine the quality of the fish oil produced. The content of the overall treatment combinations FFA was in the range of 1.12-4.01% (Figure 2). The free fatty acids content of tilapia fish oil tends to increased with the increasing temperature and period of extraction. The hydrolysis of triacylglycerol caused increased FFA content [22], as well as the oxidation by free radicals and double [2] using the wet rendering method for tilapia, 3.85-7.15%. The value of standard FFA according to IFOS [6] is ≤ 1.5%. FFA value of the extracted tilapia oil at a temperature of 5°C for 1 hour was within IFOS.
3.3.2. *Tilapia Peroxide Value (PV)*

Peroxide value analysis is used to determine the hydroperoxide content that results from primary oxidation. The high hydroperoxide value indicates low oil quality. Hydroperoxide can be formed by the binding of unsaturated fatty acids with free oxygen [24]. The oxidation process is accelerated by supporting factors, such as temperature and light [2]. The peroxide value of tilapia oil tends to increase with the increasing extraction temperature and period. [25] state that the oxidized by oxygen of unsaturated fatty acids can increased PV of fish oil. [22] reported that the peroxide value in tilapia oil from steam jacket extraction was 3.96-9.35 mEq/kg. The peroxide value of all treatment combinations extraction temperature and period have been higher than IFOS [6].

3.3.3. *Tilapia p-Anisidine Value (p-AV)*

The secondary oxidation of oil can be indicated by the p-anisidine value[3]. Secondary oxidation occurs due to the decomposition of hydroperoxide components which produce products in the form of ketones, aldehydes, alcohols, lactones, acids, hydroxy components, dienal, hydrocarbons, epoxides, and monomer or polymer compounds which are measured by p-AV [22]. The anisidine value of tilapia fish oil tends to increase with increasing temperature and extraction time (Figure 4). The p-anisidine value of extracted tilapia fish oil was in the range of 5.57 to 25.59 mEq/kg. The treatment at 50°C for 1 hour was the lowest p-anisidine value. This result is higher than tilapia fish oil (*O.niloticus*) from wet
extraction rendering temperature of 25-90°C for 25-45 minutes, namely 4.2-6.5 mEq/kg [2]. p-AV can be affected by several factors, such as extraction temperature and period. The temperature during the extraction process can cause pigment oxidation and produce a dark color in fish oil [23]. Aldehyde compounds and their derivatives due to oxidation can affect the rancidity of oil [26].

Figure 4. p-Anisidine value of tilapia oil

3.3.4. Tilapia Total Oxidation (TOTOX)
The total value of oxidation is the sum of twice the peroxide value and the anisidine value of fish oil. Oil that has a high peroxide value and p-anisidin number is an indicator of low oil quality [27]. According to IFOS [6], TOTOX should be less than 26 mEq/kg. The extracted tilapia oil TOTOX increased with increasing temperature and period of extraction was in range of 20.59-66.40 mEq/kg. Similar results were shown by [2] namely totox tilapia fish oil (O. niloticus) from dry rendering extraction at a temperature of 25-90°C for 25-45 minutes tends to increased with increasing extraction temperature and period. The total oxidation of extraction at 50°C for 1 and 2 hours has been met the IFOS requirements. Based on the quality characteristics of the best tilapia fish oil obtained from the extraction temperature of 50°C for 1 hour, it has FFA, p-AV, and TOTOX values that meet the IFOS requirements. The peroxide value in the best-extracted tilapia fish oil was then performed with 5% magnesium silicate bleaching. Tilapia oil before and after bleaching is presented in Table 2.

Figure 5. Total oxidation of tilapia oil
The bleaching process using adsorbents can absorb impurity components in oil such as colloid suspensions (gum and resin), pigments, and oil degradation products (peroxide) [21]. PV of tilapia (*Oreochromis* sp.) Oil was 4.76 ± 0.03 mEq/kg with a decrease of 36.62%. [15] reported that the use of magnesium silicate was able to reduce PV of sardines by 59.9%. The use of magnesium silicate can reduce pigment components, free fatty acids, and other impurity components in oil [28]. The use of 5% (w/w) magnesium silicate in Table 2 was able to reduce FFA to 0.25 ± 0.03%, p-AV to 4.99 ± 0.38 mEq/kg, and TOTOX to 14.52 mEq/kg. The characteristics of fish oil from magnesium silicate bleaching have met IFOS standards (Table 2).

### Table 2: The tilapia oil quality characteristics from magnesium silicate bleaching

| Parameter       | Before      | After       | IFOS 2014 |
|-----------------|-------------|-------------|-----------|
| FFA (mEq/kg)    | 1.18 ± 0.03 | 0.25 ± 0.03 | ≤ 1.5     |
| PV (mEq/kg)     | 7.51 ± 0.64 | 4.76 ± 0.03 | ≤ 5.0     |
| p-AV (mEq/kg)   | 5.57 ± 0.52 | 4.99 ± 0.38 | ≤ 20      |
| TOTOX (mEq/kg)  | 20.59 ± 0.76| 14.52 ± 0.45| ≤ 26      |

#### 4. Conclusion

Tilapia oil extracted at 50°C for 1 hour has the best quality characteristics with a yield of 3.31%. Tilapia fish oil from bleaching of magnesium silicate has FFA, PV, p-AV, and TOTOX that meet International Fish Oil Standards (IFOS) requirements.

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