Composition of Volatile Flavor Compound on Fresh Asian Redtail Catfish

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ABSTRACT

The purpose of this research was to identify the composition of volatile flavor compound of asian redtail catfish (Hemibagrus nemurus). The method of this research was experimental method of fresh sample. The extraction sample method in this research was Solid Phase Microextraction (SPME) and used Gas Chromatography-Mass Spectrometry (GC/MS) to detect the compound on the sample. The volatile flavor compound were successfully detected with total 23 compounds on fresh sample and mostly the compounds were derived from hydrocarbons, aldehyde, alcohols, ketones, ester, and others. The highest proportion that detected was hexanal compound (26.78%) The proximate analysis was also identified. The result of proximate analysis on fresh sample were water content (79.21%), ash (1.09%), protein (17.21%), and fat (2.17%)

Keywords: Asian redtail catfish; fresh; volatile flavor compound; proximate.

1. INTRODUCTION

Fisheries production in Indonesia keep increasing every year based on Marine and Fisheries Ministry’s data in 2018. Total of national fisheries production in 2017 was 23 million ton and increased in 2019 with total 23.86 million ton [1]. Based on those data, it could
give a potential to the fisheries production will increase more.

Asian redtail catfish (*Hemibagrus nemurus*) is one of catfish that lives in fresh water as river, lake, swamp, and sometimes could be found on brackish water [2]. Asian redtail catfish is Indonesia’s endemic and was found in several place as Java, Sumatra, and Kalimantan [3]. As we know that fish have a lot of protein and also fat that could affect flavor volatile compounds that were formed [4], [5].

Flavor is sensation of food material that was accepted by human sense such as taste and aroma that affects human senses through the respiratory and when consumed [6],[7],[8]. Flavor compound has two category, Flavor compounds consist of volatile flavor compounds that give a sensation of aroma and non-volatile compounds that provide a sensation of taste. Flavor could change due to changes in time and processing method [9].

Volatile flavor is a component of flavor compounds that gives an aroma sensation that has the ability to evaporate quickly and can give an initial impression or called top notes [10], [8]. Aroma is one of the important aspects in the fish processing because the quality level of fish can be determined from the aroma which produced from fish. Generally, the composition of volatile flavor compounds in fish comes from groups of aldehydes, alcohols, ketones, acids and hydrocarbons [11].

Currently, research on the composition of volatile flavor compounds in fishery products in Indonesia has not been carried out. This research is important to study the composition of volatile flavor compounds in fish which is used as a basic data on the aroma composition of fish commodities in Indonesia and can be developed or applied to produce a new product. Therefore, based on the background that has been described, research on the composition of volatile compounds of fresh Asian redtail fish needs to be carried out.

2. MATERIALS AND METHODS

2.1 Time and Place of Research

The research was done on March to June 2021. this research was done in three places. the first one was at the Laboratory of Fisheries Processing Faculty of Fisheries and Marine Science, Padjadjaran University for the sample preparation. Analysis of volatile flavour compound was done at the Laboratory of Flavor Balai Besar Riset Tanaman Padi Sukamandi, Subang. Proximate analysis was done at Laboratory of Konservasi Satwa Langka dan Harapan, Pusat Antar Universitas (PAU).

2.2 Research Materials

The tools that used in this research were alumnum foil, cling wrap, cool box, gas chromatography-mass spectrometry (GC/MS), label, gas stove, kjeldahl set, soxhlet, steamer, fillet knife, zip lock, furnace, digital scale, waterbath. The materials used in this research were fresh asian redtail catfish sample, water, HCl,CuSO₄, K₂SO₄, NaOH , H₂SO₄, and kloroform.

2.3 Research Methodology

The method that used in this research was experimental method on fresh fish sample. The sample was extracted with solid phase microextraction (SPME) method to identify volatile flavor compounds using tools called Gas Chromatography-Mass Spectrometry (GC/MS). fresh sample was also tested for its proximate consisting of water, ash, protein, and fat content as proximate analysis data.

2.4 Research Procedures

2.4.1 Sample preparation

Sample preparation was carried out at the Laboratory of Pengolahan Hasil Perikanan, Faculty of Fisheries and Marine Science, Padjadjaran University. the sample was cleaned first and then cut into fillet shapes. the weight of the sample for identification of volatile compounds was 50 grams and 40 grams for the proximate analysis. The sample was packed with alumunium foil and then put label for naming the sample and covered with cling wrap and then put it in zip lock plastic.Samples were taken with coolbox to its destinations to be identified.

2.4.2 Identification of volatile flavor compounds

Identification method was used based on research from Guillen and Emeralde (2002) which was modified by Pratama (2011). Identification of volatile flavor compounds using Gas Chromatography and Mass Spectrometry
(GC/MS) tools. Sample extraction was carried out using the Solid Phase Micro Extraction (SPME) method to evaporate volatile compounds in the sample, using DVB/Carboxen/Poly Dimethyl Siloxane fiber as an absorbent for volatile flavor compounds. A total of 1.5 g of the refined sample was put into a vial for SMPE size 22 ml. The extraction temperature of samples was 40°C for 45 minutes each (in water bath). Fiber was inserted into the sample injector of the GC/MS tool and then set the tool. The setting of the tool were: GC column was HP-INNOWax (30 mx 250 m x 0.25 m), helium carrier gas, initial temperature was 45°C (hold 2 minutes), increasing temperature of 6°C/minute, final temperature of tool was 250°C (hold 5 minutes) with time 32,775 minutes. The results obtained in the form of a chromatogram [12].

2.4.3 Proximate analysis

Proximate analysis was also carried out on the sample consists of water, ash, protein, and fat contents based on AOAC (Association of Official Analytical Chemist) procedures.

2.5 Data Analysis

The mass spectra of the detected compounds were compared with the mass spectra patterns contained in the data center or NIST library version 0.5a (National Institute Standard and Technology). The data of volatile flavor compounds were analyzed using Automatic Mass Spectral Deconvolutin and Identification System (AMDIS) software (Mallard and Reed 1997) to recorrect from the compound mass spectra data. The data of both analyzes were explained in a description.

3. RESULTS AND DISCUSSION

3.1 Identification of Volatile Flavor Compounds on Fresh Asian Redtail Catfish

Identification of volatile flavor compounds was done to see the forming compounds of the aroma of the sample. Based on the identification of volatile flavor compounds using the GC/MS, the samples produced 23 compounds that consists 6 compounds of hydrocarbon, 7 compounds of aldehydes, 8 compounds of alcohol, and 1 compound of ketone and ester each. The most dominating compound in fresh Asian redtail catfish is hexanal compound with a proportion of the compound was 26.78%. The composition of volatile flavor compounds in fresh Asian redtail catfish is shown in Table 1.

Hydrocarbons are derived from the autoxidation of lipids and the decomposition of carotenoids [13]. Hydrocarbon group has a high aroma threshold so the contribution to the aroma in fish is usually less [14]. Compounds with the largest proportion in the hydrocarbon group is naphthalene compound, which are 20.77%. The compound was detected at 18.5236 minutes with an area that was 1998555. Other compounds that detected were d-limonene (0.3%), cyclohexene, 1-methyl-4-(1-methylethenyl)-, (S) - (0.3%) and so on (Table 2).

Naphthalene compound was one of the compounds belongs to the hydrocarbon group that has largest proportion detected in sample. This compound is basically Polycyclic Aromatic Hydrocarbons (PAHs) produced from natural combustion, especially those that found in the air, such as burning biomass, gasoline or fuel, and tobacco, most of that have toxic properties [15]. PAH compounds contained in foods usually occur due to contamination. The chemical composition of food can be formed due to environmental factors such as temperature, weather, and environmental conditions. Naphthalene compounds detected in fish are caused by contamination from various aspects such as industrial waste, combustion, fertilizers, plastics, and so on [16]. Naphthalene compounds detected in fresh Asian redtail catfish were probably due to contamination with environmental pollutants. Other compounds such as cyclohexene, 1-methyl-4-(1-methylethenyl)-, (S)-, and hexadecane were also detected in other fish such as gourami and carp [17], [5].

The aldehyde group in fish meat is produced from the oxidation of the carbon-carbon double bonds of unsaturated fatty acids present in fish meat and saturated fatty acids [17]. The aldehyde group is considered a product of lipid autoxidation [18]. The aldehyde group generally has a characteristic like fruity aroma [18]. The aldehyde group is a group of volatile flavor compounds that contribute especially to food such as meat because their aroma threshold is lower than alcohol and ketones [14].

The results of the identification of volatile flavor compounds on sample showed that in the aldehyde group, the compound with the largest proportion was hexanal compound, which was 26.78%. The compound with the second largest
The green sensation like aroma 

Other compounds, such as nonanal, came from the oxidation of oleic acid in fish which give some sensation like green, fatty, and orange flavor. The pentanal compound was derived from the oxidation of linoleic acid which produce a flavor description like sharp, sour, and mushroom-like, while the 4-heptenal and 2,6-Nonadinal, (E,Z)-compounds were produced from linoleic acid and eicosapentenoic acid which produce a flavor description of green and fishy [21]. The green flavor gives the impression of freshly cut leaves or grass [24].

ketone is produced through the oxidation of unsaturated fatty acids, amino acid degradation, or microbial oxidation. Ketones are an important class of volatile compounds in fresh fish and contribute to a fresh fish-like aroma [21], [18]. Characteristics aroma of ketone are fruity and floral aromas [19]. The results of the identification of volatile flavor compounds on sample showed that in the ketone group, the compound that detected was 2,3-Octanediol with 21.36%. The compound is described as a metallic aroma. Metallic flavor or aroma is described as a distinctive aroma on a metal surface [24]. The compound 2,3-Octanediol was also found in brown trout and rainbow trout [25], [21].

Alcohols are formed by the breakdown of secondary hydroperoxides from fatty acids [11]. The alcohol group usually has a small

### Table 1. Composition of volatila flavor compounds on fresh asian redtail catfish

<table>
<thead>
<tr>
<th>Group</th>
<th>RT</th>
<th>Compound</th>
<th>Area</th>
<th>Proportion (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrocarbon</td>
<td>18,5236</td>
<td>Naphthalene</td>
<td>1998555</td>
<td>20.77</td>
</tr>
<tr>
<td></td>
<td>14,775</td>
<td>D-Limonene</td>
<td>28692</td>
<td>0.30</td>
</tr>
<tr>
<td></td>
<td>14,7803</td>
<td>Cyclohexene, 1-methyl-4-(1-methyleneyl)-(S)-</td>
<td>28692</td>
<td>0.30</td>
</tr>
<tr>
<td></td>
<td>16,5696</td>
<td>Undecane</td>
<td>27611</td>
<td>0.29</td>
</tr>
<tr>
<td></td>
<td>20,5395</td>
<td>Hexadecane</td>
<td>12350</td>
<td>0.13</td>
</tr>
<tr>
<td></td>
<td>19,3323</td>
<td>Azulene</td>
<td>9558</td>
<td>0.10</td>
</tr>
<tr>
<td>Aldehyde</td>
<td>9,7814</td>
<td>Hexanal</td>
<td>2576927</td>
<td>26.78</td>
</tr>
<tr>
<td></td>
<td>12,4721</td>
<td>Benzaldehyde, 4-ethyl-</td>
<td>521676</td>
<td>5.42</td>
</tr>
<tr>
<td></td>
<td>16,3789</td>
<td>Nonanal</td>
<td>272855</td>
<td>2.84</td>
</tr>
<tr>
<td></td>
<td>5,6697</td>
<td>Pentanal</td>
<td>8946</td>
<td>0.09</td>
</tr>
<tr>
<td></td>
<td>15,2207</td>
<td>4-Heptenal</td>
<td>4851</td>
<td>0.05</td>
</tr>
<tr>
<td></td>
<td>7,691</td>
<td>Butanal</td>
<td>1934</td>
<td>0.02</td>
</tr>
<tr>
<td></td>
<td>16,4438</td>
<td>2,6-Nonadialen, (E,Z)-</td>
<td>993</td>
<td>0.01</td>
</tr>
<tr>
<td>Keton</td>
<td>13,6598</td>
<td>2,3-Octanediol</td>
<td>2055700</td>
<td>21.36</td>
</tr>
<tr>
<td>Alcohol</td>
<td>13,6215</td>
<td>2-Octen-1-ol</td>
<td>1680652</td>
<td>17.47</td>
</tr>
<tr>
<td></td>
<td>16,3641</td>
<td>1-Hexanol, 2-ethyl-</td>
<td>230621</td>
<td>2.40</td>
</tr>
<tr>
<td></td>
<td>14,0193</td>
<td>1-Butanal, 4-ethoxy-</td>
<td>45566</td>
<td>0.47</td>
</tr>
<tr>
<td></td>
<td>15,6717</td>
<td>1-Hexanol</td>
<td>36429</td>
<td>0.38</td>
</tr>
<tr>
<td></td>
<td>15,667</td>
<td>1-Pentanol</td>
<td>27796</td>
<td>0.29</td>
</tr>
<tr>
<td></td>
<td>15,8612</td>
<td>1-Octanol, 2-methyl-</td>
<td>27796</td>
<td>0.29</td>
</tr>
<tr>
<td></td>
<td>16,1977</td>
<td>1-Octanol, 2-butyl-</td>
<td>12062</td>
<td>0.13</td>
</tr>
<tr>
<td></td>
<td>9,8197</td>
<td>1-Penten-3-ol</td>
<td>8545</td>
<td>0.09</td>
</tr>
<tr>
<td>Ester</td>
<td>16,0088</td>
<td>Carbonic acid, nonyl prop-1-en-2-yl ester</td>
<td>3737</td>
<td>0.04</td>
</tr>
</tbody>
</table>

*RT: Retention Time (minute)
contribution to the flavor of the food unless it has a high concentration or is an unsaturated alcohol. The alcohol group generally has a distinctive plant and earthy aroma [18]. Aromas such as earthy give the effect of humus or moist soil [24]. The results of the identification of the volatile flavor compounds of sample showed that the detected compounds consisted of 7 compounds. Compound 2-Octen-1-ol is the compound with the largest proportion with 17.47% followed by 1-Hexanol, 2-ethyl- with 2.40%. The compound 2-Octen-1-ol affects the level of distinctive aroma of fish and 1-Hexanol is described as a grassy flavor. These two compounds were also found in freshwater fish such as grass carp [26].

The results of identification of volatile flavor compounds in fresh asian redtail catfish that belongs to the ester group was Carboxic acid, nonyl prop-1-en-2-yl ester with a proportion of 0.04%. The ester group in fish comes from the results of the acid and alcohol esterification process formed from fat metabolism [5].

Table 2. Proximate analysis results of fresh asian redtail catfish

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Results (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water Content</td>
<td>79.21±0.00</td>
</tr>
<tr>
<td>Ash Content</td>
<td>1.09±0.03</td>
</tr>
<tr>
<td>Protein Content</td>
<td>17.21±0.17</td>
</tr>
<tr>
<td>Fat Content</td>
<td>2.17 ±0.12</td>
</tr>
</tbody>
</table>

* The value is based on the average value of two repetitions with the standard deviation

3.2 Proximate Analysis

Proximate analysis was done on sample of fresh asian redtail catfish. The proximate analysis included water, ash, protein, and fat content. The results of the proximate analysis are shown in Table 2.

3.2.1 Water content

Water content is used to determine the amount of water contained in food. Water content is one of the parameters to determine the quality of a food ingredient such as durability, freshness level, and acceptance level [27], [28], [29]. Based on the proximate test on fresh asian redtail catfish, the water content of sample and its standard deviation is 79.21±0.00% According to research by Susilowati et al. (2017), the water content in asian redtail catfish is 78.06% [30].

3.2.2 Ash content

Ash content is used to determine the total minerals in foodstuffs that are not burned and become volatile substances (Hutomo et al. 2015). Based on the results of the proximate analysis, the ash content analysis on sample was 1.09±0.03%. According to research by Susilowati et al. (2017), the ash content of asian redtail catfish was 1.30% [30]. Ash content in a food is influenced by the type of food and the method of ashing [17]. The use of water during the washing process can reduce the mineral content of foodstuffs because minerals dissolve in water. Processing method can also affect the availability of minerals in foodstuffs [31].

3.2.3 Protein content

Protein is an important nutritional content, especially for humans for the growth process and is widely contained in foodstuffs such as fish. Protein content can be different depends on several factors such as the type of species, the environment, nutrients, fishing methods, storage and processing methods [17].

The results of protein content on fresh asian redtail catfish was 17.21±0.17%. According to research by Susilowati et al. (2017), the protein content of asian redtail catfish was 17.59% [30]. High temperatures during the steaming process can cause protein denaturation [32]. According to Buckle et al. (1987), protein content in fish is also influenced by water content and fat content. The higher protein content in fish, the lower the water content can be [27].

3.2.4 Fat content

Fat is a nutritional content that used as a source of energy for the human body. Analysing the fat content of a food ingredient is used to determine the quality of the food ingredient. Fats contribute to the formation of volatile compounds. The amount of fat content in foodstuffs can affect the rancidity of these foodstuffs due to the fat oxidation process [29]. The results of the fat content on sample was 2.17 ± 0.12% According to research conducted by Susilowati et al. (2017), the fat content in asian redtail catfish was 4.51% [30]. Similar to the protein content in fish, the fat content in fish is also influenced by the water content in the fish.

4. CONCLUSION

Volatile flavor compounds identified in fresh asian redtail catfish were 23 compounds
consisting of 6 compounds of hydrocarbon, 7 compounds of aldehydes, 8 compounds of alcohol, and 1 compound of ketones and esters each. The most dominant volatile flavor compounds in asian redtail catfish was hexanal compound with a proportion of 26.78%. This compound has the characteristics of being green-like, pungent, fatty or rancid. The results of the proximate test on fresh asian redtail catfish were water content of 79.21%, ash content of 1.09%, protein content of 17.21%, and fat content of 2.17%.

CONSENT
As per international standard or university standard, participants' written consent has been collected preserved by the author(s).

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COMPETING INTERESTS
Authors have declared that no competing interests exist.

REFERENCES