Global Scientific JOURNALS

GSJ: Volume 8, Issue 7, July 2020, Online: ISSN 2320-9186
www.globalscientificjournal.com

VOLATILE COMPONENTS OF NARROW-BARRED SPANISH MACKEREL (SCOMBEROMORUS COMMERSON) MEAT WASH-WATER BROTH

Rusky Intan Pratama*[1], Iis Rostini[1], Yuniarti MS[2], Damar Pratama Putra[3]

[1] *Staff at Fisheries Product Processing Laboratory, Faculty of Fisheries and Marine Science, Universitas Padjadjaran, West Java, Indonesia*
[2] *Staff at Faculty of Fisheries and Marine Science, Universitas Padjadjaran, West Java, Indonesia*
[3] *Graduate student at Faculty of Fisheries and Marine Science, Universitas Padjadjaran, West Java, Indonesia*
*) *E-mail: ruskyisblue@gmail.com*

Key Words
broth, flavor, mackerel, volatile components, volatile compounds

ABSTRACT

Volatile components generally affect the overall aroma of food commodities including fisheries commodities such as narrow-barred Spanish mackerel. This research aims to identify volatile components of broth which prepared from mackerel fish meat (*Scomberomorus commerson*) washed water. The mackerels were collected from the local fish landing site in Karangsong, Indramayu, West Java. The method used in this study was to identify volatile compounds using Gas Chromatography/Mass Spectrometry (GC/MS) on broth samples with Solid Phase Micro Extraction (SPME) to extract volatile components from the sample. To support the main volatile data, the samples were also analyzed for their proximate composition. The volatile components analysis successfully identifies as many as 121 compounds in mackerel washed water broth sample with *diethyl phthalate* is the highest proportion compounds. Furthermore, the proximate analysis results showed that the sample has 98.13% water content, 1.21% ash, 0.33% lipid, and 0.33% protein content.

1. INTRODUCTION

Mackerel fish are commonly found in several areas of Indonesian waters. According to Indonesia's Ministry of Marine and Fisheries Affair, the national production volume of the sea captured Narrow-barred Spanish mackerel in 2017 reached up to 300.690.761 kg, slightly higher compared to sea captured volume of tuna which reached 293.233.165 kg [1]. Narrow-barred Spanish mackerel (*Scomberomorus commerson*) categorized as pelagic fish which commonly lives nearby the seawater surface. Most of those Spanish mackerel are wildly caught from the eastern part of Indonesia (Maluku Province).

In Indonesia, narrow-barred Spanish mackerel or *tenggiri* fish in the local language is commonly used as the main ingredient for various traditional and modern culinary dishes or processed fish-based products such as *pempek, siomay, meatball, otak-otak,* and other fish jelly products. *Tenggiri* fish is wildly used due to the sharp and unique aroma characteristics it provides to the products. Fish based products made from mackerel fish are usually processed using high temperatures such as fried, baked, boiled, or steamed. Similar to other foodstuffs, fresh fish or fish-based products will have differences in chemical composition and their aroma characteristics. These affected by many internal and external contributing factors such as feed, environment, reproduction phase, and processing type used. Each processed product made from mackerel fish meat has its characteristics, especially in terms of flavor characteristics. [2] studied the volatile components in flavor concentrates from crayfish processing waste and found that the concentrates were still contained volatiles such as hydrocarbons, aldehydes, alcohol, and organic acid groups. Mackerel meat wash-water is considered as waste from the washing stage of the mackerel fish preparation stage. This water then processed into broth assuming it still has many flavors affecting compounds contained in it.

Flavour is a combination of taste and aroma and can be influenced by pain sensations (spicy, pungent), heat, cold, and real tactile sensations when food is consumed. Flavour is sensed by taste receptors in the mouth and aroma receptors in the nose. The flavor component is generally divided into two groups, the first one contributes to taste characteristics and the second one contributes to the aroma characteristics of a product. The flavor component which contributes to aroma is volatile components. These components are sensed by the aroma receptors of the olfactory organ in the nasal cavity [3], [4], [5]. To study the aroma characteristic of a product, volatile compounds identification method using solid-phase microextraction (SPME), and gas chromatography/mass spectrometry (GC/MS) is common to carry out [6]. [7]. Volatile compounds are generally derived from enzymatic reactions, lipid oxidation, microbial activity, thermal reactions products, or by-products, moreover, they can also derive from the environment [8]. Fresh fish are usually having fewer volatile compounds compare to processed one (using high temperature). This phenomenon can be happened mainly due to the formation of new volatile compounds after the heating process. The presence of thermal oxidation and decomposition of fatty acids, especially unsaturated fatty acids are one of the main contributing factors for these newly developed compounds [4], [9].

In the culinary world, a broth is known as a clear liquid deriving its essence from a combination of meats, vegetables, and herbs that have simmered in water for a long time [10] and usually used in soups. Broth's composition provides a characteristic flavor, various important nutrients such as protein, various minerals which are absorbed more easily), color, and believe to have several health-related properties such as fatigue and flu curing properties. Boiling in simmered heat will provide the desired broth sensory characteristics, furthermore, volatiles will be released from the food matrix. Thus, the identification analysis of its volatile components would provide significant information regarding broth aroma characteristics and its compound constituent which produced from mackerel meat immersed water.

2. MATERIALS AND METHODS

2.1 Samples Preparation

Narrow-barred Spanish mackerel fish samples were collected from the fish landing site in Karangsong, Indramayu, West Java. Sample preparation was carried out at the Fisheries Product Processing Laboratory, Faculty of Fisheries and Marine Sciences, Padjadjaran University. Proximate analyses were carried out in the Inter-University Centre Laboratory, Bogor Agriculture Institute, and volatile compound analysis were carried out in Flavour Laboratory, Indonesian Centre for Rice Research, Sukamandi, Subang, West Java.

Samples were transported to the laboratory using a cool box containing layers of bulk ice. Upon arriving, samples were then cleaned and filleted for the meat. The fillet was then weighed (500 grams each) and placed in a container filled with bulk ice. Afterward, the fillet was immersed and washed in aquadest water (in a stainless bowl) with 1:2 ratios for 5 minutes. After the fillet was removed, the remaining water was placed in a stainless steel pot and heated for 90 minutes using low heat simmered temperature (65^{o}C) [11]. Subsequently, the broth was cooled and filtered to remove its remained solid parts. The broth was then placed in a glass bottle after the volume has been measured and tightly closed. Samples were then placed in a cool box containing packed bulk ice 10-15°C and promptly transported to each analysis laboratory.

2.2 Volatile Component Analysis

Sample volatile compounds were analyzed by procedures according to modification from [6] procedure. Water-bath is used for sample extraction and Gas Chromatography (GC) (Agilent Technologies 7890A GC System) and Mass Spectrometry (MS) apparatus (Agilent Technologies 5975C Inert XL EI CI/MSD) is used for detecting and identifying the volatile compounds. The sample extraction method was carried out by Headspace/Solid Phase Micro Extraction (HS/SPME) using DVB/Carboxen/Poly Dimethyl Siloxane fiber (Fuse Silica Black with 75 µm thickness) which was preconditioned first before used. The sample's extraction time used was 80^{o}C for 30 minutes. GC column used was HP-5MS (30 m x 250 µm x 0,25 µm), helium carrier gas, the initial temperature was 45^{o}C (hold 2 minutes), temperature's escalation as much as 6^{o}C/minutes, the final device temperature was 250^{o}C (hold 5 minutes) with an overall running time 53,75 minutes.

2.3 Proximate Analysis

Samples proximate analysis were carried out according to [12] standard. Moisture was calculated based on the gravimetric method, and total inorganic content (ash %) through the combustion of organic matter in a muffle furnace. Total protein content was determined by the Kjeldahl method and calculated as % nitrogen x 6.25. Total lipid content was determined by the Soxhlet system and the results expressed in %.

2.4 Data Analysis

The obtained results from proximate analysis samples were calculated and showed as mean value and its deviation standards and then were discussed descriptively. Compound mass spectrums detected from GC/MS were compared with the mass spectrum pattern which was available in the computer database or NIST (National Institute of Standard and Technology) library 0.5a version. The resulting data from volatile compounds analysis were discussed descriptively based on the tentative identification (based on retention times, mass spectra and comparing them with library database) and semi quantification intensity of the compounds detected from analyzed samples was based on peak area counts arbitrary units [6], [25].

3. RESULTS AND DISCUSSION

3.1 Volatile Compounds

The extraction temperature used was 80^{o}C and this was picked due to the liquid state of the sample. At lower temperatures, least volatiles would be extracted from the samples [13]. If the sample is more solid such as fish meat, then the temperature used is usually lower [9]. The extraction temperature chosen was high enough to release volatile compounds from the liquid sample in the water-bath without making it over boiled. The analysis result showed that the Spanish mackerel immersed water broth sample has 121 volatile compounds identified. Most of the compounds detected can be categorized as major compound groups such as aldehydes, hydrocarbons, ketones, alcohols, esters, organic acids, and others (Table 1).

Table 1. Volatile compounds detected in Spanish mackerel wash-water broth and their concentration in percentage area counts proportion

Retention Time	Compounds	Area	Proportion (%)
	Aldehydes		
2.8643	Pentanal	37566925	3.28
2.7157	Butanal, 3-methyl-	10962508	0.96
12.58	Nonanal	10669617	0.93
8.513	Benzaldehyde	8128679	0.71
30.4774	Tetradecanal	4647807	0.41
4.6124	Hexanal	3928025	0.34
9.94	2,4-Heptadienal, (E,E)-	3772732	0.33
15.5173	Decanal	2242301	0.20
29.0504	Octanal, 2-(phenylmethylene)-	2179174	0.19
14.2211	2-Nonenal, (E)-	2003336	0.17
7.08	Methional	1887039	0.16
6.8719	Heptanal	1479277	0.13
11.266	2-Octenal, (E)-	1265100	0.11
17.1168	2-Decenal, (E)-	1195559	0.10
32.4752	Heptadecanal	305174	0.03
18.5498	2,4-Decadienal, (E,E)-	260498	0.02

Retention Time	Compounds	Area	Proportion (%)
10.9033	Benzeneacetaldehyde	7008640	0.61
17.4141	Benzeneacetaldehyde,-ethylidene-	4410480	0.39
14.3757	Benzaldehyde, 4-ethyl-	1289868	0.11
	Hydrocarbons (aliphatic, cyclic, aromatics)		
22.6822	Cyclopropane, nonyl-	92952715	8.11
13.3827	2-Hexene, 3-methyl-, (Z)-	20147268	1.76
13.4957	Cyclopropane, 1,1-diethyl-	16171566	1.41
19.6081	Tridecane, 4-methyl-	12645016	1.10
12.9903	Cyclohexane, 1,2,3-trimethyl-	11577174	1.01
14.5898	1-Nonene	9019404	0.79
12.247	Cyclopentane, ethyl-	5524812	0.48
27.4331	Cyclotetradecane	5496268	0.48
13.1508	4-Nonene	3875253	0.34
28.4439	2,6-Diisopropylnaphthalene	3753256	0.33
12.7525	Octane, 4-methyl-	3542086	0.31
28.2536	Naphthalene, 1,2,3-trimethyl-4-propenyl-, (E)-	2859349	0.25
14.9941	Naphthalene	2745884	0.24
15.2379	Dodecane	2302071	0.20
27.5995	1,7-di-iso-propylnaphthalene	2162076	0.19
23.2174	Pentadecane	1574268	0.14
29.4428	Hentriacontane	1159123	0.10
19.8579	Tridecane, 2-methyl-	1113752	0.10
29.2585	trans-1,2-Diphenylcyclobutane	1064257	0.09
27.8612	Heptadecane	1050410	0.09
22.3849	Undecane	1038744	0.09
28.0693	Heptadecane, 2,6-dimethyl-	961087	0.08
21.3325	Caryophyllene	948676	0.08
15.9038	Cyclohexane, 1,2,4-trimethyl-	865974	0.08
20.7201	Tetradecane	855219	0.07
15.7195	Undecane, 2,6-dimethyl-	853387	0.07
27.992	Dodecane, 2,6,10-trimethyl-	832578	0.07
18.0681	Tridecane	749841	0.07
25.126	Cetene	621060	0.05
28.872	Octadecane	590135	0.05
24.1985	Hentriacontane	566495	0.05
15.7849	Cyclohexene, 1,2-dimethyl-	533925	0.05
27.1595	Naphthalene, 1,2,3-trimethyl-4-propenyl-, (E)-	514543	0.04
26.886	Cyclotetradecane	424243	0.04
23.6693	Butylated Hydroxytoluene	418215	0.04

Retention Time	Compounds	Area	Proportion (%)
24.7395	Dodecane	412360	0.04
30.0077	Nonadecane	335698	0.03
32.0412	Decane	234088	0.02
26.7849	Hexadecane, 2,6,11,15-tetramethyl-	211793	0.02
31.9163	1-Nonadecene	167844	0.01
	Alcohols		
10.4514	1-Hexanol, 2-ethyl-	18636873	1.63
9.0541	1-Octen-3-ol	12932962	1.13
13.7811	(S)-(+)-6-Methyl-1-octanol	8286972	0.72
6.1346	1-Hexanol	4428785	0.39
25.7979	Benzenemethanol, 3,4-dimethoxy-	3364141	0.29
14.6968	Levomenthol	3242632	0.28
23.7644	2,4-Di-tert-butylphenol	2133313	0.19
16.2665	4,8-Decadien-3-ol, 5,9-dimethyl-	1294009	0.11
24.0141	Acetyleugenol	1036858	0.09
24.472	1-Octanol, 2-butyl-	858635	0.07
16.1833	4,8-Decadien-3-ol, 5,9-dimethyl-	736933	0.06
8.0849	Z-4-Dodecenol	727913	0.06
14.4827	1-Pentanol, 2,4,4-trimethyl-	675957	0.06
24.6028	Ethanol, 2-(octadecyloxy)-	644924	0.06
26.3093	Epicedrol	618875	0.05
21.2076	1-Decanol, 2-hexyl-	597921	0.05
17.2714	1-Undecanol	463954	0.04
18.2049	2-Hexadecanol	453434	0.04
	Ketones		
11.6822	Ethanone, 1-(1H-pyrrol-2-yl)-	13650602	1.19
12.4135	3,5-Octadien-2-one, (E,E)-	5383569	0.47
22.1292	trans-Geranylacetone	3858277	0.34
8.8757	Cyclobutanone, 2,3,3-trimethyl-	3109201	0.27
27.2487	-iso-Methyl ionone	2185719	0.19
26.4817	Benzophenone	1904580	0.17
9.7795	3-Hepten-2-one, 5-methyl-	1850878	0.16
17.9671	2-Undecanone	1645432	0.14
27.7422	1-(4-Benzylphenyl)ethanone	1386046	0.12
22.5633	Cyclohexanone, 2-ethyl-	1113984	0.10
20.8627	Bicyclo[3.2.1]oct-3-en-2-one, 4-methyl-	1086358	0.09
19.0254	Pyrethrone	636541	0.06
19.2752	3-Nonen-2-one	626024	0.05

Retention Time	Compounds	Area	Proportion (%)
	Organic acids		
3.2567	Acetic acid	25464791	2.22
24.906	Nonahexacontanoic acid	503396	0.04
26.119	Cyclooctaneacetic acid, 2-oxo-	320442	0.03
	Esters		
32.612	Hexadecanoic acid, methyl ester	336733	0.03
33.9796	Hexadecanoic acid, ethyl ester	162464	0.01
	Others		
25.6731	Diethyl Phthalate	545344890	47.61
7.5081	1H-Pyrrole, 1-ethyl-	44624933	3.90
3.8989	Pyrrole	30306739	2.65
5.0524	Pyrazine, methyl-	23472080	2.05
20.1909	Benzimidazole, 2-amino-1-methyl-	20343999	1.78
30.5666	Isopropyl myristate	5917226	0.52
12.1876	2,3-Dimethyl-5-ethylpyrazine	3521673	0.31
8.7865	Dimethyl trisulfide	3441508	0.30
30.2812	2-Ethylhexyl salicylate	3331074	0.29
16.8135	2-Isoamyl-6-methylpyrazine	3186700	0.28
12.033	Pyrazine, 2-ethyl-3,5-dimethyl-	1850956	0.16
31.5298	1-Butyl 2-isobutyl phthalate	1816860	0.16
31.286	Galoxolide	1623872	0.14
25.9525	2-(5-Aminohexyl)furan	1471155	0.13
26.6363	3-Hydroxypyridine monoacetate	1429604	0.12
7.2286	Pyrazine, 2,3-dimethyl-	1429098	0.12
33.4087	Butyl 2-ethylhexyl phthalate	1097664	0.10
18.3357	2-Acetylaniline	1081899	0.09
19.9887	Disulfide, di-tert-dodecyl	955063	0.08
17.7054	Benzenamine, N,N-diethyl-3-methyl-	946217	0.08
16.0941	Furan, 3-phenyl-	874495	0.08
14.9049	Pyrazine, 2,3-dimethyl-5-(1-propenyl)-, (E)-	867335	0.08
17.8006	Dihydrocarveole	828954	0.07
18.9006	2-Oxo-1-methyl-3-isopropylpyrazine	710277	0.06
21.7606	3,3'-Bis(1,2,4-oxadiazolyl)-5,5'-diamine	363367	0.03
11.4384	3-Hydroxypyridine-N-oxide	260380	0.02

According to Table 1. as much as 19 aldehyde compounds were detected in the sample with pentanal (3,28%) which has the highest compound proportion among other aldehydes. Aliphatic, cyclic, and aromatic hydrocarbons were also detected in samples, as much as 40 compounds, with nonyl-cyclopropane (8,11%) is the most abundant in these groups. Hydrocarbons in this sample were the highest in compound quantity compared to other groups of compounds. Alcohols group were also detected in the broth sample as much as 18 compounds with 2-ethyl-1-hexanol (1,63%) has the highest proportions among other alcohols. From the table, ketones were also detected as much as 13 compounds with1- (1H-pyrrol-2-yl) -ethanone (1,19%) is the compounds which has the highest proportion in this group. The following detected group was organic acids which were found as much as 3 compounds with acetic acid (2,22%) are the most abundant compound in this group. Moreover, as much as 2 compounds of esters group were also detected with hexadecanoic acid, methyl ester (0,03%) has the highest proportion. Other group compounds detected in the sample were 26 compounds, with the diethyl phthalate (47,61%) is the most abundant compound in broth sample.

Volatile compounds are derived from enzymatic reactions, lipid autoxidation, results of microbial activity, thermal reactions products, and their environment [9]. According to [14], [15], [16], aldehydes, aliphatic alcohols, and ketones groups are mostly formed by the oxidation of fats and fatty acids as well as by the degradation of amino acids during the processing process which involved heat. The product formed by this process depends on the fatty acids contained, the isomer of hydroperoxide formed, and the stability of the decomposition product. The broth sample in this study was produced by boiling the Spanish mackerel wash-water at shimmered temperature for (65-70ºC) in a period. This heating process and the chemical composition of wash-water affected by Spanish mackerel meat composition, in turn, will affect the remained sample's volatile component. Pentanal and 3-methyl-, butanal compounds detected in the sample were presumably produced from the oxidation of oleic and linoleic acids [8]. Pentanal was known to have green flavor characteristics [17], [18], and was also detected in fresh *patin* catfish (*Pangasius hypophthalmus*) and Nile tilapia (*Oreochromis niloticus*) volatile component [19].

Hydrocarbon compounds group was also detected in the broth sample from Spanish mackerel immersed water. The most volatile compounds identified from the hydrocarbon group were many straight and cyclic chain hydrocarbons. This group is known to derive from decarboxylation reaction and the splitting process of fatty acids carbon chains, a secondary reaction from carotenoid and unsaturated fatty acids thermal oxidations [4], [7], [20]. Nonyl-cyclopropane was the highest proportion detected in the broth sample. Other detected hydrocarbons such as pentadecane and tridecane were previously detected in squid [21], fresh *patin* catfish (*Pangasius hypophthalmus*), and Nile tilapia (*Oreochromis niloticus*) [19]. Hydrocarbon alkane group which have saturated chains are produced from decarboxylation and carbon chain separation from fatty acids [20].

As stated earlier, alcohol could be formed from fatty acids secondary hydroperoxide decomposition. Most alcohol are minor contributor for food flavor due to its high odor threshold [4], [7], [15], [20], [22], [23]. [14] stated that the lipid hydroperoxides splitting would produce alcohol, alkanes, alkenes, and alkynes. The most abundant alcohol compound in the broth sample is 2-ethyl-1-hexanol and previously was detected in fresh *patin* catfish (*Pangasius hypophthalmus*) and Nile tilapia (*Oreochromis niloticus*) volatile component [19]. Other alcohol compounds, 1-hexanol was previously detected in fresh and cooked silver carp [4], wild and cultured sea bream [8], raw black bream [6], Spanish mackerel fish and *patin* catfish (*Pangasius hypophthalmus*) [9], [19].

Ketones are likely to result from oxidation of lipids (especially unsaturated fatty acids) during heating, as well as thermal degradation, amino acid degradation, and Maillard reactions. All of those are possible mechanisms for the formation of ketone components. Ketones were previously found in freshwater lobsters have odour such as cream and cheese [2], [4], [6], [7], [9], [15], [19], [20], [23], [24], [25]. Ketone compound 1- (1H-pyrrol-2-yl) -ethanone (1,19%) or 2-acetylpyrrole is the most abundant ketones detected in this sample. Unfortunately, there has not been any information considering the detection of this compound in fisheries commodities. However, this compound is commonly found in cereals or cereal products and also present in cooked apple, asparagus, wheat bread, tea, roasted peanut, popcorn, potato chips, licorice, and other foodstuffs[1].

The organic acids group was also detected in the broth sample. Acid is an important decomposition of cellulose and hemicellulose [26]. Acid can also be formed by triglyceride hydrolysis in fish by lipase [27]. Acetic acid was the highest acid proportion of this group. Acetic acid compounds identified in the previous samples were also identified in the volatile fish sauce which contributes to vinegar-like and pungent aroma [28]. It was also identified in the volatile component of fresh and steamed milkfish (*Chanos chanos*) [29].

Two esters were detected in the broth sample, none of them were in a large proportion (0.01-0.03%). It was known that the ester group compound found in fish samples, is derived from lipids thermal degradation products [6], [20]. Hexadecanoic acid, methyl ester or methyl palmitate is a fatty acid methyl ester which has a role as a metabolite[2].

Volatile compounds in fishery commodities generally derive from various chemical compound groups including, sulfurous and nitrogenous compounds [4], [22]. These groups of compounds are commonly categorized as other groups. The compound which has the highest proportion in this sample is diethyl phthalate (47.61%). Diethyl phthalate is a colorless liquid that has a bitter, disagreeable taste. This synthetic substance is commonly used to make plastics more flexible. Products in which it is found include toothbrushes, automobile parts, tools, toys, and food packaging. Diethyl phthalate can be released fairly easily from these products, as it is not part of the chain of chemicals (polymers) that makes up the plastic. Diethyl phthalate is also used in cosmetics, insecticides, and aspirin. This compound also is commonly used in making perfumes, plastics, mosquito repellent, and many other products[3]. It is suspected that the broth raw materials used have been contaminated by this compound from the polluted environment.

3.2 Proximate Analysis

Water content, ash, lipid, and protein content of Narrow-barred Spanish mackerel meat wash-water broth (% wet basis) sample

[1] National Center for Biotechnology Information (NCBI). 2020. PubChem Database. 2-Acetylpyrrole, CID=14079, https://pubchem.ncbi.nlm.nih.gov/compound/2-Acetylpyrrole (accessed on July 3, 2020)
[2] National Center for Biotechnology Information (NCBI). 2020. PubChem Database. Methyl palmitate, CID=8181, https://pubchem.ncbi.nlm.nih.gov/compound/Methyl-palmitate (accessed on July 3, 2020)
[3] National Center for Biotechnology Information (NCBI). 2020. PubChem Database. Diethyl phthalate, CID=6781, https://pubchem.ncbi.nlm.nih.gov/compound/Diethyl-phthalate (accessed on July 3, 2020)

were analyzed and the results are shown in Table 2. Proximate analysis results can provide general information on sample nutritional and chemical composition.

Tabel 2. Proximate Analysis Results of Spanish Mackerel Wash-Water Broth

Parameters	%
Moisture	98.13
Ash	1.21
Lipid	0.33
Protein	0.33

The characteristics of water are altered once going into solutions, by interaction with other substances [30]. The water or moisture content in food would determine its nutritive value, texture, taste, and shelf-life stability throughout storage [9]. High water content could be resulting in bacteria, molds, and yeast easily multiplies, thus a series of changes could occur in a food product. The water content analysis result showed that Spanish mackerel wash-water broth has 98,13% water content, whereas according to [31], fresh whole mackerel fish contained 65.58% moisture content. This high water content is expected due to the broth sample state is liquid. Water used in this study was aquadest and it acts as a dispersing medium and solvent for various broth components which contained many volatile compounds.

Ash content analysis showed that the sample has 1.21% inorganic matters. Ash content represents residual inorganic substance from organic materials combustion. The results obtained from ash content determination would describe the total quantity of minerals contained in a commodity. The ash content obtained would depend on the type of material and how to incinerate it. Generally, minerals contained in a sample can be categorized as organic and/or inorganic salts. Taste characteristics of a food product are more affected by these minerals present and their influence on the aroma is rarely studied. The analysis of total ashes determines the amount of residual non-volatile substances in samples after the removal of the organic substances through the incineration process [32]. It was known that whole mackerel fish contains 1.68% ash content [31].

Lipid is an important substance to maintain human health in general. Lipid's insolubility in the water turns out to be a significant unique of its characteristic which separates them from other nutritional constituents in the food matrix such as proteins and carbohydrates. Oils that have a greater proportion of unsaturated fatty acids are liquid at room temperature [33] and fish are commonly rich in these types of fatty acids. Mackerel, which has high oil content, is rich in omega-3 polyunsaturated fatty acid [34]. According to [35], variability in lipid content is also affected by fish tissue. The dark muscle part of the Narrow-barred Spanish mackerel was identified to contain 20% lipid and only 4% of lipid present in its white muscle part. To be exact, whole mackerel fish has 16.52% lipid content [31]. It is commonly recognized that oily fish such as mackerel are prone to lipid oxidation and rancidity reactions due to its high content of polyunsaturated fatty acids. Based on the results from lipid analysis, the Spanish mackerel wash-water broth sample has 0.33% lipid content, due to most of the broth is water, and the meat portion which contains lipid was separated. The lipid content affected the quantity of several volatile components such as long-chained ethyl esters, many ketones, and hexanoic acid. Free unsaturated fatty acids most probably will undergo oxidization reaction and forming lipid hydroperoxides. These peroxides are unstable and promptly deteriorate into secondary oxidation volatile compounds such as hydrocarbons (alkanes and alkenes), ketones, aldehydes, alcohols, and furans [36].

Protein has an important function for the body aside from its principal function as a great energy source. Spanish mackerel meat wash-water broth has 0.33% protein content. [31] study showed that mackerel as a whole fish contained 15.57% protein content. Mackerels are well known abundant in biologically active amino acids and protein such as glutamic acid, lysine, aspartic acid, histidine, arginine [34]. Most of those amino acids, which considered non-volatile components, are affecting the taste characteristics of a commodity. However, there are also volatile compounds derived from peptides and/or amino acids, for instance, volatile compounds which contain nitrogenous and/or sulfurous compounds or element such as pyrazines or amines. Amino acid breakdown process provides distinct volatile compounds compared to those developed from lipid decomposition reaction. Such volatile compounds include sulfides, pyrazines, and other branched-chained amino acids [36].

Conclusion

Based on this study on volatile flavor compounds identification of mackerel fish meat wash-water broth that has been carried out, several conclusions could be obtained. The broth was still had characteristics aroma from many volatile components detected as 121 volatile compounds in total have been discovered from the sample using the SPME extraction method and GC/MS. Most of the volatile flavor compounds detected were came from several groups of compounds such as aldehydes, various aliphatic, cyclic and aromatic hydrocarbons, alcohols, ketones, esters, organic acids, and others compound group. Compounds that have the largest proportion of samples from the other group diethyl phthalate 47,61% which is a common plasticizer and environmental pollutant. The proximate analysis results showed that samples from mackerel fish meat wash-water broth had a water content of 98,13%, ash content of 1,21%, lipid content of 0,33%, and protein content of 0,33%. The results obtained from this study could provide useful basic information regarding compounds that could affect aroma characteristics of the sample and its safety, nevertheless, more studies should be performed regarding these matters.

References

[1] Centre for Data, Statistics, and Information, *2017 One Data of Marine and Fisheries. Centre for Data, Statistics and Information*, Jakarta: Indonesia Marine and Fisheries Ministry Affairs, pp. 224-225, 2018

[2] Y.J. Cha, H.H. Baek, C.Y. Hsieh, "Volatile components in flavor concentrates from crayfish processing waste", *J. Sci. Food Agric.*, Vol. 58, pp. 239-248, 1992.

[3] J.R. Botta, "Freshness quality of seafood: a review", In: Shahidi F and Botta JR, (Eds.), "Seafood: Chemistry, Processing Technology, and Quality", Glasgow: Blackie Academic and Professional, pp. 140-167, 1994.

[4] J.K. Liu, S.M. Zhao, S.B. Xiong, "Influence of recooking on volatile and non-volatile compounds found in silver carp *Hypophthalmichthys molitrix*", *Fish Sci.*, Vol. 75, pp. 1067-1075, 2009.

[5] P. Naknean, M. Meenune, "Review article factors affecting retention and release of flavor compounds in food carbohydrates", *Int. Food Res. J.*, Vol. 17, pp. 23-34, 2010.

[6] M. Guillen, and M. Errecalde, "Volatile components of raw and smoked black bream (*Brama raii*) and rainbow trout (*Onchorhynchus mykiss*) studied by means of solid-phase microextraction and gas chromatography/mass spectrometry", *J Sci Food Agric.*, Vol. 82. pp. 945-952, 2002.

[7] M. Linder, R.G. Ackman, "Volatile compounds recovered by solid-phase microextraction from fresh adductor muscle and total lipids of sea scallop (*Placopecten magellanicus*) from Georges Bank (Nova Scotia)", *J. Food Sci.*, Vol. 67, pp. 2032-2037, 2002.

[8] C. Alasalvar, K.D.A. Taylor, F. Shahidi, "Comparison of Volatile of Cultured and Wild Sea Bream (*Sparusa uratus*) during Storage in Ice by Dynamic Headspace Analysis/ Gas Chromatography-Mass Spectrometry", *J. Agric. Food Chem.*, Vol. 53, pp. 2616-2622, 2005.

[9] R.I. Pratama, I. Rostini, E. Rochima, "Amino Acid Profile, and Volatile Compounds of Raw and Steamed Catfish (*Pangasius hypophthalamus*) and Narrow-barred Spanish Mackerel (*Scomberomorus commerson*)", *IOP Conf. Series. Earth and Environmental Science*, Vol. 116, pp. 1-17, 2018.

[10] C.A. Wright, *The Best Soups in The World.* Hoboken, New Jersey: John Wiley & Sons, pp. 1-2, 2010.

[11] A. A. Damuringrum, "Study on Tilapia Meatball Characteristics with Addition of Flavour Powder from Tiger Shrimp Head Extract). Undergraduates Thesis. Bogor: Marine Science and Fisheries Faculty. Bogor Agricultural Institute. 2002

[12] Association of Official Analytical Chemist (AOAC), *Official Methods of Analysis of AOAC International 18th Edition*. Gaithersburg, USA: AOAC International, 2005.

[13] G. Duflos, F. Moine, V.M. Coin, P. Malle, "Determination of Volatile Compounds in Whiting (*Merlangius merlangus*) Using Headspace–Solid-Phase Microextraction–Gas Chromatography-Mass Spectrometry", *J. Chrom. Sci*. Vol. 43, pp. 304-312, 2005.

[14] C.T. Ho, Q. Chen, "Lipids in food flavors: an overview", In: Ho, C.T., and Hartman, T.G., (Eds.), "Lipids in Food Flavors", Washington DC: American Chemical Society, pp. 2-14, 1994.

[15] H. Sakakibara, M. Hosokawa, I. Yajima, "Flavor constituents of dried bonito (katsuobushi)", *Food Rev. Int.*, Vol. 6, pp. 553-572, 1990.

[16] I. Yajima, M. Nakamura, H. Sakakibara, "Volatile flavor components of dried bonito (katsuobushi) II. from neutral fraction", *Agric. and Bio. Chem.*, Vol. 47, pp. 1755-1760, 1983.

[17] I. Peinado, W. Miles, G. Koutsidis, "Odour characteristics of seafood flavor formulations produced with fish by-products incorporating EPA, DHA, and fish oil", *Food Chem.,* Vol. 212, pp. 612-619, 2016.

[18] O. Lazo, L. Guerrero, N. Alexi, K. Grigorakis, A. Claret, Z. A. Perez, R. Bou," Sensory characterization, physicochemical properties, and somatic yields of five emerging fish species", *Food Res. Int.*, Vol. 100, pp. 396-406, 2017.

[19] R.I. Pratama, I. Rostini, E. Liviawaty," Volatile components of raw Patin Catfish (*Pangasius hypophthalmus*), and Nile Tilapia (*Oreochromis niloticus*). *IOP Conf. Series. Earth and Environmental Science*, Vol. 176, pp. 1-8, 2018.

[20] H.Y. Chung, I. K. S. Yung, W. C. J. Ma, J. Kim, "Analysis of volatile components in frozen and dried scallops (*Patinopecten yessoensis*) by gas chromatography/mass spectrometry", *Food Res. Int.*, Vol. 35, pp. 43-53, 2002.

[21] Y. Jin, Y. Deng, J. Yue, Y. Zhao, W. Yu, Z. Liu, H. Huang, "Significant improvements in the characterization of volatile compound profiles in squid using simultaneous distillation-extraction and GCxGC-TOFMS", *J. Food,* Vol. 13, pp. 434-444, 2015.

[22] U. Tanchotikul, T. C. Y. Hsieh, "Volatile flavor components in crayfish waste", *J Food Sci.*, Vol. 54, pp. 1515-1520, 1989.

[23] H. Sakakibara, T. Yanai, I. Yajima, K. Hayashi, "Changes in volatile flavor compounds of powdered dried bonito (katsuobushi) during storage", *Agric. Bio. Chem.*, Vol. 52, pp. 2731-2739, 1988.

[24] L. Toth, K. Potthast, "Chemical aspects of the smoking of meat and meat products", In: Chichester, C.O. (Ed.), "Advances in Food Research", New York: Academic Press Inc., 1984.

[25] M.D. Guillen, M.C. Errecalde, J. Salmeron, C. Casas, "Headspace volatile components of smoked swordfish (*Xiphias gladius*) and cod (*Gadus morhua*) detected by means of solid-phase microextraction and gas chromatography-mass spectrometry", *Food Chem.,* Vol. 94, pp. 151-156, 2006.

[26] J. Rozum, "Smoke flavor", In: Tarte, R. (Ed.), "Ingredients in Meat Product. Properties, Functionality, and Applications", New York: Springer Science, pp. 211-226, 2009.

[27] H. Sakakibara, J. Ide, T. Yanai, I. Yajima, K. Hayashi, "Volatile flavor compounds of some kinds of dried and smoked fish", *Agric. Bio. Chem.*, Vol. 54 pp. 9-16, 1990.

[28] H.N. Mohamed, Y.C. Man, S. Mustafa, Y.A. Manap, "Tentative Identification of Volatile Flavor Compounds in Commercial Budu, a Malaysian Fish

Sauce, Using GC-MS", *Molecules*, Vol. 17, pp. 5062-5080, 2012.

[29] R.I. Pratama, N. Puspitawati, H. Hamdani, I. Rostini, "Volatile Flavor Compounds Composition of Fresh and Steam Milkfish (*Chanos chanos*)", *Global Scientific Journal*, Vol. 7, No. 1, pp. 1006-1013, 2019.

[30] J. Blahovec, "Role of water content in food and product texture" *Int. Agrophysics.* Vol. 21, pp. 209-215, 2007.

[31] V.V. Ramakrishnan, A. E. Ghaly, M. S. Brooks. S. M. Budge, "Extraction of Proteins from Mackerel Fish Processing Waste Using Alcalase Enzyme", *J. Bioprocess. Biotechniq,* Vol. 3, No. 2, pp. 1-9. 2013.

[32] J.C. de Menezes, G. B. V. Borges, F. C. O. Gomes, M. L. A. Vieira, A.R. Marques, A. M. R. Machado, "Volatile compounds and quality analysis in commercial medicinal plants of *Camellia sinensis*", *Ciência Rural, Vol.* 49, Np. 3, pp. 1-10, 2019.

[33] M.R. Vishnuraj, G. Kandeepan, V., Shukla, A. Arun, T. P. Ramees, J.J. Kattoor, S. B. Sarangam, M. Mohan, D. Bhattacharya, "Comprehensive Lipid Analysis of Foods with special highlight on Foods of Livestock Origin", *Bev. Food W.,* Vol. 43, No. 3, pp. 47-50, 2016.

[34] Y.H. Chang, H. Wenni, H. J. Chen, C. C. Lin, "The composition and health benefits of mackerel concentrate", *MOJ Food Process Technol.*, Vol. 7, No. 3, pp. 88–90, 2019.

[35] S. Nazemroaya, M.A. Sahari, M. Rezaei, Identification of Fatty Acid in Mackerel (*Scomberomorus commersoni*) and Shark (*Carcharhinus dussumieri*) Fillets and Their Changes during Six Month of Frozen Storage at -18˚C S. *J. Agr. Sci. Tech.,* Vol. 13, pp. 553-566, 2011.

[36] B. Nordvi, Ø. Langsrud, B. Egelandsdal, E. Slinde, G. Vogt, M. Gutierrez, E. Olsen, "Characterization of Volatile Compounds in a Fermented and Dried Fish Product during Cold Storage", *J. Food Sci.*, Vol. 72, No. 6, pp. 373-380, 2007.