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VOLATILE COMPOUND COMPOSITION OF SPRAY DRY FLAVOR POWDER FROM NARROW-BARRD SPANISH MACKEREL BROTH

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ABSTRACT

Volatile components generally affect the aroma characteristics acceptance of a product including fisheries commodities such as narrow-barred Spanish mackerel. This research aims to identify the volatile component of flavor powder which prepared from narrow-barred Spanish mackerel broth. The Spanish mackerel were collected from the local fish landing site in Karangsong, Indramayu, West Java, Indonesia. The method used in this research was to identify the sample's volatile compounds using Gas Chromatography /Mass Spectrometry (GC/MS) and using Solid Phase Microextraction (SPME) (800C; 30 minutes) as its extraction method. To support the main volatile data analysis, the samples were also analyzed for their proximate composition. The volatile compound analysis has succesfully detected as much as 87 volatile compounds from the broth sample. Volatile compounds detected were mostly derived from aldehydes, alcohols, hydrocarbons, ketones, organic acid, ester, and other groups of compounds. The highest proportion of volatile compounds detected in the sample was pyrazine, trimethyl- (14,04%). The proximate analysis results showed that Spanish mackerel flavor powder had a moisture content of 4,72%, ash content of 2,31%, lipid content of 0,24% and protein content of 8,07%.

Keywords: aroma, flavor powder, narrow-barred Spanish mackerel, proximate, volatile component

1. INTRODUCTION

Indonesia has great potential in capture fisheries sector specifically marine water fish such as Spanish mackerel (*Scomberomorus commerson*). This commodity is one of the leading commodities in Indonesia captured fisheries production, mainly due to its wide range of application as a raw material. Based on official data from [1], the volume of marine capture fisheries production for Spanish mackerel species in 2016 was equal to 225,936 tons and in 2017 was increased by 438,658 tons. The principal reason behind this high increased of Spanish mackerel production volume in Indonesia was due to its important economic value and high protein content.

Figure 1. Scomberomorus commerson [14]

Spanish mackerel is classified as large pelagic marine water fish which has 19.23% protein content. This high protein content is specifically content in the meat portion of Spanish mackerel. Furthermore, it also contain essential fatty acid omega-3 and vitamins which are valuable for human growth and endurance. Public consumption volume of mackerel fish meat has increased due to its wide application in processed fish based products such as crackers, meatballs, dumplings, *pempek* and seafood dishes. Spanish mackerel can be processed or cooked in various ways depending on the processor taste. Each processed product would have a special characteristic due to the differences in chemical and flavor composition as a result of the processing stage [2].

Flavor is an important component in the acceptance of food. Flavor component is generally divided into two groups, first one is volatile flavor component which contributes to aroma characteristics of a product and second is non-volatile flavor components which contributes to taste characteristics. Based on their physical form, flavors can be classified into three categories, solid, paste and liquid for. Drying process could change liquid flavor form into solid powder form through drying process.

Drying is a process of reducing the water content to achieve certain water intensity, thus this process could accelerate the chemical and biological damage rate .There are various types of food drying equipment, several of which are drum dryers, conveyor dryers, spray dryers, etc. The advantages of spray drying compared to other drying method are the relatively simple in operating the equipment, its large capacity and short processing time. The final result of the materials which went through a spray drying process is to develop into a fine powder form. A broad range of flavor powder is available in the market starting from seasoning powder made from extracted meat to mixed herbs. Flavoring powder has a distinctive taste and aroma characteristics and differ based on its raw material used. The product aroma characteristic is affected by volatile compounds contained in the products and the processing stages.

In several countries such as China, Japan and Canada, the research regarding volatile component identification from fisheries commodity has been carried out from more than a decade. [3], studied concerning the effect of reheating on volatile and non-volatile compounds found in silver carp (*Hypophthalmichthys molitrix*) and [4], studied concerning volatile compounds in smoked salmon products. Research on the volatile flavor composition of fishery products in Indonesia is still rarely performed. According to [5], numerous research topics concerning volatile compounds identification which were found in Indonesia originated from agricultural commodities. However for fishery commodities, similar research is still scarce. Information about volatile flavors composition is important to identify product's aroma characteristics and as a reference for similar research. The composition of the volatile compounds detected in fishery products is mostly derived from aldehydes, alcohols, ketones, and hydrocarbons. The purpose of this study was to identify the volatile compounds composition contained in the Spanish mackerel flavor powder.

2. RESEARCH METHOD

2.1 Research Location

Narrow-barred Spanish mackerel samples were taken from local fish landing site in Karangsong, Indramayu West Java. Sample preparation was carried out in the Fisheries Product Processing Laboratory, Fisheries, and Marine Sciences Faculty, Universitas Padjadjaran. The spray drying method was carried out at Central Laboratory, Universitas Padjadjaran. The volatile compound analysis was carried out in Flavor Laboratory, Indonesian Centre for Rice Research, Sukamandi, Subang, West Java and proximate analysis were carried out in Inter-University Centre Laboratory, Bogor Agriculture Institute.

2.2 Research Methods

The method used was experimental method to identify volatile compound composition and proximate flavor powder from Narrow-barred Spanish mackerel boiled water broth. And then the final research will be discussed in a comparative descriptive based on the identification and the semi quantification intensity of the compounds detected from the analyzed samples.

2.2.1 Sample Preparation

1) As much as 3 kg Spanish mackerel samples were taken from the landing site in a cool box which contained layers of bulk ice.
2) Samples were then separated between the heads, shells, and meats.
3) The Spanish mackrel meat portion were subsequently washed and boiled (65^{o}C for 90 minutes in aquadest water with 1:2 ratio.
4) Spanish mackerel broth was then filtered and 15% maltodextrin was added to the broth as a filler. The sample was then pour into glass bottles covered with aluminum foil and cling wrap.
5) Spanish mackerel broth samples were subsequently dried with spray dryer (inlet temperature 170°C and outlet temperature 80°C, during 1.5 hours) at Central Laboratory Universitas Padjadjaran until a develop into powder form.
6) After the drying process finished, flavor powder sample was weighed, labeled and packed using three different packaging layers (packaging was aluminum foil, clinging wrap plastics and zip-lock plastic bag). The purpose of this layered packaging is to minimize the changes and degradation which could happen such as air, light, and temperatures to the samples during transportation to the analysis laboratory.
7) The tight packed samples were then transported to Flavor Laboratory for volatile component analysis and Inter-University Centre Laboratory for proximate analysis.

2.2.2 Proximates analysis

Sample was then analyzed for its moisture, ash, protein and lipid content. The proximate analysis according to [6].

2.2.3 Volatile compound analysis

Volatile components was analyzed according modification of [7] procedure. The analysis was carried out using water bath for samples extraction and Gas Chromatography (GC) (Agilent Technologies 7890A GC System) and Mass Spectrometry (MS) apparatus (Agilent Technologies 5975C Inert XL EI CI/MSD) for detecting and identifying the volatile components. The sample extraction method was done by Headspace Solid Phase Micro Extraction (HS/SPME) using DVB/Carboxen/Poly Dimethyl Siloxane fiber. Sample extraction time used on water bath was 45^{o}C for 45 minutes. GC column used was DB-5 (60 m x 0,25 mm x 0.25 mm), helium carrier gas, the initial temperature was 45^{o}C (hold 5 minutes), temperature escalation as much as 5^{o}C/minutes, final device temperatures 250^{o}C (hold 5 minutes) with an overall running time 36 minutes.

2.2.4 Data Analysis

Samples volatile components mass spectrums detected from GC/MS were then compared with the mass spectrum pattern which was available in the computer database or NIST (National Institute of Standard and Technology) library 0.8L version. The data then were further analyzed with Automatic Mass Spectral De-convolution and Identification System (AMDIS) software.

3. Result and Discussion

3.1 Volatile compounds

Volatile compounds analysis result of Spanish mackerel flavor powder showed that as much as 87 volatile compounds were succesfully detected from the sample. Volatile compounds identified were derived from aldehyde groups (10 compounds), alcohol (16 compounds), hydrocarbons (47 compounds), ketones (2 compounds), organic acids (2 compounds), esters (5 compounds) and others (5 compounds). The result of volatile flavor analysis from Spanish mackerel flavor powder is shown in Table 1 with the compound proportion based (area percentage) on highest to lowest abundance.

Table 1. The result of volatile component from Spanish mackerel flavor powder

Group	RT	Compound	Area	Proportion (%)
Aldehydes	2.2578	Pentanal	351389193	9.99
	12.2708	Nonanal	224097938	6.37
	1.7524	Butanal, 3-methyl-	143829291	4.09
	11.2362	2-Octenal, (E)-	28960031	0.82
	15.3449	Decanal	27677880	0.79
	10.5227	Benzeneacetaldehyde	13022286	0.37
	8.18	Benzaldehyde	12579122	0.36
	9.6368	2,4-Heptadienal, (E,E)-	7635272	0.22
	6.5924	Heptanal	5745527	0.16
	7.9838	2-Heptenal, (Z)-	1991864	0.06
Alcohol	10.1006	1-Hexanol, 2-ethyl-	174345768	4.96
	4.2616	2,3-Butanediol	81049772	2.30
	6.7767	Ethanol, 2-butoxy-	20427672	0.58
	23.3482	1-Hexadecanol, 2-methyl-	16277083	0.46
	16.76	1-Dodecanol, 2-hexyl-	16039681	0.46
	18.6925	1-Dodecanol, 2-methyl-, (S)-	12996666	0.37
	22.1233	1-Octanol, 2-butyl-	9386240	0.27
	21.3206	1-Dodecanol, 2-octyl-	9249571	0.26
	26.3033	Ethanol, 2-(dodecyloxy)-	7304862	0.21
	17.3487	1-Decanol, 2-octyl-	7049430	0.20
	25.8514	2-Ethyl-1-dodecanol	5239603	0.15
	25.4709	Ethanol, 2-(tetradecyloxy)-	3334463	0.09
	6.1167	1-Hexanol	2907311	0.08
	4.1011	2,3-Butanediol, [S-(R*,R*)]-	2836657	0.08
	31.3634	2-Ethyl-1-dodecanol	1452425	0.04

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Group	RT	Compound	Area	Proportion (%)
	7.6984	2-Heptanol, 6-methyl-	1361171	0.04
Hydrocarbons	12.6335	Nonane, 3,7-dimethyl-	173382583	4.93
	12.9189	Decane, 3,6-dimethyl-	135018910	3.84
	11.7773	Decane, 3,7-dimethyl-	123575338	3.51
	12.5146	Undecane, 5-methyl-	110470388	3.14
	11.4206	Tridecane, 6-methyl-	98912485	2.81
	11.5871	Octane, 2,3,3-trimethyl-	72735914	2.07
	11.9854	Undecane, 4-methyl-	67328196	1.91
	12.0924	Tridecane, 4-methyl-	61254007	1.74
	13.0973	Hexane, 3,3-dimethyl-	56000651	1.59
	13.9714	Undecane, 2-methyl-	47010758	1.34
	14.1617	Undecane, 3-methyl-	46448725	1.32
	15.1665	Dodecane	42625121	1.21
	17.5152	1-Tridecene	38155888	1.08
	13.1687	Hexane, 2,3,4-trimethyl-	35664328	1.01
	13.4303	Undecane, 5,7-dimethyl-	33963691	0.97
	12.7941	Hexadecane, 2,6,11,15-tetramethyl-	31732917	0.90
	14.5779	1H-Indene, 1-methylene-	30372159	0.86
	14.7979	9-Eicosene, (E)-	26662961	0.76
	19.2038	2,6-Octadiene, 2,6-dimethyl-	22813979	0.65
	22.6584	1-Pentadecene	21333378	0.61
	11.0935	Dodecane, 2,7,10-trimethyl-	19900333	0.57
	15.6244	6-Tridecene, (Z)-	16757246	0.48
	20.3574	Tetradecane	15433863	0.44
	10.7724	Undecane, 3,6-dimethyl-	14419600	0.41
	27.0347	1,3-di-iso-propylnaphthalene	13811817	0.39
	18.2882	5-Tetradecene, (E)-	13492130	0.38
	27.7601	Naphthalene, 1,2,3-trimethyl-4-propenyl-, (E)-	13415471	0.38
	17.7352	Tridecane	13260959	0.38
	17.2714	7-Tetradecene	11862387	0.34
	27.4449	Heptadecane	11828474	0.34
	27.5698	Dodecane, 2,6,11-trimethyl-	11710152	0.33
	17.1525	3-Tetradecene, (E)-	11638691	0.33
	27.9741	2,6-Diisopropylnaphthalene	11342283	0.32
	23.7406	8-Heptadecene	11220582	0.32
	19.5963	1-Undecene	10455372	0.30
	26.226	3-Octadecene, (E)-	10193478	0.29

Group	RT	Compound	Area	Proportion (%)
	20.1492	2-Tetradecene, (E)-	9747360	0.28
	18.1276	6-Tetradecene, (E)-	8342628	0.24
	28.1168	1,7-di-iso-propylnaphthalene	7374736	0.21
	18.0503	Decane, 3,8-dimethyl-	7280753	0.21
	15.4757	Nonane, 4,5-dimethyl-	6897639	0.20
	26.3866	Pentadecane	5700191	0.16
	29.5736	Heptadecane	5512196	0.16
	28.0514	1,4-di-iso-propylnaphthalene	4981333	0.14
	29.6628	Octadecane	4342358	0.12
	30.7509	3,3-Diethylpentadecane	1785876	0.05
	31.6071	Nonadecane	1754626	0.05
Ketones	8.9946	Cyclobutanone, 2,3,3-trimethyl-	9589140	0.27
	8.4416	Acetophenone	6305433	0.18
Organic acids	3.774	Acetic acid	15819278	0.45
	20.8271	cis-Vaccenic acid	10557658	0.30
Esters	3.0308	Acetic acid, methoxy-	95730620	2.72
	25.2093	Sulfurous acid, 2-pentyl undecyl ester	49196860	1.40
	18.9838	Sulfurous acid, hexyl tridecyl ester	15725620	0.45
	26.0298	Carbonic acid, decyl pentadecyl ester	13174765	0.37
	20.7438	Pentafluoropropionic acid, dodecyl ester	4665697	0.13
Others	9.2324	Pyrazine, trimethyl-	493922328	14.04
	19.7687	2-Ethyl-3-hydroxyhexyl 2-methylpropanoate	32031342	0.91
	5.5935	Hydrazine, 1,1-dimethyl-	19113705	0.54
	9.9222	Acetylpyrazine	8316368	0.24
	6.4497	3-Furanmethanol	5236108	0.15
	Total		3517496612	100

RT : *Retention Time* (minutes)

3.1.1 Aldehydes

Aldehydes compound group were also detected in the flavor powder sample and pentanal (9.99%), nonanal (6.37%) and butanal, 3-methyl- (4.09%) were the top three most abundant volatile compounds. Pentanal compounds are commonly derived from lipid oxidation which typically occurs in fish meat [8]. Most aldehydes detected in fish meat derive from the double carbon bonds oxifation of unsaturated fatty acids discovered in fish meat or saturated fatty acids [2]. Pentanal compounds are recognized to have green flavor characteristics [9].

3.1.2 Alcohols

Volatile compound analysis was succesfully detected alcohols group of compound which generally be formed through the secondary decomposition of fatty acid hydroperoxides [10]. Several compounds from this group with highest proportion values were 1-hexanol, 2-ethyl- (4.96%), 2,3 butanediol (2.30%) and ethanol, 2 butoxy (0.58%). The 1-hexanol, 2-ethyl- was also identified in [5], study specifically in volatile composition of fresh patin catfish and fresh and steamed mackerel samples. The alcohol component generally produces sweet, fruity, alcoholic, balsamic, and green aroma descriptor depending on the molecular structure [11].

3.1.3 Hydrocarbons

Hydrocarbon groups (mostly aliphatic, cyclic and aromatic) were identified in this studied sample. Hydrocarbon compounds could be derived from the decarboxylation reaction and the carbon chain reaction of fatty acids and thermal oxidation of unsaturated fatty acids [12]. Type of compound with has the highest proportion value in this compound group is nonane, 3,7-dimethyl- (4,93%). This compound was also discovered in [13], study regarding identify and quantify the principal volatile compounds from freshly harvested adductor muscle and from total lipids of the sea scallop specifically in lipids found in the seashells of the *Placopecten magellanicus* species.

3.1.4 Ketones

Ketones could be produced from microbial oxidation, amino degradation or lipid oxidation. The ketone group of compounds could also derived from thermal oxidation or degradation process of unsaturated fatty acids, amino acid degradation or oxidation of microorganisms [3]. Cyclobutanone, 2,3,3-trimethyl- (0,27%) and acetophenone (0,18%) were the identified volatile compounds which have high proportion compared to other ketones in this flavor powder sample. Cyclobutanone, 2,3,3-trimethyl- was previously found in Spanish mackerel broth [14]. Acetophenone compound is an organic compound which classified as aromatic ketone. Acetophenone compound was previously identified in traditional fermented smoked fish (*Katsuobushi*). The aroma produced by the spray dried flavor powder samples tends to have a fishy and sweet aroma. The flavor powder aroma can be retained due to the addition of maltodextrin which also act as a filler. This is consistent with the statement from [15], which stated that maltodextrin could protect the stability of the aroma during the drying process using a spray dryer. According [5], study, ketone compounds group which were detected in samples were known to contribute to the sweet aroma of *crustaceans*.

3.1.5 Organic Acid

The organic acids compound group which generally found in fishery products could act as an antibacterial and antioxidant compound. This report is similar with [16], which stated that organic acids produced from the pyrolysis process could serves as an inhibitor of bacterial growth. Organic acid volatile compounds with the highest proportion value is acetic acid (0.45%). Acetic acid is a compound which commonly derived from lipid oxidation [4]. Acetic acid are generally responsible for the increased of unpleasant odors during the storage process [17]. Acetic acid was previously identified in traditional smoked fish in Indonesia, *ikan pe,* made from marine water stingray. Acetic acid compounds are also found in fish sauce which has vinegar-like aroma [18].

3.1.6 Esters

Esters may be developed from lipid metabolism through acid esterification with alcohol [19]. Ester compounds which has the highest proportion were acetic acid, methoxy- (2.72%), sulfurous acid, 2-pentyl undecyl ester (1.40%) and sulfurous acid, hexyl tridecyl ester (0.45%). The class of ester compounds found in fish are generally originates from acid and alcohol esterification which was previously formed from lipid metabolism [5]. An acetic acid, methoxy, is commonly derived from one of the methyl hydrogens which is replaced by a methoxy group.

3.1.7 Other compounds

Pyrazine trimethyl (14.04%) is a compound with the highest proportion value in Spanish mackerel flavor powder compared to other types of compounds. Pyrazine compounds are commonly formed during boiling and drying process due to the applying of heat [20]. Pyrazine compound, trimethyl- was also discovered in [21], study of pyrazine and volatile compound in cocoa beans that generally pyrazine, trimethyl- gives a distinctive popcorn aroma and is often found in the fermentation process, extraction results of palm sugar, cocoa bean products, and fish sauce. Compound which categorized in other group of compound is generally nitrogenous, furans or sulfurous types of compound.

3.2 Proximate analysis

The proximate analysis provides general information regarding sample's chemical composition, the nutritional content. The difference in the results showd could be influenced by the raw material chemical composition, commodity type the processing stages. Proximate analysis comprise of water content, ash content, lipid content, and protein content. The Spanish mackerel flavor powder proximate analysis results are shown in Figure 2.

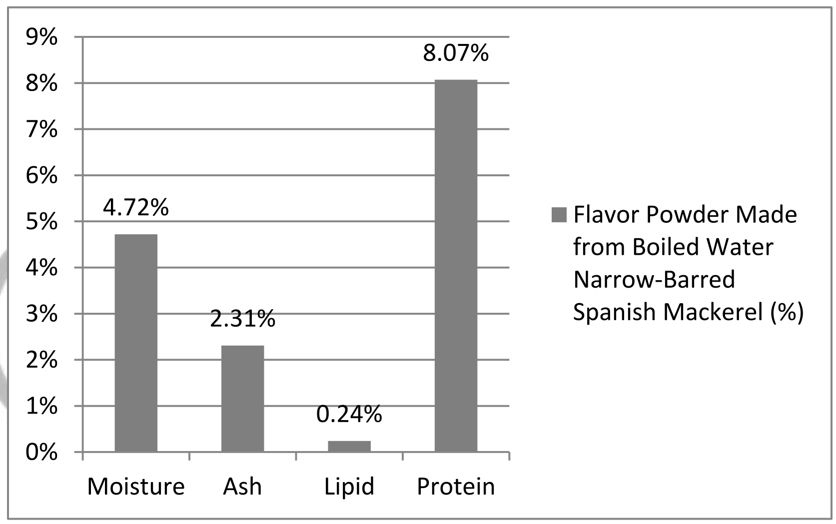

Figure 2. Proximate analysis results of narrow-barred Spanish mackerel flavor powder (%)

3.2.1 Moisture Content

Moisture content determination is the most commonly perform analysis in fisheries commodity. The amount of water or moisture in food often determines the nutritional value, taste characteristics, and the shelf life stability throughout storage. Analysis result shows that the Spanish mackerel flavor powder has 4.72%. The analysis result indicates that sample's moisture content exceeds the Indonesian National Standards [22] moisture content limit for flavor powder (chicken based), which established at a maximum value of 4.0%. The moisture content value of samples exceeds the maximum limit specified, this can be determined related to the addition of fillers containing maltodextrin. Maltodextrin has hygroscopic properties which are the ability to absorb air thereby increasing water content with Maltodextrin added [23].The moisture content of a dry product such as flavor powder could be influenced by the environment moisture and humidity. The product dry surface and hygroscopic characteristic of a dry product are able to absorb moisture from the surrounding environment possibly during the preparation or storage of samples.

3.2.2 Ash Content

The ash content analysis result in Figure 2 shows that the sample has an ash content of 2.31%. This measured content value could be affected by initial raw material mineral content which also depends on raw material type and combustion method. Ash content of food shows the total minerals contained in it. Marine water fish meat is known to have high minerals content [24]. Ash content measured in fisheries commodity could be influenced by species, growth phase and various environmental factors.

3.2.3 Lipid Content

The analysis results of lipid content in Figure 2 shows that the Spanish mackerel flavor powder has a lipid content of 0.24%. Based on [22], a flavor powder (chicken based) should have a minimum of 2% lipids. The measured lipid content value could affect by raw material types, and also filleting process thus the fat rich portion from the belly part of the fish were separated. Lipid content and water content have a negative correlation, hence if the lipid content in a product is at a low level then the water content value should be high. Furthermore, a decrease in the lipid content value could be caused by an oxidation reaction. According to the study of proximate compotition Spanish mackerel [25], the chemical composition of freshwater and marine water fish shows differences. Most marine water fish have lower lipid content compared to freshwater fish. Low levels of lipids could be affected by environmental factors and water and fat loss during the heating involve process [5]. Lipid content in fish meat directly affects the aroma and taste intensity [5].

3.3.3 Protein Content

Proteins are macromolecules which constitutes from a series of amino acids. Volatile ketones group could be produced from the degradation of amino acids apart from lipid oxidation. The protein content analysis result in Figure 2 shows that Spanish mackerel flavor powder has 8.07% protein. Based on [22], flavor powder should have a minimum 6% protein. The amount of protein content could be affected by the heating involved process. The protein content measured would depends on the number of ingredients added and is largely influenced by the water content of the material. Heating processes such as boiling and drying can have an effect on the structure and functional properties of proteins in the material. This change is partly due to the denaturation of the protein caused by temperature changes during the heating process [26]. Various types of volatile compounds detected and identified from the sample are mostly derive from protein and lipid components, thus the types of volatile compounds are related to the sample's chemical compounds variability contained [5].

4. CONCLUSIONS

Groups of compounds that were detected in the Spanish mackerel flavor powder generally derived from aldehydes, alcohols, hydrocarbons, organic acids, ketone, ester, and others (nitrogenous compounds and furans). The volatile components identification result of flavor powder had successfully detected 87 types of compounds with pyrazine, trimethyl- (14.04%) is the highest proportion compound in this sample. Proximate analysis results showed that the Spanish mackerel flavor powder has moisture content of 4,72%, 2.31% ash content, 0.24% lipid content and 8.07% protein content.

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