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VOLATILE COMPOUND COMPOSITION OF CONVENTIONAL METHODS FLAVOUR POWDER FROM NARROW-BARRED SPANISH MACKEREL'S BONES

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ABSTRACT

The volatile flavour is a sensation that is obtained when smelling food that is felt by the sense of odour. Volatile component are groups of compounds affecting overall product's flavour and consumers acceptance due to their effect on the aroma characteristic. The objective of this research was to identify the volatile components composition of flavour powder from mackerel's bones by drying the conventional method. Solid-Phase Micro-extraction carried out to extract the component on 80^0C for 30 minutes. The volatile compunds were detected and indentified using Gas Chromathography/Mass Spectrometry. The research results were analyzed in a comparative descriptive. The volatile compounds analysis has successfully detected as much as 145 volatile compunds from flavour powder mackerel's bones. Most of detected component derives from aldehydes, ketones, alcohol, hydrocarbons, organic acid, esters, and other grup of compounds. The highest proportion of volatile compunds detected in the sample was maltol (11,43%).

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

1. INTRODUCTION

The official data of the Ministry of Maritime Affairs and Fisheries of the Republic of Indonesia stated that the volume of marine capture fisheries production for narrow-barred Spanish mackerel in 2016 amounted to 225,936 tons and in 2017 increased by 438,658 tons [1]. Narrow-barred Spanish mackerel (*Scomberomorus commerson*) is a pelagic sea fish that is popular by the community because the meat is not fishy and the taste of the meat is delicious [2]. Fish meat contains compounds that are very useful for humans, namely protein, fat, carbohydrates, vitamins and mineral salts. Fish is a very potential source of animal protein because it contains the largest protein component after water in its body [3]. Processing of mackerel has been done by many people such as pempek, otak-otak, meatballs, dumplings, crackers.

Figure 1. Scomberomorus commerson [21]

This processing industry will certainly produce fish waste consisting of bones, thorns, heads and tails as well as liquid waste during the processing process. Fishbones are wastes produced from the fish processing industry [4]. Fishbones have three main elements, namely calcium, which is the content in bone among other body parts of fish, phosphorus and carbonate [5].

Extracted by boiling in fish will produce liquid broth concentrate. The resulting liquid broth can be processed as a flavouring ingredient (flavouring) by means of drying because the savoury taste of the flavouring ingredient from this liquid broth can increase preference for food products. Processing step can affect the flavour characteristics of product [6]. Flavour can produce sensations from food ingredients when placed in the mouth caused by taste and aroma [7]. Sensations that appear on flavour are caused by volatile chemical components (aroma sensation) and nonvolatile (taste sensation) [8]. The volatile flavour is a sensation that is obtained when you smell a portion of food that is felt by the odour sense. Groups of aldehydes, alcohols, ketones, acids and hydrocarbons are usually the composition of volatile compounds detected in fishery products [9], while the nonvolatile flavour is the component responsible for taste. One way to form flavour powder from foodstuffs is by drying, this drying method can be done on a liquid form flavour to produce flavour powder.

Drying is the process of reducing a partial portion of the water from a material using heat energy. The water content is reduced to the extent that microbes cannot grow anymore [10]. Liquid form flavours can be dried with conventional and modern methods. The advantages of conventional drying methods are that the tools are easy to obtain and the application is quite easy while the disadvantage of conventional drying methods is the length of the drying process [11]. Teflon is one of the frying pan type which can be used to dry the liquid broth and alter it to become powder form. The resulting powder in the form of flavour powder act as a flavouring agent that can be added as flavour to food.

Flavour powder is included in food additives that can give and add product aroma. The manufacture of this product is expected to be able to compete with other flavours that are preferred by the community [12]. The application of flavour powder is easier and more practical because it can be used as a flavouring agent in various processed products. Information about the composition of volatile powder flavour compounds in fishery products has not been much studied.

2. RESEARCH METHOD

2.1 Research Location

Narrow-barred Spanish mackerel samples were taken from the 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 and the volatile compound analysis were carried out in Flavor Laboratory, Indonesian Centre for Rice Research, Sukamandi, Subang, West Java.

2.2 Research Methods

The method used was an experimental method to identify volatile compound composition from Narrow-barred Spanish mackerel's bones powder flavour. 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

The samples were taken from the local fish landing site in Karangsong, Indramayu, West Java. Sample preparation was carried out in the Fisheries Product Processing Laboratory, Fisherires and Marine Sciences Faculty, Padjadjaran University and volatile compunds analysis were carried out in Flavor Laboratory, Indonesian Centre for Rice Research, Sukamandi, Subang.

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 bones and meats.
3) The Spanish mackerel bones are separated with meat and boiled (65^{o}C for 90 minutes in aquadest water with 1:2 ratio).
4) Spanish mackerel broth was then filtered and 10% maltodextrin was added to the broth as a filler. The sample was then poured into glass bottles covered with aluminum foil and cling wrap.
5) Sample of Spanish mackerel broth were subsequently dried with conventional methods using teflon frying pan.
6) After the drying process finished, flavour powder sample was pulverized until smooth, 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 tightly packed samples were then transported to Flavor Laboratory for volatile component analysis.

2.2.2 Volatile compound analysis

Volatile components were analyzed according to the modification of [21] Damuringrum research procedure. The analysis was carried out using a 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 fibre. 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.

2.2.3 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 Deconvolution and Identification System (AMDIS) software.

3. Result and Discussion

3.1 Volatile compounds

Volatile compounds analysis result of Spanish mackerel flavour powder showed that as much as 145 volatile compounds were successfully detected from the sample. Volatile compounds identified were derived from aldehyde groups (20 compounds), ketones (2 compounds), alcohol (16 compounds), hydrocarbons (47 compounds), organic acids (2 compounds), esters (5 compounds) and others (5 compounds). The result of volatile flavour analysis from Spanish mackerel flavour 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

Sample	RT (min)	Compound	Area	Proporsi (%)
Aldehyde	12.604	Nonanal	243403081	9.91
	2.6445	Pentanal	187074671	7.62
	10.8737	Benzeneacetaldehyde	98797805	4.02
	2.1629	Butanal, 3-methyl-	66040938	2.69
	9.6073	Octanal	63661776	2.59
	15.5176	Decanal	47002428	1.91
	4.5235	Hexanal	41882128	1.71
	11.2305	2-Octenal, (E)-	40986330	1.67
	6.9613	Methional	36730667	1.50
	5.2429	Furfural	21831998	0.89
	9.8808	2,4-Heptadienal, (E,E)-	21449714	0.87
	6.8067	Heptanal	21163348	0.86
	8.4597	Benzaldehyde	20323802	0.83
	17.0992	2-Decenal, (E)-	12309198	0.50
	5.6472	2-Hexenal, (E)-	10133487	0.41
	8.317	2-Heptenal, (Z)-	10120881	0.41
	18.6332	2,4-Decadienal, (E,E)-	6917373	0.28
	15.797	2,4-Nonadienal, (E,E)-	4501130	0.18
	29.0446	Octanal, 2-(phenylmethylene)-	2241784	0.09
	29.1576	Heptanal	2084939	0.08
Ketones	11.6527	Ethanone, 1-(1H-pyrrol-2-yl)-	115619413	4.71
	12.3127	3,5-Octadien-2-one, (E,E)-	63836427	2.60
	7.098	Ethanone, 1-(2-furanyl)-	46512758	1.89
	17.9673	2-Undecanone	24826913	1.01
	9.084	2,3-Octanedione	18849750	0.77
	19.2694	3-Nonen-2-one	15667697	0.64
	22.1176	trans-Geranylacetone	12588101	0.51

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Sample	RT (min)	Compound	Area	Proporsi (%)
	20.8392	Bicyclo[3.2.1]oct-3-en-2-one, 4-methyl-	10666828	0.43
	15.1905	2-Decanone	8052058	0.33
	16.6948	2,3-Octanedione	5372205	0.22
	27.243	β-iso-Methyl ionone	5259731	0.21
	22.5041	Cyclohexanone, 2-ethyl-	3739354	0.15
	19.3705	1-(2-Pyrazinyl)butanone	3579542	0.15
	27.7187	1-(4-Benzylphenyl)ethanone	3079890	0.13
	8.0137	Ethanone, 1-(1-cyclohexen-1-yl)-	2770669	0.11
	20.643	1-Methyl-2-decalone	2322464	0.09
	26.476	Benzophenone	2175206	0.09
	19.097	Pyrethrone	2030559	0.08
	22.837	α-Isomethyl ionone	1677981	0.07
Alcohol	13.0559	Maltol	280608566	11.43
	5.8375	2-Furanmethanol	50499195	2.06
	10.4694	1-Hexanol, 2-ethyl-	48701643	1.98
	13.7932	(S)-(+)-6-Methyl-1-octanol	16685095	0.68
	19.7986	3-Allyl-6-methoxyphenol	15604842	0.64
	14.4889	1-Octanol, 2-butyl-	10983020	0.45
	10.6894	3,5-Octadien-2-ol	9502291	0.39
	14.7089	Levomenthol	9216312	0.38
	9.0245	1-Octen-3-ol	8796480	0.36
	14.4175	1-Pentanol, 2,4,4-trimethyl-	7449463	0.30
	24.0262	Acetyleugenol	6198738	0.25
	23.7468	2,4-Di-tert-butylphenol	5928643	0.24
	6.5629	2-Methylenecyclohexanol	5417098	0.22
	16.2132	4,8-Decadien-3-ol, 5,9-dimethyl-	4250153	0.17
	8.091	Z-4-Dodecenol	4030446	0.16
	20.3338	Phenol, 2,4,6-trimethyl-	3894664	0.16
	17.5273	1-Decanol, 2-ethyl-	3608551	0.15
	15.9694	1-Decanol, 2-hexyl-	3011877	0.12
	24.383	1-Dodecanol, 2-hexyl-	2400315	0.10
	24.4841	1-Octanol, 2-butyl-	2375468	0.10
	25.8041	1-Decanol, 2-octyl-	2358690	0.10
	23.5446	1-Dodecanol, 2-octyl-	2220424	0.09
	28.6344	Phenol, 4-(1-methyl-1-phenylethyl)-	1945640	0.08
	26.3035	Epicedrol	1944568	0.08
	18.9721	Ethanol, 1-(1-cyclohexenyl)-	1830976	0.07
	23.4733	1-Decanol, 2-hexyl-	1753634	0.07
	21.8857	Cycloheptanol, 2-methylene	841514	0.03
Hydrocarbons	23.2354	Pentadecane	74787276	3.05
	13.4067	Cyclopropane, 1,1-diethyl-	36538050	1.49
	13.5019	3-Dodecene, (E)-	30346444	1.24
	27.8614	Heptadecane	23673840	0.96

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Sample	RT (min)	Compound	Area	Proporsi (%)
	15.2857	Dodecane	19696595	0.80
	27.9981	Heptadecane, 2,6-dimethyl-	17032728	0.69
	22.6705	Cyclopropane, nonyl-	16212007	0.66
	21.3922	Caryophyllene	12175920	0.50
	18.0684	Tridecane	10833673	0.44
	14.59	1-Nonene	10097081	0.41
	20.7203	Tetradecane	6748056	0.27
	17.337	1-Methyl-2-methylenecyclohexane	6410407	0.26
	16.897	2,6-Octadiene-1,8-diol, 2,6-dimethyl-	6319416	0.26
	20.4943	Cyclotetradecane	4970747	0.20
	8.8997	1-Octene, 3-methyl-	4915104	0.20
	19.6262	Tridecane, 4-methyl-	4189496	0.17
	17.2597	Dodecane, 3-methyl-	3990288	0.16
	22.2722	Undecane	3522477	0.14
	15.7138	1-Methylnorbornene	3409820	0.14
	8.8402	Heptane, 3-methylene-	3190236	0.13
	24.7398	Dodecane	3084148	0.13
	24.1987	Hentriacontane	2993891	0.12
	16.344	Cyclopentane, 1-pentyl-2-propyl-	2979761	0.12
	26.7079	Hexadecane, 2,6,11,15-tetramethyl-	2719823	0.11
	26.8803	Cyclotetradecane	2646354	0.11
	28.2479	Naphthalene, 1,2,3-trimethyl-4-propenyl-, (E)-	2597089	0.11
	16.4392	Cyclotridecane	2538006	0.10
	27.6057	1,7-di-iso-propylnaphthalene	2234756	0.09
	7.9424	1,3-Pentadiene, 2,4-dimethyl-	2182716	0.09
	30.0079	Nonadecane	1908400	0.08
	22.4268	Allo-Aromadendrene	1780796	0.07
	28.4798	2,6-Diisopropylnaphthalene	1651680	0.07
	27.5046	1,3-di-iso-propylnaphthalene	1639778	0.07
	23.6754	Butylated Hydroxytoluene	1079220	0.04
	28.5571	1,4-di-iso-propylnaphthalene	962143	0.04
	28.8187	Octadecane	800461	0.03
	32.0533	Decane	781186	0.03
	26.3927	Benzene, (1-pentylheptyl)-	743665	0.03
Organic acids	17.6521	Nonanoic acid	20119899	0.82
	24.9122	Nonahexacontanoic acid	2153061	0.09
Esters	27.3857	Acetic acid, trifluoro-, dodecyl ester	2771253	0.11
	17.7949	Trifluoroacetic acid, pentadecyl ester	2689417	0.11
	33.9322	Hexadecanoic acid, ethyl ester	454404	0.02

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Sample	RT (min)	Compound	Area	Proporsi (%)
	32.6063	Hexadecanoic acid, methyl ester	261072	0.01
Others	15.0002	Pyrazine, 2,3-dimethyl-5-(1-propenyl)-, (E)-	35648607	1.45
	25.6078	Diethyl Phthalate	27140400	1.11
	14.2154	Pyrazine, 3,5-diethyl-2-methyl-	26966860	1.10
	3.804	Pyrrole	25236955	1.03
	20.1197	Benzimidazole, 2-amino-1-methyl-	14698207	0.60
	19.864	cis,cis- and cis,trans-1,9-dimethylspiro[4.5]decane	13243440	0.54
	27.0527	1,1'-Biphenyl, 2,2',5,5'-tetramethyl-	11741831	0.48
	12.0629	Pyrazine, 2-ethyl-3,5-dimethyl-	11428577	0.47
	5.0229	Pyrazine, methyl-	10701780	0.44
	10.2019	Pyrazinamide	10516075	0.43
	8.7451	Dimethyl trisulfide	8752617	0.36
	7.6094	1H-Pyrrole, 1-ethyl-	7853387	0.32
	11.8727	Pyrazine, 2,6-diethyl-	7286555	0.30
	18.3241	2-Acetylaniline	7103782	0.29
	30.2814	2-Ethylhexyl salicylate	6229965	0.25
	16.8019	2-Isoamyl-6-methylpyrazine	5574816	0.23
	16.0883	Furan, 3-phenyl-	4618705	0.19
	17.4678	Benzeneacetaldehyde, α-ethylidene-	4451546	0.18
	21.7311	3,3'-Bis(1,2,4-oxadiazolyl)-5,5'-diamine	3743524	0.15
	19.9592	Disulfide, di-tert-dodecyl	3683982	0.15
	25.9468	2-(5-Aminohexyl)furan	3062843	0.12
	21.9987	2,6-Dodecadien-1-al	2942668	0.12
	25.4354	3-Methyl-4-phenyl-1H-pyrrole	2606719	0.11
	25.2987	Diethyltoluamide	2213525	0.09
	30.5668	Isopropyl myristate	2115230	0.09
	26.9754	Methyl dihydrojasmonate	2056626	0.08
	19.5073	Pyridine, 2-(1-methyl-2-pyrrolidinyl)-	2042665	0.08
	18.9127	2-Oxo-1-methyl-3-isopropylpyrazine	1647027	0.07
	22.9322	1,3,5-Triazine-2,4-diamine, 6-phenoxy-	1585132	0.06
	23.3543	Cashmeran	1550873	0.06
	18.55	3-Isoamyl-2,5-dimethylpyrazine	1543538	0.06
	31.2862	Galoxolide	1156008	0.05
	26.6365	3-Hydroxypyridine monoacetate	1026990	0.04
	31.53	1-Butyl 2-isobutyl phthalate	1023139	0.04

Sample	RT (min)	Compound	Area	Proporsi (%)
	33.409	Butyl 2-ethylhexyl phthalate	574468	0.02
		Total	2455945032	100

RT : *Retention Time* (minutes)

Aldehydes compound group were detected in the flavour powder sample, nonanal (9.91%) is the highest proportion on this group. Nonanal compounds are known to provide flavour characteristics of fatty, green, and citrus [13]. High aldehyde content can indicate that fish have undergone a high oxidation process, low aldehyde content indicates that fish have not been subject to high degradative oxidation [14].

The highest proportion of ketone compounds identified in the sample of mackerel flavoured powder are compound 1- (1H-pyrrol-2-yl) -ethenone with a proportion of 4.71%. ketones compounds are involved in the formation of aromatic reactions with food-making compounds and also these ketone compounds have flavour-forming properties [15]. The result of fatty acid oxidation and amino acid degradation that occurs during processing causes the formation of ketones. Most of the ketones are known to be present in volatile substances, it is assumed that these components are responsible for the aroma that resembles oxidized fish oil [16].

Based on Table 1 of the volatile compounds identified in the alcohol group the sample. Maltol compound in the sample of tenggiri's bones flavour powder has a proportion value of 11.43%. Aliphatic alcohols, aldehydes and ketones are thought to be largely formed by the oxidation of fats and fatty acids and by the degradation of amino acids during the processing. Fat oxidation is known as a peroxidation process and the process will increase along with decreased water activity. Hydroperoxide will form and then break down into aldehydes and ketones that are responsible for rancidity [15].

The hydrocarbon is the group that has the highest number of compounds compared to the other groups. some of the cyclic hydrocarbons identified in fish are the result of a secondary reaction from the thermal oxidation (heating) of the caratenoid and other unsaturated fats [9]. Mackerel's bones flavour powder sample has 38 compounds in the hydrocarbon group where pentadecane compound (3.05%) is the highest proportion compound in the sample of mackerel's bones powder. some of the cyclic hydrocarbons identified in fish are the result of secondary reactions from carotenoid oxidation (heating) and other unsaturated fats [9].

Organic acids found in the sample are produced from pyrolysis processes which function as inhibitors of bacterial growth [18]. The group of organic acid compounds in the meat powder sample and mackerel bone detected as many as 4 compounds in which nonanoic acid compounds with a proportion of 0.24% as the compound with the largest proportion value among other organic acid compounds. Nonanoic acid compounds are also found in raw and smoked blackfish [19].

The compound in the ester group that has the largest proportion value in the mackerel flavoured powder sample is trifluoro-, dodecyl ester with a proportion value of 0.11%. The esters in this sample generally come from the acid and alcohol esterification process that was previously formed from the results of fat metabolism [19].

Other groups successfully detected as much as 39 volatile compunds, nitrogen, furan, sulfur or other groups in fish. Pyrazine 2,3-dimethyl-5- (1-propenyl) -, (E) - is the compound with the largest ratio value of 1.45%, the second largest is Diethyl Phthalate (1.11%) and Pyrazine, 3, 5-diethyl-2-methyl- (1.10%). Diethyl Phthalate compounds are used in making perfumes, mosquito repellents, plastics and other products. Diethyl Phthalate compound is a clear liquid that does not contain and does not smell, which is heavier than air and insoluble in water so that it can dissolve in the air. Primary hazards that can be caused is that it can be used to spread to the environment, in addition to this it is also easy to penetrate the soil, pollute the air and nearby airways, irritate the eyes and irritate the skin [20].

4. CONCLUSIONS

Groups of compounds that were detected in the Spanish mackerel flavour powder generally derived from aldehydes, alcohols, hydrocarbons, organic acids, ketone, ester, and others (nitrogenous compounds and furans). The volatile components identification result of flavour powder had successfully detected 145 types of compounds with maltol (11.43%) is the highest proportion compound in this sample.

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