



# **VOLATILE COMPONENTS OF SPRAY DRIED FLAVOR POWDER FROM NARROW-BARRED SPANISH MACKEREL (*SCOMBEROMERUS COMMERSON*) IMMERSED WATER**

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## **Key Words**

*aroma, flavor powder, mackerel, proximate, Spanish mackerel, volatile component*

## **ABSTRACT**

Volatile flavor components could affect the aroma characteristics of a commodity. The objective of the study was to identify the components of volatile flavor compounds contained in spray dried flavor powder made from narrow-barred Spanish mackerel (*Scomberomerus commerson*) meat which immersed in water. Experimental method was used in this research with extracting volatiles by boiling the samples (65°C for 90 minutes) and drying in spray dryer (inlet and outlet temperature 170° C and 80° C, respectively). Subsequently, the sample was extracted using Solid Phase Micro-extraction (SPME) (80°C, 30 minutes) and the volatile developed were identified by Gas Chromatography/Mass Spectrometry (GC/MS). Additionally, proximate analysis was carried out to support the main volatile component results which were then semi quantified and descriptively analyzed. The volatiles component analysis successfully detected 81 compounds in flavor powder sample with *nonane, 3,7-dimethyl-* (6,43%) had the highest proportions compared to other volatile compounds. Most of the detected compounds derived from various groups such as aldehyde, alcohol, hydrocarbons, ketone, organic acid, ester and other group of compound. The proximate analysis result showed that the sample had 4.89% moisture content, 0.72% ash, 0.23% lipid and 1.14% protein content. Volatile components developed could be originated from product of auto-oxidation, enzymatic reactions and various reaction product which affected by high temperature processing parameters.

## 1. INTRODUCTION

Narrow-barred Spanish mackerel (*Scomberomorus commerson*) or “tenggiri” in local language, is one of the most popular sea captured fisheries commodity in Indonesia due to its high production volume and vast application in various traditional dishes. The captured production volume for this commodity in Indonesia on 2016 was amounted to 225.936 ton and it had been increased on the following year up to 438.658 ton [1]. Most of the Indonesian people used Spanish mackerel for the main ingredients in many traditional dishes such as *siomay*, *pempek*, crackers and *otak-otak* which were commonly found along various region in Indonesia.

This seawater fish species is widely known for its high nutrition content especially on protein and fatty acids composition. Moreover, people enjoy consuming it due to its unique flavor characteristics when it is applied on those various fish jelly-like dishes. Spanish-mackerel has 19.23% protein content according to [2] and most of them contained in the meat portion together with omega-3 fatty acids, minerals and vitamins which beneficial for human growth and health [3].

Every process performed in the fisheries processing industry or unit will generally produce liquid waste in form of water with all of the fisheries materials contain in it. Many traditional fisheries processing unit in Indonesia had not been optimally utilize this generated waste and they directly discharge it to the gutter, rivers or even soil. Traditionally, since centuries ago in Indonesia, waste which generated from washing or boiling process were utilized further to become several products such as fish sauce and a type of fish paste called *petis*. These proves that people from then had already acknowledge that fish processing waste water was still contain many useful components which can be utilized further as secondary products. Some of the components contained in them are known to affect product flavor and give its characteristics.

Each of the fisheries product possess its particular organoleptic characteristic, mainly due to its own unique flavor component composition as a result from chemical composition of the ingredients or the processing treatments [4]. Flavor characteristic considered as an important element in food acceptance [5]. Components which help to construct flavor perception could be divided into two categories, namely non-volatile components which influence taste sensation, and volatile components which influence aroma sensation of a product [6]. According to their physical form, flavor could be categorized into solid, paste and liquid [7]. Liquid flavor could be processed further into powder form by using drying procedures.

Currently, there are various drying machinery which commonly use to dry foodstuff. Several of them are drum dryer, conveyor dryer, spray dryer, cabinet dryer and so on. Drying food using spray dry is known to have several advantages compared to other machinery, namely for its high capacity throughput so it can produce higher volume of product, brief processing time and the operating procedures are relatively simpler [8]. The final product which produce from spray dryer is usually in fine dry powder form, for example commercial flavor powder. Flavor powder, which generally contain main ingredient extract, fillers and other material, has a distinctive aroma characteristic which could be influenced by its constituent volatile components.

Numerous research has been carried out concerning volatile aroma component on fisheries commodities for a particular purpose. Several of them were by [9] who studied volatile component on smoked salmon; [10], studied the effect of re-heating on silver carp (*Hypophthalmichthys molitrix*) volatile compounds and [11], who studied volatile component of wild and cultured sea bream (*Sparus uratus*) during storage. Whereas, such studies on fisheries commodities volatile component identification were rarely performed in Indonesia, hence the objective of this study are to identify volatile compounds and chemical composition of spray dried flavor powder produced from Spanish mackerel meat immersed water. The obtained information from this study are beneficial to recognize the aroma characteristic and provide basic data for future development of flavor powder produced from liquid waste.

## 2. MATERIALS AND METHODS

### 2.1 Samples Preparation

Fresh Narrow-barred Spanish mackerel fish samples were brought in cool box with chilled temperature from the fish landing site area in Indramayu, West Java, Indonesia. Sample preparation was performed at the Fisheries Product Processing Laboratory, Faculty of Fisheries and Marine Sciences, Universitas Padjadjaran, spray-drying process was carried out at the Central Laboratory, Universitas Padjadjaran. Proximate analyses were carried out at the Inter-University Centre Laboratory, Bogor Agriculture Institute, and volatile compound analysis were carried out at Flavour Laboratory, Indonesian Centre for Rice Research, Sukamandi, Subang, West Java.

Upon arrival at the preparation laboratory, samples were then washed with clean water, eviscerated and filleted for their meat. The meat portion were then immersed and washed in aquadest water with 1:2 ratios for 3 minutes in a stainless bowl. Afterward, the fillet was separated and the residual water was put in a boiler pot for heating in the course of 90 minutes with low heat temperature (maximum 65°C) [12]. The broth developed was then cooled and filtered in order to separate the undesirable remains. After the volume had been measured, the broth was then stored in a tightly closed glass bottle held in refrigeration temperature until it was used.

The broth samples were given 15% maltodextrin (based on broth volume) which act as a filler and mixed until homogenous [13]. Subsequently, samples were placed in glass bottle and brought to Central Laboratory and perform spray-drying process. The initial liquid flavor was dried in a spray-dryer machine with 170°C inlet temperature and 80°C outlet temperature in the course of 90 minutes

[8]. The dried sample was then packed in an aluminum foil which wrapped using cling wrap plastic, labeled and packed inside a zip-lock sealed plastic. This packaging was carried out to reduce the changes and damage which could be caused due to air, light and temperature [14]. As much as 30 g of sample were transported to Flavor Laboratory for performing volatile component analysis and 100 g were taken to a different laboratory for proximate analysis purpose.

## 2.2 Volatile Compound Analysis

Volatile compounds identification was performed based on [15] study with modification. Sample's volatiles extraction achieved with Solid Phase Micro-extraction with DVB/Carboxen/Polydimethylsiloxane fiber as the absorber. Water bath was used to heat the sample with 80°C temperature for 30 minutes. The Gas Chromatography (Agilent Technologies 7890A GC System) and Mass Spectrometry (MS) apparatus (Agilent Technologies 5975C Inert XL EI CI/MSD) was used in this study to identify various volatiles detected. GC column used was DB-5 (60 m x 0,25 mm x 0,25 mm), helium carrier gas, the initial temperature was 45°C (hold 5 minutes), temperature's escalation as much as 5°C/minutes, the final device temperature was 250°C (hold 5 minutes) with an overall running time 36 minutes and yielding chromatograms.

## 2.3 Proximate Analysis

All proximate analysis procedures were performed based on [16]. Moisture content analysis was determined with the gravimetric method; ash content was determined by combusting the samples in a muffle furnace on 550°C until the mass was constant. Protein content percentage was determined with Kjeldahl method and calculated as % nitrogen x 6.25 and Soxhlet system was used to determine percentage of total lipid content.

## 2.4 Data Analysis

The detected compound mass spectrums from the GC/MS were compared with the mass spectrums in NIST (National Institute of Standard and Technology) 0.8L library data base. Those data afterwards were analyze further using Automatic Mass Spectral Deconvolution and Identification System (AMDIS) software [17]. The data obtained from volatile component analysis and proximate analysis were then discussed descriptive-comparatively with various scientific references.

# 3. RESULTS AND DISCUSSION

## 3.1 Volatile Component

The result from volatile compound analysis showed that as much as 81 volatile compounds had been successfully detected in spray dried Spanish mackerel immersed-water flavor powder. The identified volatile compounds were classified into several group of compound, namely 11 volatile compounds originated from aldehyde group, 16 compounds from alcohols, 42 compounds from hydrocarbons, 2 compounds from ketones, 2 compounds from organic acid, 4 compounds from esters and other group of compound consist of 4 volatile compounds. Hydrocarbons group had the highest quantity of volatiles compared with other groups. Table 1 showed all the detected compounds and their proportion percentage based on each compound peak area as a semi-quantitative concentration from the highest compound abundance to the lowest.

Table 1. Volatile compounds detected in spray-dried Spanish mackerel immersed-water flavor powder

Groups	Retention Time	Compounds	Area	Proportion (%)
Aldehyde	12,2831	Nonanal	338127795	5,13
	2,2284	Pentanal	162414007	2,46
	1,7825	Butanal, 3-methyl-	112244686	1,70
	15,3512	Decanal	77561379	1,18
	11,2425	2-Octenal, (E)-	73514024	1,11
	26,6842	2-Nonenal, 2-pentyl-	20085124	0,30
	10,529	Benzeneacetaldehyde	16276372	0,25
	96609	2,4-Heptadienal, (E,E)-	13302075	0,20
	8,1804	Benzaldehyde	12424481	0,19
	6,5928	Heptanal	11650360	0,18
	7,9782	2-Heptenal, (Z)-	8251347	0,13
Alcohol	10,1187	1-Hexanol, 2-ethyl-	341805177	5,18
	4,2501	2,3-Butanediol	74839321	1,13
	16,7663	1-Dodecanol, 2-hexyl-	40803712	0,62
	18,6869	1-Dodecanol, 2-methyl-,	35474479	0,54

Groups	Retention Time	Compounds	Area	Proportion (%)
		(S)-		
	6,7593	Ethanol, 2-butoxy-	33240574	0,50
	21,321	1-Dodecanol, 2-octyl-	21574318	0,33
	17,355	1-Decanol, 2-octyl-	18091636	0,27
	23,3664	1-Hexadecanol, 2-methyl-	17793281	0,27
	25,4237	Ethanol, 2-(tetradecyloxy)-	9706196	0,15
	23,6756	1-Decanol, 2-hexyl-	9519013	0,14
	8,7452	1-Octen-3-ol	7904136	0,12
	25,8577	2-Ethyl-1-dodecanol	7765094	0,12
	26,3037	Ethanol, 2-(dodecyloxy)-	7531577	0,11
	6,3371	1,3-Butanediol	5688252	0,09
	7,6809	2-Heptanol, 6-methyl-	1732896	0,03
	31,3815	2-Ethyl-1-dodecanol	1633489	0,02
<i>Hydrocarbons</i>	12,6577	Nonane, 3,7-dimethyl-	424410938	6,43
	12,9431	Decane, 3,6-dimethyl-	334976461	5,08
	12,5328	Undecane, 5-methyl-	296878640	4,50
	11,7955	Decane, 3,7-dimethyl-	232771318	3,53
	11,4566	Tridecane, 6-methyl-	206280026	3,13
	11,635	Octane, 2,3,3-trimethyl-	195812413	2,97
	12,0036	Undecane, 4-methyl-	160490643	2,43
	13,1155	Hexane, 3,3-dimethyl-	151205710	2,29
	12,1106	Tridecane, 4-methyl-	147431706	2,24
	17,5274	1-Tridecene	142454922	2,16
	13,9836	Undecane, 2-methyl-	133926819	2,03
	14,1739	Undecane, 3-methyl-	121488111	1,84
	12,396	Undecane, 3,8-dimethyl-	110356685	1,67
	11,8966	Octane, 2,7-dimethyl-	102947500	1,56
	13,2701	Hexadecane, 3-methyl-	99654371	1,51
	13,1869	Hexane, 2,3,4-trimethyl-	96272686	1,46
	12,8182	Hexadecane, 2,6,11,15-tetramethyl-	94580123	1,43
	13,4425	Undecane, 5,7-dimethyl-	91508784	1,39
	15,1788	Dodecane	69338279	1,05
	20,4945	Undecane, 2-methyl-	57631693	0,87
	22,6647	1-Pentadecene	54623440	0,83
	10,7728	Undecane, 3,6-dimethyl-	52828451	0,80
	11,0998	Dodecane, 2,7,10-trimethyl-	46440138	0,70
	15,6306	6-Tridecene, (Z)-	44962942	0,68
	19,1923	2,6-Octadiene, 2,6-dimethyl-	40136035	0,61
	18,2945	5-Tetradecene, (E)-	36121394	0,55
	14,7982	9-Eicosene, (E)-	34166116	0,52
	14,7388	6-Dodecene, (Z)-	32713425	0,50
	17,7415	Tridecane	32385180	0,49
	27,0469	1,3-di-iso-propylnaphthalene	25000482	0,38
	19,6026	1-Undecene	23496048	0,36
	18,0566	Decane, 3,8-dimethyl-	21239097	0,32
	15,482	Nonane, 4,5-dimethyl-	19239317	0,29
	22,0345	9-Octadecene, (E)-	15403429	0,23

Groups	Retention Time	Compounds	Area	Proportion (%)
	17,1528	3-Tetradecene, (E)-	15079732	0,23
	27,9804	2,6-Diisopropyl naphthalene	12449327	0,19
	28,1172	1,7-di-iso-propyl naphthalene	8625546	0,13
	26,3869	Pentadecane	8512363	0,13
	27,7664	Naphthalene, 1,2,3-trimethyl-4-propenyl-, (E)-	6120068	0,09
	28,0518	1,4-di-iso-propyl naphthalene	5507625	0,08
	30,8464	1-Octadecene	4715134	0,07
	31,6075	Nonadecane	1836546	0,03
<i>Ketones</i>	8,8998	Cyclobutanone, 2,3,3-trimethyl-	9333946	0,14
	8,436	Acetophenone	8461718	0,13
<i>Organic Acids</i>	20,8334	cis-Vaccenic acid	25829848	0,39
	6,1587	2-(2-Butoxyethoxy)acetic acid	3226250	0,05
<i>Ester</i>	3,0371	Acetic acid, methoxy-	177075163	2,68
	10,9452	Oxalic acid, isobutyl nonyl ester	59374438	0,90
	18,9842	Sulfurous acid, hexyl tridecyl ester	40887506	0,62
	20,7442	Pentafluoropropionic acid, dodecyl ester	11918181	0,18
<i>Others</i>	9,2328	Pyrazine, trimethyl-	334566318	5,07
	9,9285	Acetylpyrazine	15680640	0,24
	6,456	3-Furanmethanol	5417397	0,08
	31,0485	Dibutyl phthalate	2798341	0,04

Hydrocarbon compounds showed in Table 1 includes aliphatic, aromatic and other hydrocarbon derivatives other than furan and phenol. The highest hydrocarbon compound proportion respectively were 3,7-dimethyl-nonane, (6,43%), 3,6-dimethyl- decane, (5,08%) dan 5-methyl- undecane, (4,50%). Nonane, decane and undecane were previously identified in smoked catfish [14] and star anise seed [18]. Hydrocarbons compound group are known to have high aroma threshold; hence they are often considered to have a minimum impact on overall product aroma. This matter is in accordance with [19] which stated that alkanes compound group are not generally contribute significantly to food odor. Most alkanes compound are common detected in seafood [14].

Volatile alcohols were also identified as much as 16 types compounds with 2-ethyl-1-hexanol (4.72%), 2,3-butanediol (1.03%) had the highest proportion respectively. Alcohol compounds can be formed as a result of lipid oxidation and fatty acids as well as by the degradation of amino acids during the processing (Ho et al. 1994). 2-ethyl-1-hexanol were previously detected in smoked salmon [11]. The second most abundant alcohol compound was 2,3-butanediol which known to develop from hydroxylation in C2 and C3 carbon atoms, namely butanediol, glycol and secondary alcohols. 2,3-butanediol compounds were previously identified in mackerel and cod [20]. This compound is responsible for the distinctive aroma of cocoa found in cocoa butter. Moreover, this compound was known to use as a precursor in plastic material [21]. Certain alcohol compounds have lower odor thresholds compared to aldehydes and ketones groups [9].

Aldehyde compounds are one of the contributors to volatile compounds that are often found in fish [10]. This group of compounds can be considered as a result of the lipid autoxidation process [11]. As much as 11 aldehyde compounds were successfully detected from the flavor powder sample. Nonanal (5.13%) was the compound which had the highest proportion in this aldehyde group. Nonanal is an aldehyde compound which could derived from unsaturated lipid. It is known to have green, citrus and fatty aroma characteristics.

Various concentration of nonanal is also often found in fishery commodities, such as shellfish and various fresh fish [22]. Nonanal was also found as one of the main compounds that contribute to fish oil [23].

Ketone compounds group are commonly found in fish and also detected in the flavor powder sample. Ketone compounds which have the highest abundance proportion respectively were 2,3,3-trimethyl-cyclobutanone (0.14%) and acetophenone (0.13%). The compound of 2,3,3-trimethyl-cyclobutanone was previously identified in the mackerel fish broth [24]. Whereas, acetophenone compounds have a role as agents for photosensitization and animal metabolism [25]. The ketone group contributes to flavor development and is involved in the formation of aromatic reaction products with other food constituents [14]. The ketones identified in the flavor powder sample could be derived from lipid oxidation (especially fatty acids) during the heating process either in the boiling or drying process using spray-dryer. This was in accordance with [26] which stated that chemical reactions during boiling process could produce numerous volatile compound which contribute to overall aroma and meat flavor which most have also been detected in the sample.

The esters were also detected in flavor powder sample as much as 4 compounds. The highest proportion ester which identified was methoxy- acetic acid (2.68%) which belongs to the family of methoxyacetic acid [27]. Esters which detected in fish could be derived from acid esterification with alcohol which was previously formed from lipid metabolism [28].

There were two organic acid compounds which were detected in flavor powder sample. Acid can be formed by triglyceride hydrolysis in fish [29]. Several organic acids produced from the pyrolysis process could serve as an inhibitor of bacterial growth [30]. Organic acid compound with had the highest proportion was cis-Vaccenic acid (0.39%). The cis-Vaccenic acid compound is an omega-7 unsaturated fatty acids [31]. Cis-Vaccenic acid compounds are often identified in plant sources as in olive oil. The cis-Vaccenic acid compound was previously detected by [32] in fish waste.

Volatile compounds group apart from previously mentioned groups which also contribute to the flavor powder sample was categorized as other group (includes furan or sulphurous and nitrogenous contain compounds). Compound which had the highest proportion value from this group was trimethyl-pyrazine (5.07%). Pyrazine compound is one of the important components in the formation of flavor in foods that are processed using high temperatures such as in chocolate and meat [33].

### 3.2 Proximate Analysis

Proximate analysis performed on spray-dried flavor powder sample consist of moisture, ash, lipid and protein content based on wet basis determination which expressed in percent. The average results of sample's proximate analysis are showed in Table 2. This type of analysis is performed to recognize the chemical composition and overall description of several macro nutrition which the sample has. The value measured is usually affected by the initial raw material chemical composition, the type of the ingredient and processing stages which the material has been undergo [14].

Table 2. Proximate analysis results of spray-dried Spanish mackerel immersed-water flavor powder

Parameters	%
Moisture	4.89
Ash	0.72
Lipid	0.23
Protein	1.14

As we can see from Table 2., spray dried flavor powder sample had 4.89% moisture content. The most important aspects which influenced moisture content on a dry product none other is the drying process in this case was spray-drying process. The higher the temperature and the longer the residence time, the less water will be contained in a product. According to [34], moisture content of a product is also influenced by the initial water content of the raw material in this case the amount of water trapped in prior boiling process. The moisture content of a dry product would also be influenced by the environment (humidity, water vapor or moisture) mainly due to the more dried up the material is become, the more hygroscopic it would be. Hence, it was possible for this product to reabsorb water from the surrounding environment during the preparation or sample storage. This matter could be also affected by the addition of maltodextrin. Maltodextrin is known to have low hygroscopic properties, consequently, that it is more difficult for maltodextrin to absorb water vapor again [35].

The results showed that spray dried flavor powder sample had 0.72% ash content. The ash content value could be affected by initial minerals content and composition of the raw material and the temperature used for heating the sample. Ash content of a product shows the total minerals contained in it [36]. Fish meat are well recognized to contain various minerals [37] and its value could be affected by species, growth phase, feed and environmental aspects [14].

Lipid content was also determined for this sample and the result showed that it had 0.23%. According [38] study, meat portion of fresh and steamed mackerel had 0.17 and 0.24% lipid content respectively. The value measured could be affected by the part of the fish or fish meat which are being analyzed as they differ in fat tissue. Lipid and moisture content have a negative relationship, hence if the lipid content value is low then the moisture content would be high. Lipid content value could also be affected by oxidation reaction [39] especially during heating process. Low levels of lipid in fish could be caused by environmental factors and loss of water and fat

during the heating process. The lipid content in fish found in meat directly affects the intensity of the aroma and taste [38]. The amount of several volatile components such as aldehyde, ketones and esters are affected by lipid content and composition.

The proximate analysis results on Table 2. showed that the sample had 1.14% protein content. According to [40], the measured protein content depends on the amount of ingredients added and is largely influenced by the moisture content of the raw material. Heating processes such as boiling and drying can have an effect on the structure and functional properties of raw material proteins. The protein could undergo denaturation caused by temperature changes during the heating process [41]. The various types of volatile compounds detected in the sample mostly originated from the ingredient itself, especially from protein and lipid content and composition, thus the number and variety of types of volatile compounds are related to variations in the chemical compounds contained in the sample [38].

## Conclusion

Based on the results spray-dried flavor powder from Narrow-barred Spanish mackerel immersed water were still containing many volatiles, hence it can potentially be used as a flavor powder product and needs further research. Most volatile compounds group that were detected in the sample were from aldehydes, alcohols, hydrocarbons, organic acids, ketones, esters and others compound group. Volatile compound analysis successfully detected 81 types of compound with 3,7-dimethyl-nonane (6.43%) had the highest proportion of peak area count. Proximate analysis results showed that the sample had 4.89% moisture content, 0.72% ash, 0.23% lipid and 1.14% protein content.

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