



## **Volatile Flavor Compounds Composition of Steamed Squid (*Loligo* sp.)**

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### **Authors' contributions**

*This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.*

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### **ABSTRACT**

Squid (*Loligo* sp.) is one of the marine fishery commodities that abundant in resources and valuable economically in Indonesia. To increase the economic value of food, processing activities are needed, steaming is one of them. Processing activities using high temperatures are suspected to affect the composition of flavor compounds in fishery products. Flavor volatile are components that act as aroma, the initial impression (top notes), and evaporate easily. This research is to study and identify volatile compounds composition found in steamed squid. The squid was taken from Indramayu, West Java, subsequently the sample preparation was conducted at the Laboratory of Fishery Processing Technology, Faculty of Fisheries and Marine Science, Universitas Padjajaran. Volatile components were analyzed at Flavor Laboratory, Indonesian Center for Rice Research, Sukamandi, Subang from February to April 2021. Volatile compounds were analyzed using Gas Chromatography / Mass Spectrometry (GC / MS) with an extraction temperature of 80°C in 45 minutes (Solid Phase Micro Extraction). The proximate analysis was analyzed at Inter-University Centre Laboratory, Bogor Agricultural Institute. The volatile compound analysis successfully detected 30 compounds in the steamed squid sample. The proximate analysis showed steamed squid contained 74.99% water content, 1.49% ash, 2.09% fat, and 21.12% protein.

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## 1. INTRODUCTION

Indonesia has great potential in marine fishery commodities. Potential marine fishery resources include large pelagic fish, small pelagic fish, shellfish, shrimp, lobster, and squid. Squid is a marine product that is quite abundant in Indonesia's marine resources. Other than that, squid is one of the marine fishery commodities that is popular and in demand by the public which can be seen by export value. The export value of squid in 2016 increased significantly by 58.62% [1].

Squid meat has advantages over other marine fishery commodities, squid does not have a backbone, easy to digest, has a distinctive taste and aroma, and contains essential amino acids such as leucine, lysine, and phenylalanine that humans need. The dominant levels of non-essential amino acids are glutamic acid and aspartic acid which contribute well to the emergence of tasty and savory effects [2]. To increase the economic value of food, processing activities are needed, steaming is one of them.

Steaming is one of the processing methods that utilize the heat of water steam. Food processing can affect the nutrition, physical form, and chemical composition of the product. Heat processing is a method that causes myosin denaturation at  $\pm 50^{\circ}\text{C}$  and actin at  $\pm 70^{\circ}\text{C}$  [3]. Changes in protein structure can affect the texture and delicacy of squid meat. Steaming can make physical changes and chemical reactions of a food product, especially its texture and flavour [4]. The steaming process has a number of advantages. One of them, is that it can minimize the risk of loss of vitamins and other food compounds that are sensitive to high temperatures [5]. The steaming process will affect the number of new volatile flavor compounds due to thermal oxidation and fatty acid decomposition, especially unsaturated fatty acids [6]. Processing using high temperatures is suspected to affect the composition of flavor compounds in fishery products [7]

Flavor is a complex component because it can be volatile or non-volatile and can change due to time and processing conditions [5]. Volatile components are components that give the sensation of smell, give the initial impression (top notes), and evaporate quickly. Oxidation caused by environmental factors such as air, light, and

temperature (heat) can affect the flavor of a food product. Changes in volatile flavors can be affected by air, light and temperature [7]

Volatile components are taste components that act as aromas, perceived by aroma receptors from olfactory organs such as the olfactory tissue in the nasal cavity. Flavor molecules (flavor volatile) are released from food in the oral cavity and volatile components move through the nasopharynx in the nose when food is swallowed [8].

There are a number of studies about the analysis of squid volatile compounds by extraction. Jin et al. identified 52 compounds in squid, using the simultaneous distillation-extraction method [9]. Huang et. al (2018) identified volatile flavor characteristics in fermented squid (88 volatile compound), using Solid Phase Microextraction (SPME) method [10].

The composition of volatile compounds detected in fishery products usually comes from groups of aldehyde, ketone, alcohols, organic acids and hydrocarbons compounds [7]. Therefore, it is possible that hydrocarbons, aldehydes, ketones, and alcohols are the volatile compounds contained in steamed squid.

The purpose of this research is to study and identified volatile compounds composition found in steamed squid.

## 2. MATERIALS AND METHODS

### 2.1 Time and Place

This research was conducted from February-April 2021. The study was conducted in the Fishery Product Processing Laboratory, Faculty of Fisheries and Marine Sciences, Universitas Padjajaran, Sumedang Regency, West Java, Indonesia for preparation sample. Volatile components were analyzed at Flavor Laboratory, Indonesian Center for Rice Research, Sukamandi, Subang.

### 2.3 Materials and Tools

The materials used are squid (*Loligo sp.*), water, HCl,  $\text{CuSO}_4$ ,  $\text{K}_2\text{SO}_4$ , NaOH,  $\text{H}_2\text{SO}_4$  (sigma aldrich), kloroform. The tools used steamer (SP-32-SS2 Supra), Gas Chromatography (Agilent Technologies 7890A GC System) / Mass

Spectrometry (Agilent Technologies 5975C Inert XL EI CI/MSD), waterbath, kjeldahl's flask, soxhlet's flask, knife, cling wrap, aluminium foil, plastic zip-lock, scale, stove, coolbox, label.

### 2.3 Research Procedure

A 2.5 kg squid sample was taken from Karangsong fish landing site, West Java. A cool box and 5 cm thick of ice cubes were used to prevent the propagation of heat from the air on the outside. Coolbox which already contains samples of squid then brought to the Laboratory of Fishery Products Processing Universitas Padjajaran to be prepared.

The procedure for preparation sample volatile compounds was carried out based on research modification by Pratama [7]. The preparation process including cleaning and washing the squid then remove the tentacles, head, internal ink, internal thin shell (visceral content) and only kept the mantle. After that the sample was steamed at 100°C, the squid meat was packaged using aluminum foil, then wrapped by cling wrap, and lastly put into zip-lock plastic. The three-layered packaging step is done since air, light, and temperature can change and damage the flavor of the sample [7]. The finished sample is then placed in a coolbox containing a sufficient amount of ice packaged in plastic to keep the temperature cool (10-15°C). The samples were then transported to the laboratory from the Preparation Laboratory in Universitas Padjajaran to the Flavor Laboratory in Sukamandi for approximately 90 minutes, and then continued to the Integrated Laboratory in Institut Pertanian Bogor, which took around 120 minutes to be analyzed for their volatile compounds and proximate composition.

The procedure for analyzing volatile compounds was carried out based on research modification by Pratama [7]. Analysis of the volatile compounds was done using a series of Gas Chromatography (Agilent Technologies 7890A GC System) and Mass Spectrometry (Agilent Technologies 5975C Inert XL EI CI / MSD) apparatus. Sample extraction was carried out using the Solid Phase Micro Extraction (SPME) method to evaporate volatile compounds in the sample, DVB/Carboxen/Poly Dimethyl Siloxane fiber was used as an absorbent for volatile flavor compounds. The extraction temperature is 80°C in 45 minutes (in waterbath). The GC column used is HP-INNOWax (30m x 250µm x 0.25µm), helium carrier gas, initial temperature 45 °C (2-

minute hold), increase in temperature 6 °C / minute, final equipment temperature 250 °C (hold 5 minutes) for 45 minutes.

Proximate analysis performed on samples of steamed squid includes moisture, ash, lipid and protein content based on the AOAC [11] procedure. Lastly, the data generated from the laboratory test of steamed samples were analyzed in a descriptive and semi quantification intensity of the compounds detected from the analyzed samples.

### 2.4 Data Analysis

The spectra of the detected compound mass were compared to the mass spectra patterns found in the data center or the NIST version 0.5a library (National Institute of Standard and Technology). The data of the volatile flavor compound components were analyzed further using the Automatic Mass Spectral Deconvolution and Identification System (AMDIS) software [12]. The data from the analysis of volatile compounds produced were discussed using a comparative descriptive study based on the identification and intensity of the semi-quantification compounds that were detected in the tested samples [7]. Data obtained from the proximate analysis of the steamed samples were calculated by the average value and standard deviation based on [13] were discussed in descriptive.

## 3. RESULTS AND DISCUSSION

### 3.1 Analysis of Volatile Compounds

The analysis results of squid (*Loligo* sp.) volatile flavor compounds showed that there are 30 volatile compounds in the steamed squid sample (Table 1). Volatile flavor compounds identified can be categorized into several major categories of compounds such as hydrocarbons, aldehydes, alcohols, ketones and their derivatives.

Hydrocarbons, aldehyde, alcohols, ketones, acid compound are the main volatile compound in fishery product [6].

The hydrocarbon group in the steamed squid sample consists of 8 compounds. The highest proportion of compounds in the hydrocarbon group is Pentadecane (48.50%). Pentadecane compound is formed from palmitic acid, which is the most common saturated fatty acid found in animals, plants, and microorganisms [14]. If the

fatty acid composition in a sample is high, the pentadecane concentration is also high [15]. Pentadecane compounds were also detected in the study of Kim et al. (2004) on dry squid (*Todarodes pacificus*) with relatively large numbers [15]. Most of the hydrocarbon group compounds show a high flavor threshold, but if this group of compounds has a high concentration, it will play an overall role in the flavor of a sample [6].

The aldehyde group identified in the steamed squid sample consist of 9 compounds. Hexadecanal is a long-chain fatty aldehyde that

is released from plasmalogens by hydrolysis. It is found in the sample as the compound with the largest proportion (5.49%). Plasmalogens are lipids containing an aldehyde chain [16]. The detected aldehyde group compounds come from the oxidation of the double carbon bonds of unsaturated fatty acids or saturated fatty acids [6]. Most of the aldehyde group compounds have been known to have aromas such as green plants, grassy, dark chocolate, malty, fatty, sweet floral, apple-like, melon-like, nutty, and fruity, are found in various fresh fish and shellfish with various concentrations [17].

**Table 1. Volatile compounds identified in steamed squid**

No	Group	RT	Compound	Area	Proportion(%)
1	Hydrocarbons	24.5286	Pentadecane	5830601	48.50
2		28.2967	Hexadecane	4254743	35.39
3		18.3348	Naphthalene	60274	0.50
4		20.4476	Undecane	87441	0.73
5		23.3408	Naphthalene, decahydro-1,6- dimethyl-4-(1- methylethyl)-	12132	0.10
6		25.9967	1,3,6-Heptatriene, 5- methyl-	1585	0.01
7		31.4921	Tetradecane	118732	0.99
8		33.4355	Oxirane, tetradecyl-	197918	1.65
9	Aldehyde	4.8694	Pentanal	133878	1.11
10		13.6417	Octanal	38023	0.32
11		17.9488	Nonanal	23179	0.19
12		20.2888	2-Hexenal, (E)-	23919	0.20
13		25.868	Heptanal	936	0.01
14		26.7087	2-Octenal, (E)-	29034	0.24
15		29.8628	2-Pentenal, (E)-	337	0.00
16		30.2371	Hexadecanal	659628	5.49
17		32.7808	2,6-Nonadienal, (E,Z)-	1000	0.01
18	Alcohol	5.2029	2-Penten-1-ol, (E)-	122858	1.02
19		22.806	2-Octen-1-ol	26271	0.22
20		27.8214	3-Ethyl-4- methylpentan-1-ol	53769	0.45
21		28.0599	1-Heptanol	7482	0.06
22		31.2778	1-Nonanol	17941	0.15
23		31.8711	2-Nonen-1-ol	41665	0.35
24		34.3918	1-Hexanol	1641	0.01
25	Ketones	14.4487	5-Hepten-2-one, 6- methyl-	9823	0.08
26		17.3018	3,5-Octadien-2-one	24429	0.20
27		20.4045	2-Heptanone	60690	0.50
28		20.434	2,3-Pentanedione	2942	0.02
29	Ester	26.3928	Carbonic acid, prop- 1-en-2-yl tridecyl ester	179204	1.49
30	Others	31.279	Furan, 2-ethyl-	940	0.01

The alcohol group in the steamed squid sample consists of 7 compounds. The highest compound proportion in steamed sample is 2-penten-1-ol, (E)- (1.02%). The compound 2-penten-1-ol was also found on the freshness level of whiting fish (*Merlangius merlangus*) [18]. In his research, 2-penten-1-ol was one of the compounds that have a significant higher number found in samples with very good freshness quality category. The compound 2-penten-1-ol is the relevant compound found during the decomposition process with different quantities. The group of alcohol compounds identified in fish is formed from the oxidation of fats, fatty acids and degradation of amino acids [19]. Alcohol compounds are generally formed from the decomposition of secondary hydroperoxides of fatty acids [20]. Alcohol generally provides fruity, oral and grassy aspects to fish [6].

The ketone group identified in the sample of steamed squid were consisted of 4 compounds. 2-Heptanone was the highest compound found in the sample (0.50%). 2-heptanone compound can give a cream and cheese-like aroma (2-heptanone: aroma threshold 140 ppb) [21]. These ketones may be products of microbial oxidation, lipid oxidation, or amino acid degradation [22]. Compounds of the ketone group can be produced from thermal oxidation (steaming) and unsaturated fatty acid degradation, amino acid degradation or oxidation by microorganisms [6].

The ester group in the steamed squid sample consists of 1 compounds that is carbonic acid, prop-1-en-2-yl tridecyl ester (1.49%). The group of ester compounds present in this sample generally comes from the esterification process of acids and alcohols that were previously formed from the results of fat metabolism [23].

The variation of numbers and types of volatile compounds are related to variations in the chemical compounds contained in the sample, mostly in the protein and fat [23], and the remainder are from aquatic environment where the fish lived [24].

These volatile compounds are created as of enzymatic reactions, microorganism activity, fat auto-oxidation, compounds formed by various thermal reactions and environmental influence, and they play a role of the commodity's aroma [17]. The type and composition of volatile compounds will depend on the type of sample,

chemical composition and processing method [23]

In previous research on flavor components in dried horse mackerel, it was stated that the flavor formed was the result of fat oxidation and also hydrolysis involving enzymes. Fat oxidation and hydrolysis that occur are derived from components of fish origin such as fat and protein. In addition, thermal conditions can increase the retro-aldol degradation of unsaturated aldehydes which can affect the flavor of the product [25]. The extraction method, type of sample, GC/MS column and the parameters of the use of the tool can also affect the number and types of compounds detected [7]

### 3.2 Proximate Analysis

Proximate analysis also performed in this research. The squid has high nutrient content, but the process involving thermal processing in general can lead to decreased nutrient content in the squid. The chemical composition of steamed squid can be seen in Table 2. From the table below, the steamed squid contained 74.9% water, 1.49% ash, 2.09% fat, and 21.12% protein.

Processing process such as steaming can decrease water content in the sample. This phenomenon is because the steamed squid is given steaming treatment at 100°C for 30 minutes which leads the water to come out. The loss of the moisture from the intercellular space caused by the steaming process increases the density of food. This is what causes the water content of the steamed commodity sample to be measurably lower than the fresh sample [5].

The ash in this product is more caused by the mineral content rather than from the processing stage of the raw material since the sample were not given any certain mineral salts, salting process, or even certain additives at all stages of the steaming procedure. The ash content in the sample comes from the mineral content in the meat. Ash is an organic substance remained from the combustion of an organic material and it describes the total amount of minerals in the material [7].

The steaming process will cause the fat content to increase. The increase of the fat content was because of the water loss. The more water comes out of the material, the greater the amount of fat content (and other nutrient levels) measured in the proximate test [7].

**Table 2. Proximate analysis samples squid steamed**

Samples	Water(%)	Ash (%)	Fat (%)	Protein (%)
Steamed squid	74.99 ± 0.09	1.49 ± 0.07	2.09 ± 0.19	21.12 ± 0.02

The steamed squid sample contained 21.1% protein. The protein content in the sample studied were affected by the environment where they lived, seasons, conditions and duration of storage and processing. The steaming process can change the protein properties of the steamed material. Similar to the fat, protein content measured in the proximate test was also increased because of the water loss during the steaming process [7]. The water content in the material will also have a major effect on the measured protein content in the material [26].

#### 4. CONCLUSION

There are 30 volatile compounds found in steamed squid. The compounds found can be categorized into hydrocarbon, aldehyde, alcohol, ketone, and other groups. The group of volatile flavor compounds identified in steamed squid are hydrocarbon (8 compounds), aldehydes (9 compounds), alcohols (7 compounds), ketones (4 compounds), esters (1 compound), and 1 other compound. Pentadecane was the largest compound found in steamed squid (48.50%). The steamed squid contained 74.99% water content, 1.49% ash, 2.09% fat, and 21.12% protein.

#### COMPETING INTERESTS

Authors have declared that no competing interests exist.

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