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VOLATILE COMPONENTS OF SPRAY DRIED FLAVOR POWDER FROM WHITE SHRIMP (*LITOPENAEUS VANNAMEI*) IMMERSED WATER

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Key Words
aroma, flavor powder, proximate, white shrimp, volatile component

ABSTRACT

This research aims to identify volatile compounds which are the flavor powder from spray dried white shrimp immersed water aroma constituents. The research method was experimental by identifying the volatile compounds and proximate composition of previously mentioned flavor powder. Initially, the white shrimp meat immersed water were boiled (65^{o}C, 90 minutes) until it became liquid broth and then spray dried (inlet and outlet temperature 170^{o} C and 80^{o} C, respectively). Solid Phase Microextraction (SPME) method was used to extract the volatile components from the sample (80^{o}C, 30 minutes) and Gas Chromatography /Mass Spectrometry (GC/MS) was used to identify the volatile flavor compound's composition. Moreover, the sample were determined for its moisture, ash, protein and lipid content to support the main data. The resulting data were then discussed descriptively based on the identification and semi-quantification of the compounds detected in the sample. The analysis result succesfully detected 93 volatile compounds in spray dried white shrimp immersed water flavor powder sample. Volatile compounds detected were mostly derived from hydrocarbons, alcohols, aldehydes, ketones, esters, organic acid which could be derived from auto-oxidation and enzymatic reactions, also various product from reaction which affected by processing parameters. Compounds that had the largest proportion is pentanal (8,992%). The proximate analysis results showed that the samples had a moisture content of 4,56%, 1.45% ash content, 5,43% protein content and 0,07% lipid content.

1. INTRODUCTION

Shrimp is one of the main fishery export commodities which highly contributes to Indonesia's revenue from this sector. Based on data from the [1], shrimp ranked first in aquaculture commodities production with the value in 2017 at 58,135,246 tons in quantity. One type of the exported shrimp is vaname shrimp (*Litopenaeus vannamei*). This shrimp comes from the subtropical area of the American west coast. Vaname is one type of shrimp that is often cultivated. This is because the shrimp have promising prospects and profits [2].

Vaname shrimp is a type of shrimp that is in great demand as foodstuff and is used as raw material for various processed products. Food derived from white shrimp typically has a distinctive aroma, delicious combintion of savory and sweet taste, and contains beneficial nutrition for health. [3] states that the composition of shrimp consists of various nutrients composition such as essential amino acids, fatty acids, macro and micro minerals. White shrimp as raw material will need to go through a number of processing stages prior being altered into food processed product. This stage generally will produce several types of waste, one of which is liquid waste.

Liquid waste is a byproduct of processed materials and has not been used or not utilized mainly due to it has a strong and distinctive odor owing to its high organic matters. White shrimp liquid waste can still be utilized, commonly as shrimp broth or shrimp paste if properly processed. The processing stages can be done by applying high or low temperature and fermentation. According to [4], processing stage could alter the texture characteristic and could produce distinctive properties such as the change in flavor on each of the processed product.

Flavor is the combine sensation of signals from taste and aroma [5]. Flavor arises because of the particular compounds (flavoring agents) although it is available in very small amounts [6]. The flavor characteristics of a commodity can be influenced by the various compounds contained in it. These compounds are commonly divided at least into two groups, namely volatile and non-volatile compounds [4], [7], [8], [9]. Flavor in meat can generally be formed due to the cooking process which involved heat. The heating process is oftenly used to reduce food moisture content in its ingredients or more commonly referred to as drying process.

Drying is the evaporation of moisture content into the air due to differences in the content of water vapor between the material that is dried with the surrounding air, where the water vapor content is lower or has a low relative humidity so that eventually evaporation occurs [10]. [11] states that the drying process can be performed by natural and artificial sources (artificial drying) for example using oven as a dryer. The artificial drying process can also be done with more modern machinery such as spray-dryer. Spray dryer can effectively transform materials that have high moisture content into dry powder in various shapes and qualities.

Flavor powder is one type of flavoring agent that has been dried into solid form. Based on the statement from [12], the dry flavor powder can be obtained through the absorption process by certain dry carriers or encapsulated by inert edible polymers such as Arabic gum or starch. Powder product quality is determined by moisture content, ash content, fineness level, metal contamination, arsenic contamination, microbial contamination, and its taste and aroma. Flavor powder can be found on the market in the form of seasoning powder which is generally extracted from certain types of meat and subsequently packed.

This research provides valuable information in recognizing the various components which formed volatile flavor or aroma. Aroma as widely known is one of the most determining components in food preferences. Research on volatile flavors has been extensively carried out for many years. [13] studied volatile compounds in roasted shrimp; [14] studied volatile flavor components of crayfish processing waste; [15] studied the volatile component from several popular Mediterranean seafood species.

However, in Indonesia similar studies and basic information regarding volatile flavor compounds identification of fishery products were still rarely found. Several studies that have been carried out in these past few years were [16] who studied the volatile component composition of fresh and steamed tiger shrimp (*Penaeus monodon*); [17] studied the composition of volatile flavor compounds in fresh and steamed tiger grouper (*Epinephelus fuscoguttatus*). Fundamental study on the volatile compounds composition needs to be performed and the resulting data could be used for further flavor related research. The purpose of this study was to identify the volatile compounds contained in the spray dried flavor powder from white shrimp immersed water.

2. MATERIALS AND METHODS

2.1 Samples Preparation

Fresh white shrimp samples as much as 5 kg were taken from Karangsong, West Java fish landing site and transported using a chilled temperature coolbox to the Fishery Product Processing Laboratory, Universitas Padjadjaran for preparation treatment. Shrimp samples were cleaned and separated between the head, shell and meat parts. The parts were then immersed and wash in aquadest water with meat: water ratio 1:2 for 3 minutes in a stainless bowl. Afterwards, the shrimp was filtered and the residual immersed water was heated for 90 minutes with low heat temperature (maximum 65°C) in a boiler pot until it became broth [18]. Filler (maltodextrin 15%) was added to the broth samples based on its volume and mixed until homogenous [19]. The thicker liquid broth was later dried by a spray dryer at Central Laboratory, Universitas Padjadjaran with an inlet temperature of 170°C, an outlet temperature of 80°C and flow speed of 15 ml/minute. Moisture content will decrease with increasing dryer temperature [20].

The yielded powder was weighed first and then packaged using aluminum foil, cling wrap, placed into a Zip-lock plastic and finally into a glass jar. This was done to minimize the possible changes and damage which could occured to the sample's flavor and could be caused by air, light and temperature [9]. Finished packaged samples were placed in a chilled temperature coolbox to be transported to the Inter-University Centre Laboratory, Bogor Agriculture Institute for proximate analysis and Flavor Laboratory, the Indonesian Center for Rice Research for performing the volatile compounds analysis.

2.2 Volatile Compound Analysis

Analysis of volatile flavor compounds was carried out based on the modification of study conducted by [9]. Volatile flavor compounds analysis was performed using a series of Gas Chromatography (GC) and Mass Spectrometry (MS) apparatus. Sample extraction was carried out using the Solid Phase Micro Extraction (SPME) method using DVB/Carboxen/Poly Dimethyl Siloxane fiber. The sample extraction temperature in the SPME method used was 80^{o}C for 30 minutes and performed using waterbath. Flavor powder samples were taken as much as 6 grams and then placed in a particular vial for SPME with size of 22 ml. The black, fused silica fiber used had a film thickness of 75 µm. This fiber must go through the conditioning stage first by heating at 300°C for 1 hour before it was used to absorb the volatiles from sample's headspace. Subsequently, the fiber is injected into the vial, between the sample and the edge of the vial cap (headspace). After the extraction was finished, the fiber was removed and injected into the gas chromatograph sample injector (Agilent Technologies 7890A GC System) and Mass Spectrometry (Agilent Technologies 5975C Inert XL EI CI / MSD) to identify the detected compounds and yielding chromatograms. The GC column used was DB-5 (60 m x 0,25 mm x 0,25 mm) with helium carrier gas and an overall running time 36 minutes.

2.3 Proximate Analysis

All proximate analysis procedures were performed based on AOAC 2005 [21]. Moisture content a was determined according the gravimetric method; ash content was determined by combusting the samples in a muffle furnace on 550^{o}C until constant mass was reached. 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 mass spectra of the compounds detected were then compared with the mass spectra pattern from the NIST library version 0.8L (National Institute of Standards and Technology) in a computer database. The volatile compound component data was then further analyzed with the Automatic Mass Spectral Deconvolution and Identification System (AMDIS) software [22]. Data resulting from the analysis of volatile compounds was discussed descriptively based on the identification and intensity of the semi-quantification of the compounds detected in the samples tested [9]. Average data obtained from the proximate analysis of all samples was calculated and discussed descriptively.

3. RESULTS AND DISCUSSION

3.1 Volatile Component

The analysis results showed that flavor powder sample had 93 types of volatile compounds consisting from several compound groups such as hydrocarbons, aldehydes, ketones, alcohols, organic acids, esters, and others compound group. The identification result is presented in Table 1 and the volatile compound analysis had succesfully detected 54 compounds which derived from hydrocarbons group (includes aliphatic and cyclic hydrocarbons), 20 compounds from alcohol group, 12 compounds form aldehyde group 1 compound form ketone, 3 compounds from esters, 2 compounds derived from organic acid group, and 1 compound was classified in other compound group (includes nitrogenous and sulphurous group of compound). The group which dominated the most was hydrocarbons with a total of 54 compounds while the compound which had the highest proportion (%) compared to other compounds was pentanal (8.992%) from aldehyds group.

Table 1. Volatile compounds detected in spray-dried white shrimp immersed-water flavor powder

Groups	Retention Time	Compounds	Area	Proportion (%)
Hydrocarbons	12.6335	Nonane, 3,7-dimethyl-	192470402	4.695
	12.9189	Decane, 3,6-dimethyl-	162249684	3.958
	12.5146	Undecane, 5-methyl-	121352148	2.960
	11.7832	Decane, 3,7-dimethyl-	98872324	2.412
	11.5811	Octane, 2,3,3-trimethyl-	98046768	2.392
	17.5211	1-Tridecene	73165102	1.785
	12.0865	Tridecane, 4-methyl-	72194130	1.761
	20.4881	Undecane, 2-methyl-	64517766	1.574
	11.9854	Undecane, 4-methyl-	64085027	1.563
	13.9713	Undecane, 2-methyl-	59858771	1.460
	11.4086	Tridecane, 6-methyl-	56624298	1.381
	14.1557	Undecane, 3-methyl-	56251595	1.372
	22.6644	1-Pentadecene	54226116	1.323
	21.5941	Cyclotetradecane	53733477	1.311
	15.1665	Dodecane	48348762	1.179

Groups	Retention Time	Compounds	Area	Proportion (%)
	13.4243	Undecane, 5,7-dimethyl-	47273611	1.153
	11.8962	Octane, 2,7-dimethyl-	47150339	1.150
	16.1595	Cyclopentane, hexyl-	44994533	1.098
	17.8838	Nonane, 5-methyl-5-propyl-	42642538	1.040
	14.5778	1H-Indene, 1-methylene-	36392953	0.888
	24.0914	Cyclopentadecane	33844602	0.826
	19.186	2,6-Octadiene, 2,6-dimethyl-	31558623	0.770
	27.0227	1,3-di-iso-propylnaphthalene	29076807	0.709
	18.2881	5-Tetradecene, (E)-	29050150	0.709
	20.3633	Tetradecane	28038989	0.684
	25.2865	Dodecane, 2,6,10-trimethyl-	27214751	0.664
	26.4698	Cyclopentane, undecyl-	27054428	0.660
	19.5011	Cyclododecane	26921235	0.657
	20.1492	2-Tetradecene, (E)-	25789393	0.629
	17.2714	7-Tetradecene	24792371	0.605
	15.6243	6-Tridecene, (Z)-	23101423	0.564
	19.5962	1-Undecene	22720126	0.554
	17.7411	Tridecane	21722748	0.530
	17.5984	7-Tetradecene, (E)-	21457442	0.523
	23.2352	Butylated Hydroxytoluene	20589516	0.502
	11.0935	Dodecane, 2,7,10-trimethyl-	19565585	0.477
	28.1168	1,7-di-iso-propylnaphthalene	16908628	0.412
	27.7601	Naphthalene, 1,2,3-trimethyl-4-propenyl-, (E)-	15026030	0.367
	18.0503	Decane, 3,8-dimethyl-	14893139	0.363
	27.5698	Dodecane, 2,6,11-trimethyl-	14739581	0.360
	10.7665	Undecane, 3,6-dimethyl-	14532312	0.355
	18.1454	6-Tetradecene, (E)-	13823200	0.337
	26.2201	3-Octadecene, (E)-	13523416	0.330
	15.7314	5-Tridecene, (E)-	12386164	0.302
	28.0514	1,4-di-iso-propylnaphthalene	11170742	0.273
	17.1584	3-Tetradecene, (E)-	10209430	0.249
	15.4757	Nonane, 4,5-dimethyl-	9927630	0.242
	26.3865	Pentadecane	9859544	0.241
	30.846	1-Octadecene	6350627	0.155
	29.6628	Octadecane	6013715	0.147
	19.073	1-Pentadecene	5211830	0.127
	29.2109	3-Octadecene, (E)-	3052384	0.074
	30.7449	3,3-Diethylpentadecane	2540563	0.062
	31.6071	Nonadecane	2079352	0.051
Alcohols	10.1243	1-Hexanol, 2-ethyl-	347107866	8.468
	2.9951	Cyclobutanol	83155600	2.029
	8.6913	1-Octen-3-ol	44513507	1.086
	11.3492	1-Octanol, 2-butyl-	42515421	1.037
	18.6925	1-Dodecanol, 2-methyl-, (S)-	26612871	0.649
	16.76	1-Dodecanol, 2-hexyl-	26043116	0.635
	21.3206	1-Dodecanol, 2-octyl-	23480585	0.573
	23.3719	1-Hexadecanol, 2-methyl-	22852265	0.557
	4.2318	2,3-Butanediol	21348718	0.521
	22.1233	1-Octanol, 2-butyl-	21199419	0.517
	25.7622	n-Tetracosanol-1	14324985	0.349

Groups	Retention Time	Compounds	Area	Proportion (%)
	10.6713	3,5-Octadien-2-ol	13031866	0.318
	17.3487	1-Decanol, 2-octyl-	11622001	0.284
	23.6752	1-Decanol, 2-hexyl-	10922009	0.266
	24.4006	2-Hexyl-1-octanol	10559274	0.258
	25.8514	2-Ethyl-1-dodecanol	10056890	0.245
	8.5308	1-Butanol, 2-methyl-	6121962	0.149
	6.3724	1,3-Butanediol	4500745	0.110
	7.7162	2-Heptanol, 6-methyl-	2541292	0.062
	31.3574	2-Ethyl-1-dodecanol	2099501	0.051
Aldehyde	2.2399	Pentanal	368608685	8.992
	12.2708	Nonanal	232923594	5.682
	1.7821	Butanal, 3-methyl-	175379710	4.278
	15.3389	Decanal	42019591	1.025
	11.2362	2-Octenal, (E)-	31767177	0.775
	26.6838	2-Nonenal, 2-pentyl-	25656428	0.626
	5.641	2-Hexenal, (E)-	17168103	0.419
	10.5286	Benzeneacetaldehyde	15590558	0.380
	9.5832	2,4-Heptadienal, (E,E)-	13724763	0.335
	6.5865	Heptanal	8381905	0.204
	8.0492	2-Heptenal, (Z)-	6972242	0.170
	8.1743	Benzaldehyde	2133501	0.052
Ketones	8.4297	Acetophenone	6491727	0.158
Esters	10.927	Oxalic acid, isobutyl nonyl ester	32120515	0.784
	18.9838	Sulfurous acid, hexyl tridecyl ester	29252186	0.714
	31.0482	Dibutyl phthalate	3748633	0.091
Organic acid	3.7027	Acetic acid	10299729	0.251
	5.8432	Butanoic acid, 3-hydroxy-	10011949	0.244
Others	9.2324	Pyrazine, trimethyl-	233219630	5.689

The compounds which identified and had highest proportion from hydrocarbons group was 3,7-dimethyl- nonane (4,695%). Most hydrocarbon compounds would show a relatively high (aroma) threshold. However, if they are available in such high amounts then they could also contribute to overall flavor of a products [8]. The 3,7-dimethyl-nonane compound is a hydrocarbon compound which was categorized in the alkane group. According to [23], alkane compounds can originate from decarboxylation and separation of fatty acids carbon chains. Some cyclic hydrocarbons identified in fish are the result of secondary reactions from thermal oxidation (heating) of carotenoids and other unsaturated fats [8].

Identified compounds which had the highest proportion from the alcohol group was 2-ethyl-1-Hexanol (8.468%). This compound has raw fish and green aroma characteristic with strong intensity [24]. [25], state that alcohol compounds are generally formed from the decomposition of fatty acids. Alcohol generally contributes to the fruity, floral and grassy aroma in fish [8]. In the study performed by [26], the 2-ethyl-1-Hexanol was also found as volatile component in fresh and steamed catfish samples. Alcohol group compounds are generally produced through the oxidation of lipids and fatty acids as well as amino acid degradation during the processing stage [27].

The compounds found in the aldehyde group which have the highest proportion was pentanal (8.992%). Pentanal compounds are classified as alkyl aldehydes which described as having almond, malt and pungent aroma charachteristics [28]. In the study carried out by [4], pentanal was also identified as volatile component in fresh common carp and its steaming product. The detected aldehyde compounds could have originated from double carbon bonds of unsaturated fatty acids or oxidized saturated fatty acids [8]. Aldehydes could provide a significant aroma impact to food ingredients (pleasant or rancid aromas). The aldehydes are commonly having lower aroma threshold values compared with alcohol, hence it can be concluded that even if the least amount of aldehydes which contained in a product then they are possibly could replace the aroma characteristics of other substances [17]. Aldehyde compounds have a variety of distinctive aromas such as green plant, dark chocolate, sweet floral apple-like, melon-like, nutty, malty, fatty, grassy, and fruity and are commonly found in various shells and fresh fish with varying concentrations [29].

There was only one compound detected in the samples which originated from ketone group and it was acetophenone (0.158%).

Acetophenone commonly known to has sweet, almond, pungent and oranges aroma characteristics [30]. Ketone compounds are generally quite reactive, hence, sensory evaluations have shown that ketones have properties which contribute in flavor development and are involved in the formation of aromatic reaction products with other food ingredients [31]. According to [9], most ketones are known to exist in volatile components of many fisheries commodities, thus ketones are likely to be produced from the oxidation of lipids (especially unsaturated fatty acids) during heating. In addition, another possible pathways of ketone compounds formation are thermal degradation, amino acid degradation and Maillard's reaction.

The highest abundance ester compound detected in the flavor powder sample was isobutyl nonyl ester- oxalic acid (0.784%). Esters in fishery commodity are generally derived from the acid esterification process with alcohol which was previously formed from the results of lipid metabolism [17], [23]. The isobutyl nonyl ester- oxalic acid compound can also be found in the volatile components of freshwater fish *Osphronemus gouramy* [17].

Volatile component analysis had succeeded in identifying acetid acid (0.251%) as one of the highest abundance organic acid in white shrimp immersed water flavor powder sample. The compound has pungent and vinegar-like aroma characteristics [30]. Acetid acid compounds can also be found in [28] study regarding the volatile component of Malaysian fish sauce and [32] study concerning changes in volatile compounds of yellowtail fish during storage.

Trimethyl- pyrazine (5.689%) was detected in flavor powder sample and classified in other group of compound. This compound is commonly discovered in cereals and cereal products. In addition, it is also detected in many foodstuffs e. g. asparagus, baked potato, wheat bread, coffee, black tea, roasted peanut, soybean and could act as a flavouring ingredient [33]. In general, pyrazine compounds group is one type of natural product which can be discovered in plants, animals, insects, marine organisms and microorganisms. The principal role of this compound in living organisms is as raw foods flavor [34]. According to [35], the aroma component of pyrazine in an ingredient can be formed through a roasting process.

3.2 Proximate Analysis

Proximate analysis series was carried out to determine the amount of overall chemical composition specifically nutrient related parameters in a food product. [36], stated that shrimp is considered as food ingredient which is very easy to deteriorate mainly due to its high moisture and protein content. The results of proximate analysis of flavor powder sample can be seen in Table 2.

Table 2. Proximate analysis results of spray-dried white shrimp immersed-water flavor powder

Parameters	%
Moisture	4.56
Ash	1.45
Lipid	0.07
Protein	5.43

Water is a major component in food that can affect the durability of a commodity. Water or in proximate analysis expressed as moisture content is the percentage of water content of an ingredient. High water content can cause the proliferation of bacteria, mold/fungi and yeast [37]. Moisture content can also be defined as amount of water which evaporate from the material through heating process on a certain temperature that does not exceed the water boiling point. The resulting value from the moisture content analysis could also determines the food freshness degree, since water is one of the important parameters which could affect the appearance, texture and flavor of the food. The flavor powder sample had 4.56% moisture content based on the results and its value in general, could be influenced by species, freshness level, growth phase and differences in environmental conditions. Moisture content can also be affected by the nature of maltodextrin which was used as a filler.

Ash content is the residues or remains from the combustion process of food material which is heated entirely in the furnace. Residues of combustion is the amount of unburnt minerals from non-evaporating substances. According [38], ash content determination in food sample has a close relationship with its mineral content and it is a parameter of the nutritional value of a product material produced by inorganic components contained in fish due to minerals importance in human general health (body regulators and growth importance). The ash content in the flavor powder sample was 1.45%. The ash content of foodstuffs and their composition depends on the type of material and how they are ignited. This could be affected by variation of mineral content in the sample studied. Ash content in the material could shows the raw estimation of minerals amount in the studied sample and shows the presence of inorganic substances which are the result of combustion of raw materials organic substances. The value in a fishery commodity could be influenced by species, growth phase, seasons, captured period and other environmental factors [9].

Based on the analysis, the lipid content of the flavor powder sample was 0.07%. Lipid is a more effective source of energy compared to carbohydrates and protein. According to [39], lipid content is related to flavor components due to its heat-resistant properties, hence, the lipid which undergoes the cooking process will melt and even evaporate (volatile) into other components such as volatile flavor component. [40], states that the lipid content of flavor powder could decreased as the amount of filler added increased. [41], stated that variation in lipid content could be influenced by the shrimp species and its growth phase when the shrimp is harvested.

Shrimp in the molting phase have a higher lipid content [42]. Other factors which could affect lipid content are the environment and the feed consumed by the shrimp. Lipids, especially triglycerides, have the role of providing the body's energy reserves, providing essential fatty acids and protecting body organs.

Protein is a source for amino acids which contains various elements of carbon, hydrogen, oxygen, and nitrogen. The results obtained in the flavor powder sample was 5.43%. According to Sebranek (2009) [43], decreased moisture content could render the protein content in the material to increase. The use of heat in food processing can reduce the percentage of moisture content which causes the percentage of protein content to increase. Increased protein levels also occur in studies of the chemical composition and vitamins of *Hapiosquilla raphidea* due to boiling process which studied by [39].

Conclusion

Further research is still needed to determine the key component which contribute to the white shrimp immersed water flavor powder overal aroma characteristics. Based on the research that has been done, several conclusions can be drawn. As much as 93 compounds have been succesfully detected from the flavor powder sample. The detected groups of volatile compounds were mostly derived from the hydcrocarbon alcohol, aldehyde, ketone, ester, organic acid and others group. Hydrocarbon dominated the volatile compounds with 54 compounds were identified from this sample. However, the most abundance volatile compound originated from aldehyde group which was pentanal (8.992%).

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