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VOLATILE COMPOUND COMPOSITION OF SPRAY DRY FLAVOR POWDER FROM WHITE SHRIMP BOILED WATER BROTH

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ABSTRACT
The research aims to identify volatile compounds and proximate flavor powder prepared from white shrimp boiled water broth. The white shrimp were collected from the local fish landing site in Karangsong, Indramayu, West Java. This research uses an experimental method by testing the composition of volatile flavor compounds and then undertaking proximate testing. The method was to identify using Gas Chromatography /Mass Spectrometry (GC/MS) on flavor powder sample with Soil Phase Micro extraction (SPME) (80°C, during 30 minutes) to extract volatile compounds. The result of analysis detected 96 volatile compounds. Volatile compounds detected were mostly derived from hydrocarbons, alcohols, aldehydes, ketones, esters, and other compounds. The highest proportion of volatile compounds detected in aldehydes groups was butanal, 3-methyl- (14.881%). The proximate analysis results showed that flavor powder from white shrimp boiled water broth sample had a moisture content of 6.19%, ash content of 3.03%, protein content of 16.45%, and lipid content of 0.20%.

INTRODUCTION

Shrimp is export commodities which largely contributes to Indonesian foreign trade. Official data from the [1] shrimp occupy the first place in the aquaculture commodities with the highest production value in 2017 increased by 58,135,246 tons. Among the exported shrimp, one of the most popular shellfish and is mainly consumed in Indonesia is white shrimp (*Litopenaeus vannameï*).

White shrimp is one of the most popular for raw material processed products because it has great tastes and contains various nutrients for human health. White shrimp nutrients composition such as essential amino acids, lipids, macro and micro minerals [2]. Processed white shrimp, with high temperature processing, low temperature processing, or fermentation in some products can change the texture and produce special attribute such as flavor characteristics of a commodity [3].

Flavor is a very important component of the eating quality of meat and determining those factors during the production and processing of meat which influence flavor quality, and there has been much research aimed at understanding the chemistry of meat flavor [4]. Flavor is a sensation that appears from the flavoring agents or derived from natural composition. Flavor compounds could be affected by various compounds contained in a product by volatile or non-volatile chemical compounds [5]; [6]; [3]. Volatile compounds are affecting aroma characteristics of a product. In general, meat flavor compounds including fisheries commodities are mostly formed due to the cooking process which involves heat to reduce water content in food such as drying.

Drying is a process by which water is removed from the food, by vaporization or sublimation, thus reducing the water available for degradation reactions of chemical, enzymatic or microbial nature [7]. Drying process can be naturally or artificially. The artificial drying process by using various drying instruments such as drum dryers, tunnel dryers, oven, various dehydrators and spray dryers. The spray dryer is more complicated. because effectively convert high moisture input into various forms of dry powders.

Flavor powder is a form of dried and solid flavoring agent. Flavor powder obtained through absorption process by means of dry material carrier. Flavor powder can easily be found in the market, many in the form of seasoning powder extracted from meat compounds and other herbs. Flavor powder is important because it could be increase flavor of products and have a unique aroma characteristics. Information of volatile compounds specially from flavor powder is important mainly to research the product aroma characteristics, contribute in providing flavor and database for further flavor research. The composition of volatile compounds detected in fishery products is mostly derived from hydrocarbons, alcohols, aldehydes, ketones, esters, and other compounds. This matter is in accordance with research about volatile compounds of roasted shrimp [8]; volatile compounds in flavor concentrates from crayfish processing waste [9]; volatile compounds of raw and smoked black bream (*Brama raii*) and rainbow trout (*Oncorhynchus mykiss*) studied by means of solid phase micro extraction and gas chromatography/mass spectrometry [10]; and influence of re-cooking on volatil and non-volatil compounds found in silver carp (*Hypopthalmihthys molitrix*) [6]. the main objective of this research was to identify the volatile compounds composition of flavor powder from white shrimp boiled water broth

MATERIALS AND METHODS

2.1 Tools and Materials

The tools used include Gas Chromatography (Agilent Technologies 7890A GC System) / Mass Spectrometry (Agilent Technologies 5975C Inert XL EI CI/MSD), spray dry, waterbath, analytic balance, jar, ziplock plastic bag, streamer pan, aluminium foil, cool box, thermometer, labeled paper, knife, gas stove, cling wrap, vial kit bottle. The material used include white shrimp, cured ice, maltodextrin, aquades.

2.2 Research Location

White shrimp meat sample was taken from local fish landing site in Karangsong, Indramayu, West Java. Sample preparation was carried out in the Fisheries Product Processing Laboratory, Faculty of Fisheries and Marine Sciences, Universitas Padjadjaran, dried sample were carried out in Central Laboratory, Universitas Padjadjaran, volatile compounds analysis were carried out in Flavor Laboratory Indonesian Centre for Rice Research, Sukamandi, Subang and proximate analysis were carried out in Inter-University Centre Laboratory, Bogor Agriculture Institute.

2.3 Research Methods

The method used was experimental method to identify volatile compounds composition and proximate flavor powder from white shrimp boiled water broth. The final research will be discussed in a comparative descriptive based on the identification and semi quantification intensity of the compounds detected from the analysis sample.

2.3.1 Sample Preparation

1) As much as 5 kg white shrimp samples 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.

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3) The shrimp meat portions were subsequently washed and boiled (65°C for 90 minutes in aquadest with 1:2 ratio.
4) Shrimp 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) Shrimp 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 bring developed into powder form.
6) After the drying process was 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 sample during transportation to the analysis laboratory.
7) The tight packed sample were then transported to Flavor Laboratory for volatile component analysis and Inter-University Centre Laboratory for proximate analysis.

2.3.2 Proximates analysis

Sample was then analyzed for its moisture, ash, protein and lipid content. The proximate analysis according to [11].

2.3.3 Volatile compounds analysis

Volatile compounds was analyzed according modification of [12] procedure. The analysis was carried out using water bath for sample 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 compounds. 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°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°C (hold 5 minutes), temperature escalation as much as 5°C/minutes, final device temperatures 250°C (hold 5 minutes) with an overall running time 36 minutes.

2.3.4 Data Analysis

Sample volatile compounds 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.

RESULT AND DISCUSSION

3.1 Volatile compounds

Volatile compounds analysis result showed as much as 96 volatile compounds were succesfully detected from the white shrimp flavor powder. The compounds detected were then categorized into several major compounds groups such as hydrocarbons, aldehydes, ketones, alcohols, esters, and others compounds.

Table 1. Volatile compounds of white shrimp flavor powder

Groups	RT (min)	Compounds	Area	Proportion
Hydrocarbons	12.6278	Nonane, 3,7-dimethyl-	111272050	2.833
	12.9073	Decane, 3,6-dimethyl-	72367506	1.843
	12.5089	Undecane, 5-methyl-	59980402	1.527
	11.4029	Undecane, 3-methyl-	35729932	0.910
	12.0927	Tridecane, 4-methyl	35703689	0.909
	22.6587	1-Pentadecene	32312140	0.823
	11.9856	Undecane, 4-methyl-	30909324	0.787
	26.47	Cyclopentane, undecyl-	30287494	0.771
	25.2808	Dodecane, 2,6,10-trimethyl-	29199937	0.743

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	19.1922	2,6-Octadiene, 2,6-dimethyl-	27763712	0.707
	27.7662	Naphthalene, 1,2,3-trimethyl-4-propenyl-, (E)-	26820585	0.683
	14.1559	Undecane, 3-methyl-	26664212	0.679
	24.0916	Cyclopentadecane	25449433	0.648
	27.0289	1,3-di-iso-propylnaphthalene	24316538	0.619
	17.5154	1-Tridecene	23685415	0.603
	21.5884	Cyclotetradecane	22592101	0.575
	16.1597	Cyclopentane, hexyl-	21672085	0.552
	13.4305	Undecane, 5,7-dimethyl-	20168624	0.514
	15.3392	Nonane, 4,5-dimethyl-	19111458	0.487
	14.5781	1H-Indene, 1-methylene-	17946511	0.457
	27.57	Dodecane, 2,6,11-trimethyl-	17082296	0.435
	20.3576	Tetradecane	15854829	0.404
	28.117	1,7-di-iso-propylnaphthalene	15846031	0.403
	23.2295	Butylated Hydroxytoluene	14408094	0.367
	26.2203	3-Octadecene, (E)-	13711453	0.349
	28.2241	1-Acetyl-4,6,8-trimethylazulene	12214697	0.311
	28.0516	1,4-di-iso-propylnaphthalene	10394797	0.265
	26.3927	Pentadecane	10142095	0.258
	19.5965	1-Undecene	10092148	0.257
	17.9613	Cyclotetradecane	9784591	0.249
	11.0878	Dodecane, 2,7,10-trimethyl-	9399048	0.239
	18.2884	5-Tetradecene, (E)-	9395460	0.239
	17.7354	Tridecane	9145357	0.233
	30.8463	1-Octadecene	8351337	0.213
	17.5986	7-Tetradecene, (E)-	8340219	0.212
	10.7667	Undecane, 3,6-dimethyl-	8114863	0.207
	29.663	Octadecane	8074439	0.206
	20.1495	2-Tetradecene, (E)-	7558155	0.192
	17.884	Nonane, 5-methyl-5-propyl-	7555188	0.192
	17.1943	3-Tetradecene, (E)-	7504958	0.191
	15.7316	5-Tridecene, (E)-	7433302	0.189
	14.4235	3,5,24-Trimethyltetracontane	7027205	0.179
	15.9338	Cycloheptane, methyl-	6827788	0.174
	17.2716	7-Tetradecene	6693284	0.170
	15.6246	6-Tridecene, (Z)-	5721745	0.146
	18.0446	Decane, 3,8-dimethyl-	4821127	0.123
	29.2111	3-Octadecene, (E)-	4235326	0.108
	30.7452	3,3-Diethylpentadecane	3205445	0.082
	31.6073	Nonadecane	2312469	0.059
	14.9824	Dodecane	1462412	0.037

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Alcohols	4.2797	2,3-Butanediol	435460264	11.088
	3.0132	Cyclobutanol	131834954	3.357
	10.1008	1-Hexanol, 2-ethyl-	74713728	1.902
	10.3089	Benzyl alcohol	21397265	0.545
	23.3603	1-Hexadecanol, 2-methyl-	16534593	0.421
	25.7684	n-Tetracosanol-1	14413710	0.367
	8.7629	1-Octen-3-ol	14143377	0.360
	22.1235	1-Octanol, 2-butyl-	11964909	0.305
	16.7543	1-Dodecanol, 2-hexyl-	11242510	0.286
	18.6868	1-Dodecanol, 2-methyl-, (S)-	9674300	0.246
	21.3149	1-Dodecanol, 2-octyl-	9309745	0.237
	25.8576	2-Ethyl-1-dodecanol	8417296	0.214
	10.6656	3,5-Octadien-2-ol	7094141	0.181
	6.331	1,3-Butanediol	6678832	0.170
	23.6695	1-Decanol, 2-hexyl-	6280792	0.160
	17.3489	1-Decanol, 2-octyl-	4334281	0.110
	31.3636	2-Ethyl-1-dodecanol	2946548	0.075
Aldehydes	1.7645	Butanal, 3-methyl-	584456857	14.881
	2.2342	Pentanal	511346048	13.020
	12.2651	Nonanal	121091205	3.083
	8.1743	Benzaldehyde	24345112	0.620
	5.6235	2-Hexenal, (E)-	22552839	0.574
	15.1667	Decanal	22235536	0.566
	11.2305	2-Octenal, (E)-	16939609	0.431
	10.5229	Benzeneacetaldehyde	8346266	0.213
	6.5867	Heptanal	4219189	0.107
	9.8154	2,4-Heptadienal, (E,E)-	3869232	0.099
Ketones	8.8878	Cyclobutanone, 2,3,3-trimethyl-	13328641	0.339
Esters	25.2095	Sulfurous acid, 2-pentyl undecyl ester	47569728	1.211
	26.03	Carbonic acid, decyl pentadecyl ester	20676287	0.526
	22.7716	Carbonic acid, eicosyl prop-1-en-2-yl ester	13266417	0.338
	18.9781	Sulfurous acid, hexyl tridecyl ester	9762638	0.249
	27.6711	Carbonic acid, eicosyl vinyl ester	8998345	0.229
	31.0484	Dibutyl phthalate	6234534	0.159
	30.4479	Carbonic acid, decyl tetradecyl ester	6225528	0.159
	26.916	Carbonic acid, decyl hexadecyl ester	5840809	0.149
	31.4825	Oxalic acid, allyl dodecyl ester	3108989	0.079
Others	9.265	Pyrazine, trimethyl-	212734527	5.417

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	11.5397	Pyrazine, 3-ethyl-2,5-dimethyl-	178120006	4.535
	11.7597	Pyrazine, tetramethyl-	133246301	3.393
	13.9002	2,3,5-Trimethyl-6-ethylpyrazine	118923315	3.028
	6.8424	Pyrazine, 2,5-dimethyl-	42741063	1.088
	7.0326	Pyrazine, 2,6-dimethyl-	27465986	0.699
	9.9105	Acetylpyrazine	6176537	0.157
	9.7024	Pyrazine, 2-ethenyl-6-methyl-	5341885	0.136
	18.2051	Pyrazine, 2,5-dimethyl-3-propyl-	5217378	0.133
Total			3927485358	100

Description : RT : *Retention Time* (minutes)

3.1.1 Hydrocarbons

The result compounds analysis with highest proportion in hydrocarbons groups is nonane, 3,7-dimethyl- (2.833%). Nonane is an organic compounds included in the alkanes groups. Research by [13] alkane generally did not have a significant contribution to food aroma. This can be observed from the research of [10] regarding bream fish volatile compounds which showed that hydrocarbons groups did not have a significant on flavor. Hydrocarbons are compounds from carbon and hydrogen atoms bound by covalent bonds. Alkane groups hydrocarbons are produced from decarboxylation and separation of carbon chains from higher fatty acids.

3.1.2 Alcohols

The result compounds analysis with highest proportion in the alcohols groups is 2.3-butanediol (11.088%). The 2,3-Butanediol is categorized as aliphatic alcohol groups which has buttery and creamy odor types. Volatile compounds in alcohol groups generally provide a minimal character in food flavor because they are commonly known to have a high threshold except the alcohol groups were attendance at high concentrations or not saturated condition [14]. The alcohol component generally produces sweet, fruity, alcoholic, balsamic, and green odors depending on the molecular structure [15].

3.1.3 Aldehydes

The result compounds analysis with highest proportion in the aldehydes groups is butanal, 3-methyl-(14.881%). Followed by pentanal as the second highest proportion compounds (13.020%) in this groups. Butanal compounds, 3-methyl- commonly recognized to has chocolate, caramel, green and nutty odor types [16] while pentanal compounds have almond, malt and pungent odor types [17]. [6] states that the detected aldehyde groups can derived from the double carbon bonds of unsaturated fatty acids or oxidized saturated fatty acids. Consequently, that most of the aldehydes are considered derive from lipid oxidation products [18].

3.1.4 Ketones

The result compounds analysis with highest proportion in the ketones groups is cyclobutanone 2,3,3-trimethyl- (0.339%). Ketones may be a product of microbial oxidation, amino degradation or lipid oxidation. The ketone compounds produced could be derived from the thermal oxidation process or degradation of unsaturated fatty acids, amino acid degradation or oxidation of microorganisms [6]. The resulting component mentioned was responsible for producing an aroma similiar to fish oil oxidation odor [19].

3.1.5 Esters

The result compounds analysis with highest proportion in the esters groups is sulfurous acid, 2-pentyl undecyl ester (1.211%). Esters compounds were possibly formed from lipid metabolism by means of acid esterification with alcohol [10]. Esters commonly recognized to have a delightful aroma, they can be applied as flavoring agents [20].

3.1.6 Other compounds

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Other compounds which usually contain nitrogenous and sulfurous compounds were categorized in others groups. The compounds which has the highest proportion in this groups is pyrazine, trimethyl- (5.417%) compounds. Pyrazine, trimethyl- or trimethyl pyrazine is a volatile compounds which has a nitrogen element. Naturally occured pyrazine compounds are important compounds which also contribute to raw and processed foods taste [21]. The compounds is known to have a roasty and earthy aroma [22]. Pirazine compounds were also identified in research regarding volatile compounds of dried salted fish.

3.2 Proximate analysis

Flavor powder from white shrimp boiled water broth was analyzed for moisture, ash, lipid and protein content [11] and it can be seen in Figure 1.

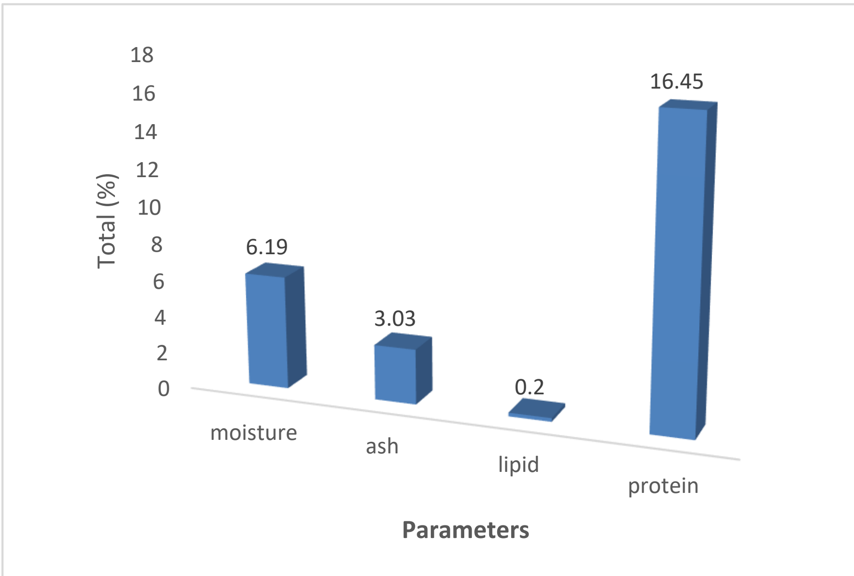

Figure 1. Proximate analysis results

3.2.1 Moisture content

Water is a major constituent in food composition which can affect it's durability. Moisture content is the percentage of water content in the material which evaporate from the heat process with certain temperature and does not exceed the water boiling point. The sample moisture content has a value of 6.19%. The analysis result indicates that the moisture exceeds the limit [23] for flavor powder (chicken based), which has a maximum value of 4.0%. The moisture content of white shrimp flavor powder could be influenced some factors such as maltodextrin as a filler. [24] Maltodextrin has hygroscopic properties which able to absorb water so increased moisture content along with the increasing proportion quantity of maltodextrin added.

3.2.2 Ash content

Ash content is the remaining residue left from the burning of food sample which are completely burned in the furnace. The remains of burning process are the quantity of unburnt minerals from non-evaporating substances. Determination of ash content has a correlation with the mineral content contained in a material. The sample's ash content showed a value of 3.03%. Ash content in fish depends on the type of fish meat composition. Lower ash content is found in white meat fish if it is compared to red meat fish. This is because the red meat contains numerous minerals which are carried by myoglobin and stored in meat [25].

3.2.3 Lipid content

Lipid is an organic compounds that exists in nature and could not dissolved in water, but lipid could dissolve in non-polar organic solvents and is a major component of adipose tissue. Lipid is more effective source of energy compared to carbohydrates and protein [26]. The lipid content of the white shrimp flavor powder showed a value of 0.20%. Lipid content value according to [23] is a minimum of 2%. Levels of lipid in flavor powder will decrease with increasing quantity of filler added. Lipid content and water content have a negative correlation, if the lipid content in a material is high ,the water content would have a low value. Variation in lipid content can be influenced by the type of shrimp and the life phase of shrimp when harvested. Shrimp in the molting phase have a higher lipid content. Other factors affecting the lipid content are the environment and feed consumed

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by the shrimp. Lipids specially triglycerides, have the role in providing the body's energy reserves, essential fatty acids and protecting the body's organs. Lipid are important function in volatile compounds. Hydrocarbons, alcohols, aldehydes, ketones, and esters are the product of lipid decomposition process through the decarboxylation process, also the decomposition of lipid carbon chain and oxidation. According to [27], lipid content is related to flavor compounds. Lipid characteristics which has heat-resistant properties. Lipid from the cooking process will melt and even evaporate (volatile) forming various compounds such as volatile flavor compounds.

3.2.1 Protein content

Protein is a source for amino acids which contain carbon, hydrogen, oxygen, and nitrogen elements [26]. Protein content according to [23] is minimal 6%. The result for sample protein content was 16.45%. This result showed that the flavor powder from white shrimp meat is high because of some factors such as heating from boiling processed. Boiling process will dissolve the nutrition component such as protein because meat from white shrimp is immersed in water. The meat from white shrimp will change chemically and physically because the strength of protein at moisture is lose . The application of heat in this research was able to reduce the sample water content which causes the increasing of protein content value. Increased protein levels also occured in research by [27] regarding chemical composition and vitamins content of *Ronggeng* shrimp (*Hapiosquilla raphidea*) as affected by boiling process.

Conclusion

Based on the research results on volatile compounds identification of white shrimp meat flavor powder, several matters could be concluded. Most of the volatile flavor compounds detected from the sample were from hydrocarbons, alcohols, aldehydes, ketones, esters, and others (sulfurous and nitrogenous compound) groupss of compound. As much as 96 volatile compounds from variuos compound groups have been succesfully identified from the white shrimp flavor powder. Compound which has the highest proportion originated from aldehydes groups, 3-methyl-butanal (14.881%). The proximate analysis results showed that white shrimp flavor powder had a moisture content of 6.19%, ash content of 3.03%, protein content of 16.45%, and lipid content of 0.20%. This research can still be improved by conducting various other analysis parameters, such as analysis of taste components and amino acid profiles. Therefore, the information obtained regarding flavor powder will be more comprehensive.

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