ANALYSIS OF FORMALDEHYDE PRESERVATIVES IN WET ANCHOVY (Stolephorus Sp.) FROM TRADITIONAL MARKETS IN MAKASSAR CITY, SOUTH SULAWESI

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Abstrak. Pengawet makanan merupakan bahan sangat penting dalam peningkatan kualitas dan produksi makanan olahan yang dapat memperpanjang umur simpan makanan tersebut. Namun demikian terdapat berbagai macam pengawet dan diantaranya pengawet yang tidak diperbolehkan seperti formaldehida. Penelitian ini bertujuan untuk mengetahui kandungan bahan pengawet formaldehida pada ikan teri (stolephorus sp.) basah dari pasar tradisional Kota Makassar Sulawesi Selatan. Bahan pengawet formaldehida diidentifikasi dengan melalui uji kualitatif dan kuantitatif. Uji kualitatif formalin menggunakan pereaksi Schryver dengan menggunakan metode analisis Spektrofotometer UV-Vis. Hasil analisis kualitatif menunjukkan bahwa semua sampel ikan teri basah mengandung formaldehida, dengan nilai konsentrasi Sampel dari Pasar Daya A; Pasar Daya B; Pasar Daya C berturut-turut 0,2702ppm; 0,1307ppm; 0,0871ppm, Pasar Antang A; Pasar Antang B; Pasar Aantang C berturut-turut 0,1612ppm; 0,2223ppm; 0,1525ppm, Pasar Pa'baengbaeng A; Pasar Pa'baeng-baeng B; Pasar Pa'baeng-baeng C berturut-turut 0,1918ppm; 0,0087ppm; 0,2877ppm, Pasar Terong A; Pasar Terong B; Pasar Terong C; berturut-turut 0,1394ppm; 0,1961ppm; 0,0043. Kadar formaldehida terendah terdapat pada Pasar Terong C dengan kadar 0.0087ppm dan kadar formaldehida tertinggi terdapat pada Pasar Pa'baeng-baeng C dengan kadar 0,2877ppm.

Kata Kunci: Formaldehida, Pengawet, Pereaksi Schryver, Spektrofotometer Uv-Vis

Abstract. Food preservatives are very important ingredients in improving the quality and production of processed foods. However there are various preservatives and such preservatives such as formaldehide are not allowed. This study aims to determine the content of preservatives in fresh anchovy (*stolephorus sp.*) from the traditional market of Makassar City South Sulawesi. Formaldehyde preservatives is identified by qualitative and quantitative test, qualitative formaldehyde was tested using Schryver reagent using UV-Vis Spectrophotometer analysis method. The result of qualitative analysis showed that all samples contained formaldehyde, with the concentration value of 0.2702ppm; 0.1307ppm; 0.0871ppm in Daya Market A; Daya Market B; Daya Market C samples, respectively. The content of 0.1612ppm; 0.2223ppm; 0,1525ppm in Antang Market A; Antang Market B; Antang Market C samples, respectively. The content of 0,1918ppm;0,0087ppm; 0.2877ppm in Terong Market A; Terong Market B; Terong Market C, samples, respectively and the content of 0.1394ppm; 0.1961ppm; 0,0043, in Pa'baeng-baeng Market A; Pa'baeng-baeng Market B; Pa'baeng-baeng Market C samples, respectively. The lowest formaldehyde level was found in Terong Market C with level of 0.0087ppm and the highest formaldehyde level was in Pa'baeng-baeng Market C with the level of 0.2877ppm.

Keywords : Formaldehyde, Preservatives, Schryver Reagent, UV-Vis Spectrophotometer

INTRODUCTION

Fresh anchovy is one of the fish that has a high protein content, which is 68.7 g/100g of fresh dried anchovy and 42 g/100g of salted dried anchovy. Anchovy protein contains a number of essential amino acids, namely amino acids that cannot be formed in the body, but must come from food. The most prominent essential amino acids in anchovy are isoleucine, leucine, lysine and valine. Besides containing essential amino acids, anchovies are also rich in non-essential amino acids. The prominent non-essential amino acids in anchovy are glutamic acid and aspartic acid, each of which reaches 1,439 and 966 mg / 100g of fresh anchovy. Other nutrients that are very meaningful from anchovy are minerals. calcium. phosphorus and iron (Sastra, 2008).

Fresh fish has the characteristics of being easily damaged and easily rot so it needs to be preserved. Preservation which is commonly used by the community is by cooling, drying and adding certain substances. Substances added to fish can be either natural or artificial substances. Based on the Minister of Health Regulation No.1168 /MENKES/PER/X/1999 the additional ingredients allowed for food are NaCl salt, sodiumtripolyphosphate (STPP), granulated sugar, sodium nitrite, sodium lactate, sodium acetate, and compounds (potassium nitrate, calcium nitrate, sodium nitrate). People generally do not use these substances for fear of changing the taste of the ingredients to be preserved. They choose prohibited

synthetic preservatives such as formaldehyde, because prices are relatively cheap, easy to obtain and their use does not require special expertise. The principle of preservation is to maintain fish as long as possible by inhibiting or stopping the activity of decomposing microorganisms. Preservation of fish will cause changes in the characteristics of fresh fish, both smell, taste, shape, and texture of the meat (Sutarni, 2013).

Formaldehyde that enters the body in amounts of 1.5 mg to 14 mg per day or in 0.1 ppm solution can be tolerated, if it exceeds the threshold it can cause interference with the organs and systems of the human body. The resulting consequences can occur in a short or short term and in the long term, through inhalation, direct contact or ingestion (Hastuti, 2010).

Wulan (2015) analyzed formaldehyde from 10 meatball samples by Uv-Vis spectrophotometer with Schryver reagents in Panakukkang District and the results were identified as many as 4 meatball samples which showed positive results with different levels of 0.357 µg/g, 0.285 µg/g, 1.504 $\mu g/g$ and 1.070 $\mu g/g$. Faradillah et al (2014) identified formaldehyde using a Uv-Vis spectrophotometer with Nash reagents on meatballs sold in Padang City. The results showed that 20 out of 42 (47.6%) samples taken from traders of meatball carts, meatball stalls, and restaurants contained formaldehyde. Putri et al. (2016)tested the formaldehyde content by Uv-Vis

¹Corresponding author: adrianimuharif@yahoo.co.id spectrophotometer with Schiff reagents on papaya (*carica papaya L.*) and pineapple (*ananas comosus L.*) fruit sold in the Raden Fatah State Islamic University Palembang and showed that all samples were tested containing formaldehyde with levels of 0.0007ppm and 0.0025ppm.

Abdullah (2013) has tested the of formaldehyde content by with spectrophotometer Scrhyver reagent on salted anchovy from the Market of Tuesday, Central and Galael of Gorontalo City and shows that the sample contains formaldehyde. Analysis of formaldehyde content by Uv-Vis spectrophotometer with Nash reagent on sunu fish from the Traditional Makassar Market by Riana (2015) also showed the presence of formaldehyde in the samples analyzed. Based on the previous description of the abuse of formaldehyde from various types of food, a study was conducted on the analysis of formalidehyde preservatives carried out on wet anchovy from the Traditional Market of Makassar City.

MATERIAL AND METHOD Instruments

The instrument used are label paper, stationery, destilator, pumpkin distillate, 100 mL volumetric flask, 50 mL and 10 mL, UV-Vis spectrophotometer, and glassware commonly used in laboratories.

Materials,

The materials used were samples of wet anchovy, 85% phosphoric acid (H₃PO₄), concentrated hydrochloric acid (HCl), Potassium Ferrisianide (III) (K₃Fe(CN)₆), phenylhydrazine, aquades, 37% formaldehyde, filter paper.

Methods

1. Sampling technique

Wet anchovy is taken from 4 different locations, namely 3 samples from Traditional Eggplant, Pa'baengbaeng, Antang and Daya markets. Wet anchovy samples taken from each trader of wet anchovy are packed in containers such as dry plastic. Plastic containers are coded. After that, samples of wet anchovy are taken from the sampling site to be carried out in the laboratory testing stage.

2. Formaldehyde Analysis Qualitative Test of Formaldehyde Compounds in Wet Anchovy Samples with Schryver Reagents

Twelve different samples of wet anchovy were mashed, each weighed as much as 30 g, then the sample was put into a distillation flask and added 200mL of distilled water. Then acidified with 85% phosphoric acid solution of 10mL. The solution is slowly distilled piped, put into a test tube and added Schryver reagent (2mL phenylhydrazine hydrochloride 1%, 5 mL concentrated HCl and 1mL K₃Fe(CN)₆). The solution will turn red if it contains formaldehyde in the sample (Riana, 2015).

3. Quantitative Test of Formaldehyde Levels on Wet Anchovy Samples with Spectrophotometric Methods

Making the mother liquor and standard. Formaldehyde 37% with concentrations. Then different the solution is put into a test tube that has been labeled, added Schryver reagent which is 1% phenylhydrazine hydrochloride as much as 2 mL, 5 mL concentrated HCl and 1mL K₃Fe(CN)₆ at each different concentration, Samples that are positive in qualitative testing, measured the absorbance with spectrophotometer at a wavelength of 518 nm (Riana, 2015).

4. Determination of Calibration Curves

Making 1000 mL Formalin 1000 ppm

Formalin solution 37% pipetted as much as 2.70 mL into a 1000 mL volumetric flask, added with distilled water, diluted to reach the boundary mark, homogenized.

Making 100 mL Formalin 10 ppm

Standard Solution 1000 ppm formalin mother liquor pipetted as much as 5 mL into a 50 mL volumetric flask, added with aquades, diluted to the boundary mark, homogenized.

Making 10 mL Formalin Standard Solution 0.2 ppm, 0.4 ppm, 0.8 ppm, 0.16 ppm and 0.32 ppm

The 10 ppm formalin standard solution is piped as much as 0.1 mL, 0.2 mL, 0.4 mL, 0.8 mL and 1.6 mL into a 10 mL volumetric flask, added with distilled water, diluted to the boundary mark, homogenized. Then each standard series solution is piped as much as 10 mL into a test tube, Scrhyver reagent is added, which is phenylhydrazine hydrochloride 1% as much as 2 mL, 5 mL concentrated HCl and 1 mL $K_3Fe(CN)_6$. Then the absorbance is measured.

Making a blank solution

Aquadest solution pipetted as much as 10 mL into a test tube, added Schryver reagent namely phenylhydrazine HCl 1% as much as 2 mL, 5 mL concentrated HCl and 1 mL K₃Fe(CN)₆. Then the absorbance is measured.

EXPERIMENTAL RESULT

QualitativeTestResultsforFormaldehydePreservativeswithSchryver reagents

Qualitative of analysis formaldehyde in wet anchovy samples can use several methods such as Nash reagent method, chromatoprat acid reagent method and Schryver reagent method (Suryadi et al, 2008). According to Schryver (1910) identification of formaldehyde by using the Schryver reagent method can be done by adding a solution of 2 mL fennilhydrazine hydrochloride solution, 1% potassium ferricianide solution of 1 mL, 5 mL concentrated hydrochloric acid into 10 mL of the material which will tested where if the food is tested. If react with formaldehyde will change color from colorless to bright red then its absorption is measured at a wavelength of 518 nm. The results of the qualitative analysis of formaldehyde with Schryver reagent on wet samples of anchovy (Stolephorus sp.) From the Makassar Traditional Market can be seen in Table 1.

Sample		reagent Color Test Schryver	Results
Daya Marker	PD A	Red	+
	PD B	Red	+
	PD C	Red	+
Antang Market	PA A	Red	+
	PA B	Red	+
	PA C	Red	+
Pa'baeng-baeng Market	PP A	Red	+
	PP B	Yellow	+
	PP C	Red	+
Terong Market	PT A	Red	+
	PT B	Red	+
	PT C	Yellow Chocolate	+

Table 1. Results of Qualitative Formaldehyde Test Analysis with Schryver Reagen

Information:

+	= Positive contains formaldehyde
-	= negative containing formaldehyde
PD	= Daya Market
PA	= Antang Market
PP	= Pa'baeng-Baeng Market
PT	= Terong Market

The prediction of the chemical reaction between formalin and Schryver reagent can be seen in Figure 1.

Based on the qualitative test of formaldehyde on anchovy (Stolephorus sp.) Wet in several Traditional Markets in Makassar City using the Schryver reagent method all samples showed that the sample contained formaldehyde, but 2 samples had very little formaldehyde so that the color produced differed from 10 other samples . This study is similar to the research conducted by Nuryahya (2015) on qualitative and quantitative analysis of formaldehyde content in some fresh fish 11 of the 12 samples tested positive for formalin and 1 sample that did not contain formaldehyde. Research on formaldehyde analysis in fresh fish with Schryver reagents

conducted by Wicaksono (2011) from Pasar Minggu shows the presence of formaldehyde in samples with an average level of 888.32 μ g/g. In addition, a study conducted by Yuaninda (2012) on formaldehyde testing in several markets in the Banten region showed that the fish observed had a very high formaldehyde content that exceeded the concentration value of formaldehyde solution which was equal to 3.75ppm which had a value of 0.032, the formaldehyde content not good for consumption. In general, the safe threshold of formaldehyde in foods that can still be tolerated in an adult's body is 1.5 mg up to 14 mg per day whereas formaldehyde in the form of water drink which can still be tolerated in the body which is 0.1 ppm (Singgih, 2013).



Figure 1. Formaldehyde reaction with Schryver Reagent

Material Quantitative Test Results Formaldehyde Preservatives With Using the method UV-Vis Spectrophotometer

First step on this research is determination maximum wavelength from formaldehyde standard series solution reacted with reagents modified by Schryver explains the reaction the chemical that occurs is condensation reaction between formaldehyde with phenylhydrazine forming phenylhydrazone, where in the presence of oxidizers will resulting in a reaction oxidation that produces a wet weak. Then wet is weak in the presence of strong acids excess will produce salt which can experience dissociation hydrolytic in dilution (Walter, 1996).

Oxidizing agent used by Schryver (1910) is potassium ferrisianide where in large quantities produce relatively stable colors, and by using concentrated hydrochloric acid as an acidic agent so that this method can be done for quantitative tests. This reagent consisted of 2 mL of 1% phenylhydrazine hydrochloride solution, 1 mL of potassium ferricianide solution, and 5 mL of concentrated hydrochloric acid, the presence of formaldehyde in the test solution was shown in bright red so that it could be used to measure formaldehyde **UV-Vis** using spectrophotometer. Color complex stability can only be maintained for 2 hours after the color complex is formed in an acidic state. Therefore, measuring levels using a spectrophotometer no more than 2 hours after the complex is formed (Azas, 2013).

Linear regression analysis y = ax + backstress + backstb. ideal linear relationship is achieved if $r^2 = 1$ or close to 1 (Harmita, 2006). The preparation for the formalin calibration curve is done by making a standard series of various measurement concentrations namely 0.02 ppm, 0.04 ppm, 0.08 ppm, 0.16 ppm, 0.32 ppm and then measuring its absorption at a wavelength of 512 nm. The formalin standard calibration curve can be seen in Figure 2. The calibration curve obtained a regression equation y = 0.2294x+ 0.02 with keephysisen (r^2) of 0.9625. The acceptance criteria of the correlation coefficient are (r^2) of ≥ 0.9999 Harmita (2006) which means that the results of the curve between absorbance and concentration have a linear relationship.

The calibration curve is then used to calculate quantitative parameters. Quantitative is a parameter in microscopic analysis and is interpreted as the smallest quantity of analytes in a sample that can still meet the criteria carefully and carefully. Quantity can be calculated statistically through a linear regression line from the calibration curve. The measurement value will be the same as the value of a in the liner line equation y = ax + b (Harmita, 2006). The quantity can be calculated statistically from the calibration curve. The measurement value will be the same as the value of a in the linear line equation y = ax + b (Harmita, 2006).



Figure 2. Formalin standard series calibration curve

Obtained line equation y = 0.2294x+ 0.02 to calculate the formaldehyde level in the sample. The samples that were analyzed quantitatively using a UV-Vis spectrophotometer were positive samples containing formaldehyde based on the results of a qualitative test, with different levels, samples from PD A; PD B; PD C with levels of 0.2702 ppm; 0,1307 ppm; 0.0871 ppm, PA A; PA B; PA C with levels of 0.1612 ppm; 0.2223 ppm; 0.1525 ppm, PP A; PP B PP C with levels of 0.1918 ppm; 0,0087 ppm; 0.2877 ppm, PT A; PT B; PT C with levels of 0.1394 ppm; 0.1961 ppm; 0.0043 ppm.

The lowest formaldehyde level was found in PT C with levels of 0.0043 ppm and the highest formaldehyde level was found in PP C with a level of 0.2877 ppm. The American Conference of Governmental and Industrial Hygienists (ACGIH) set the safe threshold of formalin in the body is 0.4 ppm (Alsuhendra and Ridawati, 2013). Whereas according to **IPCS** (International Program on Chemical Safety, that in general the safe threshold of formaldehyde in food that can still be tolerated in the adult body is 1.5 mg to 14 mg per day while formaldehyde in the form of drinking water can still be tolerated in the body, namely 0,1ppm (Singgih, 2013) The smallest formaldehyde level in the sample is 0,0087 ppm, so based on the tolerance limit of formaldehyde that can be accepted by the body in 1 day, and 9 other samples more than 0,1 ppm the sample of wet anchovy tested in the category is not safe for consumption The results determination of the of formaldehyde levels in all samples can be seen in Figure 3.



Figure 3. Graph analysis of formaldehyde levels in wet anchovy samples

CONCLUSION

Based on the results of the study it can be concluded that the wet anchovy sold in the Makassar Traditional Market with a qualitative test analysis of all samples of wet anchovy positively contains formaldehyde. The lowest level of formaldehyde is found in Terong Market sample C with a concentration of 0.0087 ppm. the highest body and formalin levels were found in Pa'baeng-baeng Market, sample C with a level of 0.2877 ppm, not safe for consumption.

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