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Reduksi Residu Sipermetrin dalam Produk Jambal Roti Ikan Manyung (*Arius Thalassinus* Ruppell)

(Reduction of Cypermethrin Residue in Jambal Roti of Giant Catfish (Arius Thalassinus Ruppell))

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ABSTRAK

Jambal Roti merupakan produk perikanan berupa ikan asin kering yang dibuat dari ikan Manyung (*Arius thalassinus* Ruppell) dengan tekstur menyerupai roti ketika selesai digoreng, ditemukan mengandung residu sipermetrin melebihi BMR yang diizinkan berdasarkan SNI dan CAC. Penelitian ini bertujuan untuk mengetahui penurunan kadar sipermetrin dalam produk Jambal Roti. Pada penelitian ini digunakan metode eksperimen mengikuti pola rancangan acak lengkap lima perlakuan diulang tiga kali. Perlakuan yang diberikan adalah A (dicuci dengan air yang mengalir selama 15 detik), B (direndam dalam air hangat dengan suhu 50^{o}C selama 15 menit), C (digoreng pada suhu 170^{o}C selama 10 menit), D (dicuci dengan air mengalir + digoreng) dan E (direndam + digoreng). Penentuan kadar residu sipermetrin produk jambal roti sebelum dan setelah pemberian perlakuan, dilakukan menggunakan kromatografi gas. Hasil menunjukkan bahwa perlakuan yang diberikan berpengaruh terhadap penurunan kadar residu sipermetrin pada Jambal Roti. Persentase penurunan kadar residu tertinggi pada perlakuan D (dicuci dengan air mengalir + digoreng).

Kata kunci : sipermetrin, Jambal Roti, ikan asin

ABSTRACT

Jambal Roti is a dry-salted fish product made from Giant Catfish (Arius thalassinus Ruppell) with a bread-like texture when fried. This product was found to contain cypermethrin residue exceeding the BMR maximum limit determined by SNI and CAC. This study aimed to determine the best processing method to minimize the residue of cypermethrin in Jambal Roti products. A completely randomize experimental methosd was employed with five treatments and the replicates of each treatment. Treatments applied were A (washing the Jambal Roti for 1 minutes with running tap water), B (soaking in warm water at 50 ° C for 15 minutes), C (frying at 170 ° C for 10 minutes), D (washing with running tap water then fried) And E (soaking then fried). The residue of cypermethrin was analyzed gas chromatography prior and after the experimental treatments. Results indicated that all treatments were capable of reducing the cypermethrin content of the Jambal Roti. The highest percentage of the cypermethrin

residue reduction was achieved by the treatment D in residual residuals in treatment D (washing with running tap water and then fried).

Keywords : *cypermethrin, jambal roti, dried salted*

INTRODUCTION

Jambal Roti is one type of salted fish that is well known in Indonesia, especially Java Island, much in demand and has its own place for salted fish enthusiasts. *Jambal Roti* is generally made from Giant Catfish (*Arius thalassinus* Ruppell) (Rochima, 2005). Characteristic of *Jambal Roti* include flavour caused by protein and fat degradation that produce methyl ketone compounds, butylaldehyde, amino acids, and other compounds. High amino acid content affects the taste of *Jambal Roti*. Other uniqueness are soft texture and compact as a result of the work of proteolytic enzymes produced by microorganisms (Rahayu *et al*. 1992)

Processing of *Jambal Roti* Giant Catfish is still done traditionally (Suaharna *et al.* 2006). Traditional processors, generally pay less attention to food safety issues produced products. Some food safety problems that come from deliberate processing are commonly found in traditional products such as *jambal roti*, salted fish, shrimp paste, pindang, peeled shellfish, peda, fish meal, shark fin and fish crackers. The processor uses dangerous additives which are prohibited for use in foods such as pesticides, formalin, borax, artificial colouring agents (Purnomo *et al.* 2002).

Jambal Roti made from Giant Catfish in Lamongan area have been found to contain pesticide residues of various active ingredients from organophosphate, organochlorine, carbamate and pyrethroid groups. Of the several active ingredients, the highest residue was obtained in the cypermethrin of 2.124 mg/kg (Amir *et al*. 2014). This cypermethrin residue is well above the maximum limit of Residual (BMR) of pesticides allowed under SNI and *Codex Alimentarius Commission*. Based on SNI 7313:2008 (Indonesian National Standardization, 2008) and the maximum limit of insecticide residual with active ingredient of cypermethrin on the poultry products of farm products of 0.05 mg/kg (Codex Alimentarius Commision, 2011)

Cypermethrin is a synthetic pyrethroid insecticide (Atessahi *et al.* 2005 ; Chakravarthi *et al.* 2007 ; Wenjun *et al.* 2007 ; Eraslan *et al.* 2008 ; Saxena *et al.* 2010 ; Muthuviveganandavel *et al.* 2011 ; Ojotiku *et al.* 2013). This compound is harmful to humans because it is a toxin that attacks the nervous system and organs associated with where the chemical enters the body, suppresses the immune system and inhibits the formation of antibodies against diseases caused by microbes (Sari *et al.* 2012).

Cypermethrin is commonly used in aerosol insecticide formulations (Kusumaningtiar and Angeliana, 2011), used to control insects or household pests (Kusumaningtiar and Angelina, 2011 ; Das and Parajuli, 2006 ; Bhusan and Saxena, 2013), pests on cotton and vegetables (Marigoudar *et al.* 2009 ; Debbab *et al.* 2014), rice and mango (Mukadam and Kulkarni, 2014) and other pests on agricultural activities Jayakumar *et al.* 2008 ; Sangha *et al.* 2011 ; Suzan *et al.* 2012 ; Masud and Singh, 2013). In *Jambal Roti*, sipermetrin is used to prevent

damage due to flies. Cypermethrin is prohibited its use in food according to Regulation of Minister of Health of Republic of Indonesia No. 722/Menkes/PER/IX/88 about a ban on the use of harmful chemicals not allowed in food. This prohibition relates to the toxic effects of cypermethrin on humans.

The use of cypermethrin which is incompatible with its function and size may threaten food safety for consumers as a result of residues left in the product (Haryati, 2006). Residue at a certain level, will endanger health especially when consumed continuously (Mutiatikum *et al.* 2002). Residual exposure can lead to acute and chronic poisoning. Chronic poisoning can occur due to continuous absorption over a long period of time even occasionally during life, albeit in very low doses (residues). Usually chronic poisoning is not accompanied by clear signs, but the result can cause damage to liver cells. Factors that contribute to this type of poisoning are cumulative properties of pesticides. The residue accumulates in fats resulting in liver and kidney damage (Mutiatikum and Isnawati, 2003). The residual cypermethrin accumulates in the human body (National Pesticide Information, 1998). Chemicals from the pesticidal content can poison the body's cells because of its ability to accumulate in the fat contained in the body. The chemical effects of pesticides in the short term cause liver necrosis (cell death), inflammation of cells, acute renal failure (Afriyanto, 2008).

Based on the description, the authors are interested in conducting research to reduce the residual cypermethrin in the product of *Jambal Roti*.

RESEARCH METHODS

The matherials used in this research are among others the product *Jambal Roti* of Giant Catfish (*Arius thalassinus* Ruppell), the standard of cypermethrin, n-hexane, acetone, diethyl ether, petroleum ether, acetontrile, methylene chloride, dodecane, sodium sulfate, helium, isooktana.

In this research, a completely randomize experimental methosd was employed with five treatments and the replicates of each treatment to obtain 15 experimental units. Working procedure in this research is sample preparation of *Jambal Roti* Giant Catfish. *Jambal Roti* Giant Catfish dipped into a solution containing cypermethrin 10 mg/kg for 5 minutes. *Jambal Roti* then treated : washing the *Jambal Roti* for 1 minutes with running tap water (Samad, 2006 ; Maruli *et al.* 2012 ; Fadwa *et al.* 2013), soaking in warm water at 50° C for 15 minutes (Budiarti *et al.* 2009), frying at 170 ° C for 10 minutes (Sumiati, 2008 ; Bello *et al.* 2010), washing with running tap water then fried, soaking then fried.

The parameters observed were the decrease value of cypermethrin residues in the product. The residual cypermethrin of *Jambal Roti* products before and after treatment, was analyzed using gas chromatography. Cypermethrin residue test refers to the procedure established (General Directorate of Food crop production, 2004), ie :

Extraction

Incorporated 5 g sample of *jambal roti* were crushed into the lid Erlenmeyer and added mixture of acetone: Dichloromethane (50: 50, v / v). For a static extraction process, the mixture is left for one night. Filtering the extraction results through a funnel with a cotton or glass wool that has been cleaned with a mixture

of Petroleum Ether and Acetone (4:1) for eight soxhlet hours. Pick up the 25 mL organic phase into a round flask. Condense in a Rotary Evaporator at the temperature of water bath 40°C, until almost dry, and then dried using nitrogen gas. Dissolving residue in 5 mL Isooktana: Toluene (90: 10, v / v).

Purification

Vaporize 20 mL of extract until almost dry by using *Rotary Evaporator* on the temperature of water bath at 40°C. Dissolving residue in 20 mL n-Hexane that contained 1 g of analytical samples. Incorporating consecutive glass wool, 5 mL n-Hexane and 1 g of activated silica gel. Mix and stir with stirring until evenly homogenous. Rinse the inner wall of the column with 2 mL of n-Hexane, til its meniscus just above the silica gel. Put 2 mL concentration of extract (equivalent to 1 g of analytic sample) into a rinse column with 3 × 1 mL n-Hexane, til its meniscus just above the silica gel. Eliminate with 20 mL eluent A (mixture of Ethyl Acetate and n-Hexane, 0.2: 99.8 v / v). Takes 10 mL the first of eluate (containing internal raw) and removes the rest of the eluate. Eliminate the pyrethroid with 35 mL eluent B (mixture of Ethyl Acetate and n-Hexane, 10: 90 v / v) and accommodate the eluate in a rounded flask and then insert 10 mL of first eluate which contained the internal standard. Evaporate with caution until dry. Dissolving residue with n-Dekana to its exact volume of 1 mL.

Determination

Inject 1-2 µL of extract into gas chromatography with Electron Capture Detector (ECD) equipped with RTX-1 column, 30 m x 0.25 mm. Oven temperature at 255°C, carrier gas of Nitrogen flowed 30 ml/min. Temperature of injector and detector were set at 280°C. The content of the tested substance were determined by comparing the width of peak area on the substance component to the area of the standard reference.

The amount of residues contained in the sample is formulated as follows (Suharna *et al.* 2006).

$$R = \frac{\frac{A_2}{A_1} x V_{i1} x K x \frac{V}{V_{i2}}}{W}$$

Description :

R = residues on sample (mg/kg)
A_2 = sample area
A_1 = standard area
V_{i1} = standard injection volume (µl)
V_{i2} = sample injection volume (µl)
K = standard solvent concentration (ng/µl)
V = last volume of concentration (µl)
W = weight of sample (g)
ng/g = 1000 = mg/kg

The data were analyzed using a pattern of a completely randomize and continued by Duncan test. Data processing is done computer program *SPSS 20 for windows*.

RESULTS AND DISCUSSION

To determine the decrease in the level residual of cypermethrin in the *Jambal Roti*, a gas chromatographic analysis of the treated *Jambal Roti* product was performed washing for 1 minutes with running tap water 100 ml/s, soaking in warm water at 50° C for 15 minutes, frying at 170 ° C for 10 minutes, washing with running tap water then fried, soaking then fried. The level of cypermethrin in *Jambal Roti* before being treated is 6,31 mg/kg. The level of cypermethrin in *Jambal Roti* after being treated can be seen in Table 1. The result of analysis of variance showed that the treatment given influenced to the percentage of decrease of cypermethrin level in product ($p<0,01$). And result of Duncan test ($p=0,05$) showed that there is difference of average decrease of cypermethrin level between treatment.

Table 1 shows that, the treatment provided can reduce the levels of cypermethrin in the *Jambal Roti* product. The highest percentage of decreased levels of cypermethrin was in the treated product was washed with running tap water then fried, and the percentage decrease in the lowest cypermethrin level in the treatment was soaked in warm water. The high percentage of decreased levels of cypermethrin in products that are washed and then fried is the influence of a combination of both. Cypermethrin has a solubility of 0.009 mg/kg in water (Haryati, 2006), by washing, the levels in the product can be reduced. With the washing with running tap water, cypermethrin will fall along carried water. And with a semipolar nature cause cypermethrin easily soluble in oil.

Table 1. Cypermethrin Level of *Jambal Roti* after Treatment

Treatment	Average (mg/kg)	Percentage Decrease (%) ±SD
Washing with running tap water	2,45	$61,23\pm0,10^{c}$
Soaking in warm water	4,91	$22,13\pm0,05^{e}$
Frying	3,40	$46,12\pm0,09^{d}$
Washing with running tap water then fried	1,64	$73,96\pm0,04^{a}$
Soaking then fried	2,23	$64,71\pm0,03^{b}$

Description: The letters followed by the same superscript letter in a single column show that the treatments are not significantly different (p = 0.05)

The level of cypermethrin in the frying treatment, reduced about 46.12% lower than the treatment was washed in running tap water. This is due to the low fat content in the *Jambal Roti* so that a little residual cypermethrin that accumulates in the fat of *Jambal Roti*. Taufik and Yosmaniar, 2010 states that residual accumulation is affected by fat content. In other words, products that have a high fat content will more easily accumulate insecticides.

With soaking, the levels in the product are reduced in small amounts, since the residual possibility of clinging back during soaking. In addition, low levels of

cypermethrin can be reduced by soaking, caused by a chemical equilibrium that is a two-way chemical reaction, in which the reaction product can change back into reactants until the concentrations of reactants and products are constant. The chemical reaction reaches equilibrium if the reaction rate to the right is equal to the reaction rate to the left so that no change occurs in the equilibrium system. Maruli *et al.* (2012) suggests that the residual pesitisida can be hydrolyzed depending on the amount of water present and the concentration of the pesticide.

Some studies show that on eggplant and okra, 30,2-92,1% cypermethrin can be reduced by washing in running tap water (Chandra *et al.* 2015), 52-92% (Kwon *et al.* 2015), 37-73,2% (Harinathareddy *et al*. 2014) and 47-53% (Vemuri *et al.* 2014) pesticide residues on tomatoes, 48,26-70% pesticide residue in cauliflower (Thanki *et al.* 2012), 90% pesticide residues on carrots (Bonnechere *et al.* 2012), 31, 29, and 26% pyrethroid synthetic pesticide residues in okra, cauliflower and eggplant (Kumari, 2008). On beef, 29-62,2% pesticide residue can be reduced through heat treatment (boiled in boiling water) for 90 minutes (Letta and Attah, 2013) and 10-18% through fermentation for 72 hours (Abou-Arab, 2002). On buffalo meat, 55,93-64,59% pesticide residue can be reduced by boiling in a pressurized pot (Muthukumar *et al.* 2010).

Cypermethrin has a molecular formula $C_{22}H_{19}Cl_2NO_3$, is semipolar because it consists of polar and non polar groups. The presence of an ether group C-O-C which is polar on the structure of cypermethrin to make cypermethrin dissolve in water and Cl_2 is non polar causing sipermetrin dissolve in oil. In the structure of cypermethrin there is also an ester group O-C=O which is more polar than ether. Group C=O in the highly charged negative cypermethrin will easily bind to the positively charged, ie H+ on H_2O.

In the frying process, ester removal occurs due to hot temperature. The removal of the ester during frying occurs in an acidic atmosphere. Jones (2014) suggests that sipermetrin with IUPAC [cyano-(3-phenoxyphenyl)methyl] 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropane-1-carboxylate will be 4-hydroxy-3-phenolxybenzoic acid because the ester breaks in the acidic atmosphere.

Jambal roti contains amino acid valine, threonine, metheonine, lysine, isoleucine, leucine, phenylalanine, aspartic acid and glutamic, serine, glysine, histidine, agrinine, cystine, proline and alanine. Of these amino acids, lysine and threonin, which are essential amino acids, are thought to co-exist with cypermethrin in the washing due to the polar nature of the amino acid. While metheonine, isoleucine and phenylalanine which is also an essential amino acid, allegedly participate dissolve with cypermethrin at the time fried because of the non-polar nature of the amino acid. Presumably the content of Eikosapentanoat (EPA) and Dokosaheksanoat (DHA) on bread jambal also dissolves with cypermethrin due to fried treatment

CONCLUSION

All treatment methods tested were able to reduce the residual levels of cypermethrin in Jambal Roti, but the best treatment was washing Jambal Roti with water flowed then fried. This method can reduce the level of cypermethrin up 74%.

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