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The Effect of Combination of Liquid Smoke To Degrees of Material (PH) Fillet Fish Tilapia

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Abstract : The aim of this research is to determine the acidity level (pH) of tilapia fillet (*Oreochromis niloticus*) given by liquid smoke of cinnamon with combination of liquid smoke treatment, soaking time, packing type and different storage time of tilapia fillet. This research was conducted in Kopertis Region X Padang laboratory, the material of tilapia fillet selected average weight 250 gram, put in ice box. Experiments were carried out experimentally by using a factorial experiment in a 5x3x3x5 Randomized Complete Design pattern with 3 replicates; Factor A consisted of liquid smoke concentration: control (0%), 5%, 10%, 15% and 20%; Factor B consists of immersion time: 5 minutes, 10 minutes and 15 minutes; Factor C consists of packaging type ie without packaging, polyethylene (PE) and polypropylene (PP) packaging and D factor consisting of storage time (days) consisting of 5 (five) levels of 0, 3, 6, 9 And 12 days. The parameters measured are the degree of acidity (pH) using a manual procedure. . The results showed that there were real interactions on the combination of four immersion treatments, concentration differences, type of packaging and different storage times against acidity (pH). Combination treatment of cigarette smoke smoke 5%, 10%, and 15% concentration, 5 minutes, 10, and 15 minutes immersion, PP packing type, PE and without packaging and storage time 0, 3, 6, 9, and 12 Day showed the highest pH yield of 6 (six) and statistically there was interaction, while the lowest pH (5.044) occurred in combination of 15% smoke smoke concentration treatment, 10 minutes immersion time, type of polyethylene (PE) packaging for 12 days storage period.

Keywords : liquid smoke, acidity degree, tilapia fillet, packaging, storage, soaking.

1
I. Introduction

The type of freshwater fish that is now being developed and cultivated in West Sumatra Province is Nila fish (*Oreochromis niloticus*). The potential of aquaculture is estimated to be 12,300 hectares^[1] with total production of 109,371.80 tons. The superiority of tilapia fish because it is easy to live, fast breeding, white meat and taste quite tasty. The way of processing that can be developed for the fish is the processing of fish fillets. The result of fishery processing is fish fillet including food which is very fast decaying (high perishable food), then fish quality must be maintained as much as possible until reaching to consumer's hand. The way to maintain quality one of them with good handling and preservation and processing into ready-to-eat products but longer lasting power such as fumigation.

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Fogging technology includes preservation of fish that have been applied for generations^[2]. The term smoking means to absorption of various chemical compounds derived from wood smoke into fish, accompanied by half drying and usually preceded by salting process. Thus, the term smoke curing encompasses the entire process that begins from the preparatory stage of the raw material to the last fuming which results in discoloration, flavor and texture of the fish. While the purpose of fumigation in fish preservation is to preserve and give the color and flavor of the special smoke in fish. Fogging aims to extend the shelf life of an ingredient, but in line with the increase in public acceptance of the smoke product, the goal begins to switch to flavor, which gives a distinctive aroma and taste.

Smoking can be done traditionally or in a modern way. Traditional fumigation can be done cold and hot by burning wood or wood powder, smoked fish in direct contact with smoke. Modern fumigation uses liquid smoke (the dispersion of vapor in liquids as a result of condensation of smoke from wood pyrolysis) as a smoking medium. The wider community, especially the coastal community, generally fog with traditional fumigation techniques, whereas this fumigation technique has many shortcomings, such as long time, inefficient use of firewood, product uniformity to produce desired color and flavor difficult to control, pollution environment, and the most dangerous is the presence of tar residues and aromatic polycyclic hydrocarbon compounds (*benzo(a)pyren*) deposited in food so that it can endanger health. Tree felling is mostly done in areas of smoked fish, to meet the source of smoke (firewood), even the mangroves that become coastal protectors were not spared from the target of logging. This situation makes an alternative use of firewood should be considered and fumigation techniques it is time to be replaced with modern fumigation.

Several studies on the production and use of liquid smoke have been carried out, among others, determining the temperature and timing of pyrolysis of rubber wood to produce quality liquid smoke^[3]. Research on cinnamon raw materials at 400°C pyrolysis temperature produces quality liquid smoke^[4]. Research of cinnamon with 400°C pyrolysis at 1500 ppm shows the highest antioxidant rate of 35.091%^[5]. Determination of antibacterial properties of liquid smoke produced from several types of softwood^[6]. Preservation of smoke tongue with liquid smoke produced from teak^[7]. The results of research Budaraga et al.^[8] get the dominant content of coconut husk liquid smoke, coconut shell and cinnamon containing acetic acid and phenol. Budaraga et al.^[9] reported the results of cytotoxic properties (the ability to kill artemiasalina) cinnamon liquid smoke at 400°C pyrolysis temperature of 19.048%. These studies all utilize hardwood and softwood separately, whereas softwoods with low lignin content will be very effective in lengthening the durability of fish and resulting in unspecific flavors^[10] when combined with other wood (hardwood).

Based on the above research, cinnamon is very appropriate to be used as a preservative. Further research results Budaraga et al.^[11] obtained the result of purification of liquid smoke cinnamon on distillation temperature 140oC not detected benzo (a) pyren, Further research Budaraga et al.^[12] to obtain the toxicity of cinnamon liquid smoke purified by deposition for 3 days by 83.75%. The result of antioxidants of liquid smoke of cinnamon by means of different purification produces strong enough antioxidants (<50 ppm)^[13]. Further research results Budaraga et al.,^[14] get the results the measurement of antibacterial properties of E. Coli on cinnamon liquid smoke purified by settling for 3 days resulted in inhibitory diameter of 34.129 mm/ppb, The existence of soaking process at the concentration of cinnamon liquid smoke that appropriately will affect the physical and chemical properties that so far no information about it. The results of Hardianto and Yuniarta^[15] research, obtained the best treatment based on chemical and organoleptic parameters in tuna obtained in treatment on storage for 2 days at room temperature and 2 hours of soaking time with coconut shell liquid smoke type.

Subsequently, Harini N, Wachid^[16] obtained liquid smoke from 3 types of liquid smoke (coconut shell, corn cob and bamboo) can be useful as a preservative indicated by the ability of drag zone and absorbance value. Liquid smoke has the ability to inhibit pathogenic bacteria (*Escherichia coli*, *Pseudomonas aeruginosa*, *Bacillus subtilis* and *Staphylococcus aureus*) at various storage periods of mujair fish (days 1, -2, -3 and -4). The results of Budaraga IK^[17] that the results of moisture content on tilapia fillet treated with polyethylene (PE) plastic treatment with a long immersion of 15 minutes on the initial day of 11.934%, and water content from a combination of four treatments included in the Indonesian national standard (SNI), with a maximum water content in smoked fish of 60 percent. The results of Budaraga IK^[18] research get a combination of 5% cinnamon liquid smoke immersion for 5 minutes on two types of polyethylene and propylene packing in the initial storage was found the protein content of 79 percent.

Based on the above description with the drying activity on the tilapia fillet resulted in decreased levels water, causing the product fillet tilapia become more durable. On the other hand fish processing conditions in the community still use the traditional way, fish fillet products in the form of jerky usually not packaged properly so easily contaminated. As long as it has never been measured its shelf life, sometimes the water content is still relatively high. The durability factor of the tilapia fillet product is closely related to pH. To determine effectiveness of liquid cinnamon smoke absorption as preservative of tilapia fish fillet preferably fillet thickness factor is considered. The preservation process will be more effective if the packaging is done. Packaging can prevent contamination with microbes and other damage. Until now there is no information about the influence of the right type of packaging and storage time for the pH of tilapia fillets that are preserved with liquid smoke.

Dewi et al.,^[19] reported the best quality of fish jerky obtained by the absence of packaging during storage. The results of Swastawati F. et al.,^[20] showed that the difference between the curing method and the type of fish gave a very significant difference ($P < 0.01$) to the proximate value (protein, fat, water, ash content). Higher content of phenol, formaldehyde, and organic acid is using a furnace. So it can be concluded that both the curing method and the fish species can be applied to the curing of fish although there is a tendency of specific characteristics of the product resulting in appearance, odor, texture and taste. This study aims to determine the degree of acidity (pH) of tilapia fish fillet given combination of liquid smoke concentration treatment, soaking period, packaging type and different storage time

II. Materials and Methods

2.1. Materials and tools

The ingredients used for the manufacture of fish fillets are black tilapia bought in crocodile market with average weight of 250 gram / cow, 70% alcohol, dolpin dyed salt table salt, clean water, purified liquid cinnamon smoke with distillation temperature 140°C the best research results Budaraga IK et.al.,^[14]. The tools used in this study are digital scales (Quattro), pumpkin powder (merck), glass beaker (merck), measuring cylinders (merck), pipettes (merck), propellet (merck) stirrer (merck), heat- Patent I Ketut Budaraga no ID S0001244^[21], Dry oven (memmert), storage rack, polyethylene plastic, polypropylene plastic, label paper, paper plate for fillet, refrigerator, freezer, pumpkin (pyrex), pH meter, erlenmeyer 125 mL (Pyrex) and 500 mL, trophy glass (Pyrex).

2.2. Research methods

2.2.1. Preparation of liquid smoke

Liquid liquid cinnamon purifying with distillation temperature 140°C best results Budaraga IK et.al.,^[14] prepared then diluted with distilled water. The concentration of liquid smoke preservative used was without liquid smoke (control), 5%, 10%, 15% and 20%.

2.2.2. Preparation of Tilapia Fillet and pickling with liquid smoke

Preparation of materials and tools such as workbenches, knives, cutouts that have been sterilized with 70% alcohol and liquid smoke cinnamon that has been purified. Tilapia selected in fresh condition refers to SNI^[22] about the specification of fresh fish about the requirements of raw materials with the characteristics of clean raw materials, free of any odors indicating decay, free of decomposition and counterfeiting marks, free of other natural properties which can degrade the quality and not harm the health, the fish used organoleptically raw materials have the characteristics of freshness: intact appearance, convex eyes, brilliant white meat incision; smell: fresh specific fish; texture: solid, compact and elastic, with weight 250 ± 10 gram. The making of tilapia fillets is done on fresh fish that has passed the freezing phase (rigor mortis) and cleanliness is always maintained by weeding the scales of fish, dispose of stomach contents, dirt, and a layer on the black stomach wall, then washed until clean to remove the rest of the dirt, blood, loose scales and also lenders. The next net fish is done from the back of the gill fin to the back of the head; incision in front of the head towards the tail along the dorsal fin using a stainless steel knife and knife made parallel so loose from the ribs when picking up the fillet. Fish fillets are done on both sides with a boneless fish block shape. The obtained fillet is then immediately inserted into the freezer temperature -20°C immediately, to prevent any deterioration. Ways to

reduce drip (water from missing muscle tissue on frozen product melted) fillet is immersed in a pure 15% salt solution for 20 seconds.

Making fillets is done quickly but carefully to avoid decay, pollution and defects due to carelessness that can adversely affect the product and to anticipate this is placed in the freezer. Waste obtained from filtration is immediately removed from the treatment to avoid product contamination. Fillets in the form of beams are easily stored, transported and handled SNI^[23]. The fish blocks were then cut in sticks ($\pm 5 \times 10$ cm in size with ± 2 cm thick) and were given a 5%, 10%, 15%, 20% and aqueous smoke control and combined with long immersion different ones are 5, 10 and 15 minutes. After completion the fillet immersion is lifted and drained and air-wind until the fillet surface is dry. The tilapia fillet is then arranged on top of the oven shelves to be evenly distributed, and dried at 70°C for 6 (six) hours.

The dried tilapia fish fillet is then cooled to room temperature for ± 20 minutes until cooled into a clean styreform and hygienic container SNI^[24], then inserted into polyethylene (PE), polypropylene (PP) packaging and without packaging then stored at room temperature and observations were made on days 0, 3 days, 6 days, 9 days and 12 days of acidity (pH)^[25].

2.3.Level of acidity (pH) (Manual Procedure)^[25].

The sample is smoothed, weighed 1 gram in a cup glass. Then added 10 mL aquadest and Stirring done. Next, the sample in the container measured its pH by using a pH meter that had been calibrated with buffer solution pH 4 and buffer pH 7. The pH value was obtained based on the reading at pH meter until the digital number indicates a constant number.

2.4.Data analysis

This research uses Completely Randomized Design (RAL) with a factor that is combination of liquid smoke concentration with soaking period, packing type and storage time to obtain 5x3x3x5 experiment x 3 replication = 675 unit experiment. The first factor consists of 5 (five) levels, namely the concentration of control liquid smoke, 5% and 10%, 15% and 20%; the second factor of immersion duration with liquid smoke consists of 3 (three) levels ie the immersion time of 5 minutes, 10 minutes and 15 minutes; the third factor of the packaging type consists of 3 (three) levels ie without packaging, polyethylene packaging (PE) and polypropylene packaging (PP) and the storage time factor (day) consists of 5 (five) levels ie 0, 3,6,9 and 12 days. Observational data in the form of protein and fat content were analyzed by diversity analysis at 5%, if significantly different with Tukey test at 5%^[26]. Tabulate data with MS Office Excel 2010 and ANOVA test using SPSS 22 program.

III. Results And Discussion

Degree of acidity (pH)

Table pH value presented in Table 1 and Figure 1 below.

Table 1. Average degree of acidity (pH) of tilapia fillet, based on differences in soaking period, liquid smoke concentration, packaging type and storage duration

Type	Time (K)	Concentration (L)	Time storage (S) (day)					Mean (L)/(K)	Interaction
Packaging	soaking (minute)	liquid smoke (%)	0 (S ₀)	3 (S ₁)	6(S ₂)	9(S ₃)	12(S ₄)		L/K
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
		0 (L ₀)	6,00	6,00	6,00	6,00	6,00	6,00	0,00
	5 (K ₁)	5 (L ₁)	5,00	5,00	5,00	5,00	5,00	5,00	0,00
		10 (L ₂)	6,00	6,00	6,00	6,00	6,00	6,00	0,00
		15 (L ₃)	5,59	5,29	5,43	5,87	5,37	5,51	-0,13
		20 (L ₄)	6,00	6,00	6,00	6,00	6,00	6,00	0,00
	Mean 5 minute		5,72	5,66	5,69	5,77	5,67	5,70	-0,08
	Interaction		0,12	0,09	0,09	0,17	0,07	0,10	

	Mean	0 (L ₀)	5,79	5,86	5,76	5,83	6,00	5,85	-0,04
	concentration	5 (L ₁)	5,33	5,33	5,33	5,33	6,00	5,47	-0,13
	Liquid smoke (L)	15 (L ₃)	5,67	5,67	6,00	5,67	5,67	5,73	0,00
		20 (L ₄)	5,69	5,45	5,60	5,93	5,53	5,64	-0,11
		20 (L ₄)	6,00	6,00	6,00	6,00	6,00	6,00	0,00
	Interaction (L)		0,15	0,08	0,15	0,19	-0,09		
	Mean								
	Time Soaking (K)		5,69	5,66	5,74	5,75	5,84	5,74	
	Interaction (B*K*L*S)		-0,06	-0,11	-0,21	-0,02	0,04		
		0 (L ₀)	6,00	6,00	5,60	6,00	6,00	5,92	0,00
	5 (K ₁)	5 (L ₁)	5,00	5,00	5,67	5,00	5,00	5,13	0,00
		10 (L ₂)	6,00	6,00	6,00	6,00	6,00	6,00	0,00
		15 (L ₃)	5,59	5,29	5,49	5,89	5,39	5,53	-0,14
		20 (L ₄)	6,00	6,00	6,00	6,00	6,00	6,00	0,00
	Mean 5 minute		5,72	5,66	5,75	5,78	5,68	5,72	-0,05
	Interaction (B1*K*L)		0,118	0,06	0,12	0,18	0,08	0,11	
		0 (L ₀)	6,00	6,00	6,00	6,00	6,00	6,000	0,000
Packaging PE (B ₃)	10 (K ₂)	5 (L ₁)	5,89	5,39	5,59	5,29	5,49	5,530	0,22
		10 (L ₂)	6,00	6,00	6,00	6,00	6,00	6,000	0,00
		15 (L ₃)	6,00	6,00	6,00	5,60	6,00	5,920	0,16
		20 (L ₄)	5,00	5,00	5,00	5,67	5,00	5,134	-0,27
	Mean 10 minute		5,78	5,68	5,72	5,71	5,69	5,72	-0,07
	Interaction (B2*K*L)		-0,38	-0,28	-0,32	-0,07	-0,29	-0,27	
		0 (L ₀)	6,00	6,00	6,00	6,00	6,00	6,00	0,00
	15 (K ₃)	5 (L ₁)	5,67	6,00	6,00	6,00	6,00	5,93	-0,13
		10 (L ₂)	5,67	5,00	5,00	5,00	5,00	5,13	0,27
		15 (L ₃)	6,00	6,00	6,00	6,00	6,00	6,00	0,00
		20 (L ₄)	5,49	5,89	5,39	5,59	5,29	5,53	0,08
	Mean 15 minute		5,77	5,78	5,68	5,72	5,66	5,72	-0,02
	Interaction (B3*K*L)		-0,14	-0,04	-0,24	-0,16	-0,28	-0,18	
	Mean	0 (L ₀)	6,00	6,00	5,87	6,00	6,00	5,97	0,00
	concentration	5 (L ₁)	5,52	5,46	5,75	5,43	5,50	5,53	0,03
	Liquid smoke (L)	10 (L ₂)	5,89	5,67	5,67	5,67	5,67	5,71	0,09
		15 (L ₃)	5,86	5,76	5,83	5,83	5,80	5,81	0,01
		20 (L ₄)	5,50	5,63	5,46	5,75	5,43	5,55	-0,06
	Interaction (L)		-0,13	-0,09	-0,15	-0,02	-0,17		
	Mean time soaking (minute) (K)		5,75	5,70	5,71	5,73	5,68	5,72	
	Interaction (B*K*L*S)		-0,03	-0,08	-0,05	0,05	0,02		
	CV = 1,70								

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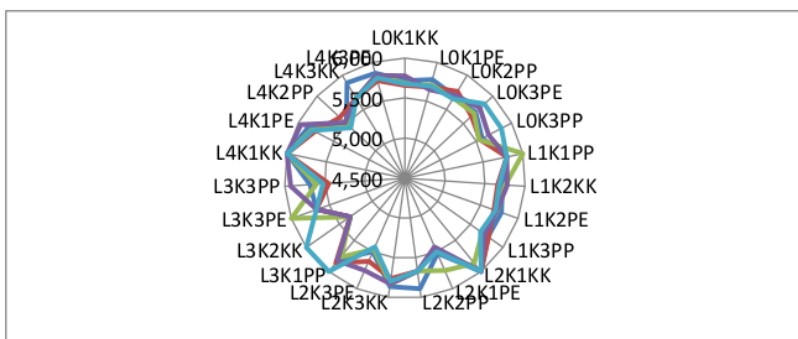


Figure 1 Average degree of acidity (pH) of tilapia fillet, based on difference of immersion time, liquid smoke concentration, packing type and storage time (0 days, 3 days, 6 days, 9 days, 12 days),

Based on Table 1 and Figure 1 above, the interaction values of the four treatment factors starting without packaging on the old soaking, the type of packaging and the length of storage are different, all providing a positive interaction value except for a 3-day storage period providing negative interaction values. This means that in the treatment of all four factors give the same response to the pH of the tilapia fillet, or in other words that the response given to each factor is smaller than if done together. Interaction values on Polypropylene (PP) packaging starting with 0.3.6 and 9 day storage showed negative interaction value, then at 12 days storage time showed positive interaction value. Negative interaction values mean the four treatment factors provide an unequal response to the pH of the tilapia fish fillet or in other words that the four treatment factors give a lower response than from each factor. Subsequently, the interaction on polyethylene (PE) packaging at 0.3 and 6 day storage gave negative interaction value, while 9 and 12 day storage gave positive interaction value.

The mean pH measurement results showed in combination of cigarette smoke bath treatment concentration of 5%, 10%, and 15%, duration of soaking 5 minutes, 10 and 15 minutes, PP packing type, PE and without packaging and storage duration 0, 3, 6, 9, and 12 days showed the highest pH yield of 6 (six) and statistically there was interaction, while the lowest pH (5.044) occurred in combination treatment of smoke concentration 15%, 10 minutes immersion time, polyethylene packing type (PE) on a 12 day storage period. This pH difference occurs due to an increase in the concentration of liquid smoke and soaking longer on the tilapia fillet so that the pH becomes more acidic, compared with the lower concentration and shorter immersion period. Figure 1 shows an increase in pH value of dried tilapia during storage. Based on the figure can be seen that during the storage of 0 days to 12 days had an insignificant increase. This is presumably because the activity of the decomposing enzyme has not worked effectively so that the pH rises but is not significant. Chamidah^[27] states that during the storage occurs the decomposition of proteins into basic compounds, among others, ammonia. The pH value of foodstuffs during storage may change due to proteins decomposed by proteolytic enzymes and the aid of bacteria to carboxylic acids, sulfide acids, ammonia and other types of acids. According to Zakaria^[28] when the amount of acid more than ammonia, the pH value becomes low. Therefore, the pH value of the dried tilapia fish fillet is produced although it is increasing but still under acidic conditions. Another cause because the condition of fish at the time of processing the fish into a fillet is already in postrigor mortis, because marked no stiffness in fish. At the end of the rigor phase as more decomposition results, the activity of bacterial decay begins to increase. When the rigor phase has passed (the fish body begins to soften) then the rate of decay will increase. Previous research by Lakoro^[29] showed the pH value of smoked roa fish was 6.06, while Tubagus^[30] study on fillet fillet showed pH value of 5.58. The low pH value of this dried tilapia fish fillet, thought to be the result of a combination process of aqueous fumes degan drying. This combination process will result in more durable products due to slower quality slowdown.

The most acid compounds in liquid smoke are carboxylic acid derivatives such as furfural, furan, and glacial acetic acid which can inhibit microbial growth^[6]. The higher the concentration of liquid smoke added to the product the lower the pH value. The presence of liquid smoke will inhibit the decline in quality of fish before processing. Darmadi^[31] states that liquid smoke will lower the pH so that it can slow the growth of micro-organisms. Liquid smoke at pH 4 is able to inhibit all bacterial decay and pathogens, whereas at a high pH of about 6.0 the inhibition of liquid smoke to bacterial growth begins to decrease.

According to Winarno^[32] smoke ingredients such as formaldehyde, acetone and phenol have the properties of killing bacteria, while volatile acids in smoke will lower the pH on the surface of fish flesh and slow the growth of micro-organisms. Furthermore Winarno^[32] states that in the storage will occur changes in humidity and temperature which is a determinant of the speed of overhaul of enzymes and bacteria in the food that can cause changes in pH for a certain period. Increased pH in fish fillets also caused by the process of decay, amino is converted into an alkaline ammonia compound. Atmadjaja^[33] (1994) stated that fish pH values ranging from 6.0-7.0 and temperatures around 25-30 ° C are ideal conditions for the growth of decaying bacteria. While low pH can inhibit contamination of decomposing micro-organisms, pathogen microorganisms and toxic-producing microorganisms will die [34]. The pH measurements were performed to determine the tendency of pH increase / decrease during treatment of tilapia fillets. The amount of pH is related to the formation of alkaline compounds during treatment and will affect the growth of microbes. The results of research Budaraga et al.^[17] showed that the highest value of tilapia fillet content was obtained in combination of treatment of soaking time of 15 minutes without immersion of liquid smoke (liquid smoke concentration 0%) of 73.686% significantly different from other treatments, the concentration of liquid smoke combination treatment 0% with a 9-day time deposit of 71.77% significantly different from other treatments, while the other treatment combinations were not significantly different. At the water content of tilapia fillets, there was no interaction on the combination of four treatments, immersion difference, immersion concentration difference, packaging type and storage time ($P < 0.05$), but interaction on the combination of three treatment differences such as immersion time, concentration and time difference storage against the water content of tilapia fillets. Interaction also occurred in the long period of immersion, the type of packaging and the storage time while the combination of two, the other three treatments did not show interaction with the content of water content of tilapia fillets. The lowest content of water content on tilapia fillet on packaging treatment on polyethylene plastic bag (PE) in long submerged for 15 minutes with storage for 0 days (11.934%) and moisture content from a combination of four treatments resulted in moisture content of BSN^[22] namely smoked fish maximum moisture content of 60%. Further research results Budaraga et al.^[18] reported that the combination of immersion for 5 minutes, a 5% smoke-smoke concentration with two types of packaging (polyethylene, polypropylene and without packaging) on the nila 0 store day showed the highest 79% protein content in tilapia fillets.

IV. Conclusion

There was a real interaction on the combination of four different immersion treatments, different concentrations, packaging types and different storage times against the degree of acidity (pH). Combination of cigarette smoke bath treatment concentration 5%, 10%, and 15%, soaking time 5 minutes, 10, and 15 minutes, packaging type PP, PE and without packaging and storage duration 0, 3, 6, 9, and 12 day showed the highest pH yield of 6 (six), while the lowest pH (5.044) occurred in combination of 15% smoke smoke concentration treatment, 10 minutes immersion time, type of polyethylene packaging (PE) for 12 days storage period.

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