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The Effect of Soaking Time and Liquid Smoke Types on the Quality of Smoked Skipjack (*Kastuwonus pelanis* L.) Fillets During Storage

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ABSTRACT

This study aims to determine the effect of soaking time in distilled and non-distilled liquid smoke of kusambi wood (Schleicera oleosa) on the quality of smoked skipjack fillets. This study used an experimental method designed with a completely randomized design (CRD) with two factor treatments. The first factor was soaking time with four levels, namely 10, 20, 30 and 40 min. The second treatment factor was the type of liquid smoke from kusambi wood (without and with distillation). The variables observed in this study focused on the quality of the smoked skipjack fillets after the 8-day storage period, including: water content (%), pH, total acid (%), fat content (%) protein (%) and phenol content (%). The results showed that the liquid smoke of kusambi wood with distillation had a pH of 3.18 which was more acidic and had higher phenol content (0.61%) and acid content (17.62%) than liquid smoke without distillation. Of the eight treatment combinations, the quality of smoked skipjack fillets after 8 days of storage, it is found that the treatment of soaking 30 min and 40 min in distilled liquid smoke is the best treatment and meets the quality requirements of SNI. This is based on the value of water content (48.46% and 49.67%), pH (4.04 and 3.96), total acid (8.84% and 9.07%), fat content (4.85% and 4.92%), protein content (18.04% and 17.83%) and phenol content in smoked skipjack fillets (0.65% and 0.61%).

1. INTRODUCTION

Fish is one of the animal products that is classified as a source of protein because it contains quite high protein (Prameswari, 2018). However, fish and other fishery products are highly perishable foodstuffs, so the market value of their processed products is determined by the degree of freshness and durability (Hiola, 2018). For this reason, it is necessary to handle fishery products. Improper handling of processed products results in fish processed foods being easily damaged because they will be contaminated with pathogenic bacteria (Sartika *et al*., 2019).

One of the fish preservation techniques that has long been widely known is smoking (Mustikawati *et al*., 2016). Various studies have shown that smoking for fish and various other food products is a preservation method that not only increases shelf life, but also gives the desired taste and color to smoked products due to the presence of phenol and carbonyl compounds. Although the original purpose of smoking was good, it turns out that the smoking process can produce compounds that are not safe for health. In addition to containing components that act as preservatives such as phenol, organic acids, smoke also contains Polycyclic Aromatic Hydrocarbon (PAH) compounds. This PAH is a carcinogenic compound that causes cancer (Darmadji & Yudiana, 2006). One of them, is benzopyrene, which has been identified as a PAH compound that has high carcinogenic properties, because it can form complexes with DNA permanently and cause mutations in genes (Oktafany *et al*., 2016). Benzopyrene is considered an indicator of carcinogenic compounds in smoked foods. Therefore, it is necessary to use smoking techniques and methods to produce smoked fish that is safer for consumption.

This effort can be done by using liquid smoke. Liquid smoke is the result of condensation originating from steam resulting from direct or indirect combustion of materials containing lignin, cellulose, and hemicellulose as well as other carbon compounds (Darmadji, 2020). This liquid smoke can be produced through pyrolysis technology. Pyrolysis liquid smoke has different qualities depending on the type of wood and the distillation process (purification). Distillation of liquid smoke is aimed at obtaining functional properties of liquid smoke, as well as removing unwanted compounds such as tar and benzopyrene (Erawati *et al*., 2015; Sahrum *et al*., 2021).

According to Swaswati *at al*. (2013) and Ghazali *et al*. (2014), fumigation using liquid smoke can produce a uniform product, safe, easy to control, providing a consistent taste and aroma, saving wood, reducing pollution and preventing deposits of tar compounds. Furthermore, according to Simon *et al*. (2005), liquid smoke has several advantages, namely easy to apply, more uniform product flavor, can be used repeatedly, more efficient in the use of smoking materials, and can be applied to various types of food ingredients. Liquid smoke can be applied in various ways such as spraying, soaking, immersing or mixing directly into food.

Important factors that need to be considered in the application of liquid smoke using the soaking method are the concentration of liquid smoke and the soaking time. The duration of soaking in liquid smoke is also a determining factor for the organoleptic quality of the product. Katiandagho *et al*. (2017) stated that soaking wood fish for 30 minutes in liquid smoke can produce better organoleptic quality than fish soaked for 10 minutes and 20 minutes. Seeing the potential of liquid smoke which has been widely used as a natural preservative, especially for fishery products, it is necessary to test the quality of liquid smoke from distillation and liquid smoke without distillation (without purification) and find the best time needed for soaking in liquid smoke. Therefore, the purpose of this study was to obtain the best type of liquid smoke and the optimum soaking time in liquid smoke in producing the best quality of smoked skipjack fillet.

2. MATERIALS AND METHODS

2.1. Materials and Equipment

The materials used in this research are: wood kuambi (*Schleicera oleosa*) for the manufacture of liquid smoke, fillet of fresh skipjack (*Katsuwonus pelamis* L) as much as 30 fillets with an average weight of 300-400 gr/fillet. The materials used for chemical

analysis are: H_2SO_4, NaOH 30%, H_3BO_3 2%, BCG+MM indicator, 0.05% HCl, selenium, agar, hexan, 70% and 90% alcohol, and distilled water. The equipment used in the research are: pyrolisator, distillation apparatus, coolbox, and microwave. While the equipment for chemical analysis of smoked skipjack tuna is analytical balance, erlemeyer, magnetic stirrer, measuring cup, petri dish, pipette, pH meter, incubator, autoclave, burette, oven (dry sterilization), spectrometer, thermometer, mortar and smoking oven.

2.2. Experiment Design and Treatment

The method used in this study is an experimental method designed in a factorial pattern with the basic design of Completely Randomized Design (CRD). The first treatment was liquid smoke from Kusambi wood with two types, namely: liquid smoke without distillation (A0) and liquid smoke from distillation (A1). The second factor is the soaking time in liquid smoke (L) with four levels, namely: 10 minutes (L1); 20 minutes (L2); 30 Minutes (L3); and 40 minutes (L4). In total there were 8 treatment combinations, each of which was repeated 3 times so that there were 24 experimental units.

2.3. Research procedure

This research was carried out from May 2021 to July 2021 at the Agricultural Product Technology laboratory, Kupang State Agricultural Polytechnic and the Chemical Laboratory of the Faculty of Science and Technology, Nusa Cendana University, Kupang. The research was conducted in two stages, namely smoke production and smoking application for fresh skipjack fillets.

The liquid smoke used in this study was made from Kusambi wood using the pyrolysis method according to the instructions from Darmadji (2020). Kusambi wood is cut into small pieces with sizes ranging from 3-5 cm, then burned in a pyrolysis tube using a burner at a temperature of 400 °C. The smoke flows through the pipe to the collection tube. The liquid smoke resulting from the pyrolysis is accommodated in a container and used as a liquid smoke treatment without distillation. In addition, to obtain liquid smoke, the smoke resulting from pyrolysis is carried out by distillation and purification by putting it into a distillation flask, and heating at a temperature of 100 °C to 150 °C. The liquid smoke produced from the distillation process is collected in a container or flask for use in the liquid smoke treatment. Furthermore, distilled and non -distilled liquid smoke was analyzed to determine its chemical composition.

In the treatment application stage, each type of liquid smoke (with and without distillation) was diluted by adding 10% fresh sap from lontar palm. Skipjack fillets that have been washed and drained are soaked in liquid smoke with a weight-to-volume ratio of 1:1, which means that 1 kg of fillet is soaked in 1 liter volume of liquid smoke. The soaking time was according to the soaking time treatment, namely: 10, 20, 30 and 40 minutes. The fillets that have been treated with soaking were then dried in an oven at 75 °C for 2 hours. After the oven, it was cooled and packaged in polyethylene plastic packaging with a thickness of 0.08 mm and stored at room temperature (28 °C) for 8 days to be observed.

2.4. Observation variable

The variables observed in this study included: the quality of liquid smoke (pH, phenol content and acid content); quality of smoked skipjack fillet which includes water content (%) using thermogravimetry (Darmadji and Yudiana, 2006), pH with a pH meter, total acid (%), phenol content (%) (Hadinoto and Yunianta, 2015), fat content (%) and protein content (%) by the spectrometer method.

2.5. Data analysis

The research data were first tested for normality and homogeneity tests. If the data is declared normal and homogeneous, then proceed with analysis of variance to test the value of differences between treatments. If there is a significant difference between treatments, then the Duncan multiple range test (DMRT) is continued at the level of = 5%. All these tests were carried out using the SPSS-25 application.

3. RESULTS AND DISCUSSION

3.1. Liquid Smoke Chemical Composition

The chemical composition of liquid smoke is a component that determines the quality of the liquid smoke produced because it acts as a preservative. The chemical composition of the liquid smoke analyzed (Table 1) was limited to pH, phenol content and acid content.

Table 1. Quality of liquid smoke from Kusambi wood

Smoke type	pH	Fenol content (%)	Acid content (%)
Without distillation	4.68	0,49	10,15
With distillation	3,18	0,61	17,62
SNI Standard	3,15-5,27	0,1-16	2-20%

The results of the analysis of the quality of the liquid smoke of kusambi wood (Table 1), show that there are differences in the quality of liquid smoke, but the quality of liquid smoke in this study is in accordance with SNI standards (Alianti *et al*., 2018). The average pH of liquid smoke from distillation of kuambi wood has a pH of 3.18 which is more acidic than the pH of liquid smoke without distillation which shows a pH of 4.68. Likewise, the phenol content and acid content, it is seen that the liquid smoke of Kusambi wood with the distillation process shows the phenol content (0.61%) and the acid content (17.62%) higher than the liquid smoke without distillation which has a phenol content and an acid content of approx. 0.49% and 10.15%. According to Alianti *et al*. (2018), phenol and organic acids function as antimicrobial substances in liquid smoke, and their role will increase if the two compounds are present together. The acidity of the liquid smoke is also influenced by the phenol content in the liquid smoke. The higher the phenol content, the more acidic liquid smoke will be (Bora & Tiri, 2021; Akbar *et al*., 2013).

3.2. Moisture Content of Smoked Fillets

The moisture content of smoked skipjack fillet is the moisture content observed during storage. This was done to determine the water content of each smoked skipjack fillet which received different soaking time and types of liquid smoke. Analysis of variance showed that there was a significant interaction effect ($P<0.05$) between the treatment duration of soaking and the type of liquid smoke on the moisture content of smoked skipjack fillet after 8 days of storage. The average moisture content of smoked skipjack fillet on the 8th day of observation is presented in Figure 1.

The SNI standard for water content for smoked fish is a maximum of 60% (BSN, 2013). Referring to the data in Figure 1, it can be seen that the soaking time of 30 and 40 minutes resulted in a moisture content of smoked skipjack fillet that met the SNI standard because it was below 60%. The treatment of soaking time of 30 minutes and

40 minutes in liquid smoke by distillation resulted in a water content of 48.46% and 49.67%, respectively, lower than the water content of smoked fish in the treatment of liquid smoke without distillation with the same soaking time, namely by 52.18% and 53.36%. Meanwhile, the treatment of soaking time of 10 minutes and 20 minutes in liquid smoke without distillation and with distillation did not show a significant difference, the average value of water content was above 61.75%-64.65% or exceeding the maximum standard required by SNI. This indicates that a long soaking time will affect the decrease in water content in the smoked skipjack fillet. On the other hand, the water content will increase if the soaking time in liquid smoke is shortened. The results of this study are in line with the research of Wally et al. (2015) which states that fish meat soaked in liquid smoke for 30 minutes will experience a decrease in water content due to the osmosis process, the amount of free water contained in fish meat will decrease due to the entry of smoked components. The increase in water content was caused by the increase in the total plate number (TPN) in smoked fish during storage. According to Mekarsari et al. (2017) and Tumonda et al. (2017), the water content of smoked fish increases due to microbial activity in fish which will produce water during the metabolic process during storage.

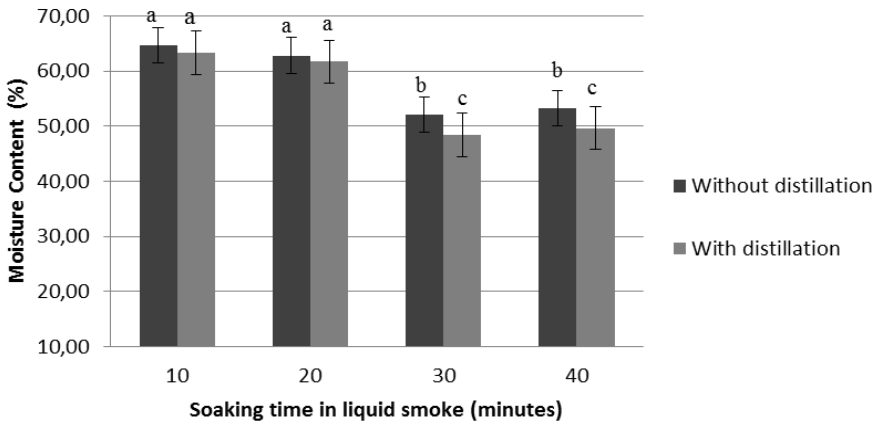

Figure 1. The effect of the combination of soaking time and type of liquid smoke on the average moisture content (%) of smoked skipjack fillet

3.3. pH of Smoked Skipjack Fillet

Analysis of variance showed that there was a significant interaction effect ($P<0.05$) between the treatment duration of soaking and the type of liquid smoke on the pH of the smoked skipjack fillet after 8 days of storage at room temperature. The average pH value of smoked skipjack fillet is presented in Figure 2.

The results of the analysis of the pH of the smoked skipjack fillet (Figure 2), showed that the lowest pH value was obtained at the treatment duration of soaking of 30 and 40 minutes in liquid smoke by distillation, respectively 4.04 and 3.96. This value was significantly different from the same soaking time in liquid smoke without distillation with pH values of 4.65 and 4.55. The highest pH of smoked skipjack fillet (between 4.83-5.35) was obtained in the treatment with 10 and 20 minutes of soaking in either distilled or undistilled liquid smoke. The decrease in pH is caused by the length of soaking, where the longer the soaking, the more liquid smoke solution will permeate. Also caused by the quality of the smoke, where the purified smoke has a higher acid content of 17.62% (Table 1) so that it affects the pH value in the skipjack filet material. This is in line with the opinion of Swastawati *et al*. (2013) and Tumonda

et al. (2017) which states that the difference in pH occurs due to the level or activity of lactic acid bacteria and the amount of organic acids contained in the smoke. Furthermore, Sirait & Suroto (2020) stated that the consequences of the smoking process can cause a decrease in water content, but also an increase in acid levels and the deposition of various smoked chemical compounds.

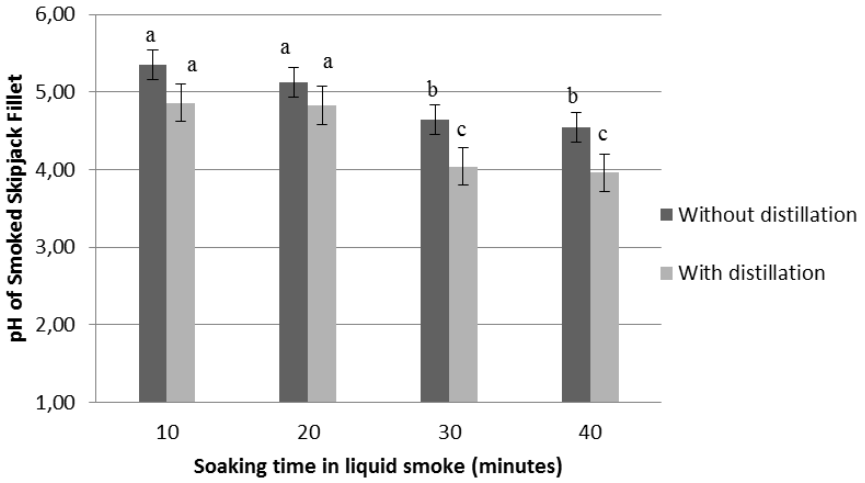

Figure 2. The effect of the combination of soaking time and type of liquid smoke on the average pH of smoked skipjack fillet

The increase in the pH of the smoked skipjack fillet during storage in the 10 and 20 minutes soaking treatment in liquid smoke without distillation was due to the activity of enzymes and bacteria starting to take place. Wally *et al*. (2015) and Alianti *et al*. (2018) stated that during storage there was a breakdown of proteins into basic compounds such as ammonia. The pH value of foodstuffs during storage can change due to the presence of proteins that are broken down by proteolytic enzymes and the help of bacteria into carboxylic acids, sulfide acids, ammonia and other types of acids.

3.4. Total Acid Smoked Skipjack Fillet

Analysis of variance showed that there was a significant interaction effect ($P<0.05$) between the treatment duration of soaking and the type of liquid smoke on the total acid of the smoked skipjack fillet during the storage period. The average value of the total acid of smoked skipjack fillet on the effect of soaking time in liquid smoke is presented in Figure 3.

Figure 3 shows that the total acid value is significantly different between the treatments of soaking duration in liquid smoke. The highest total acid value was obtained in the soaking time of 30 minutes and 40 minutes in liquid smoke with distillation of 8.84% and 9.07% and was not significantly different with those of the same soaking time in smoke without distillation with a total acid value of 7.48% and 8.27%, but was significantly different from other treatments. The total acid value of smoked skipjack fillet obtained from this study can be concluded that the soaking time of 30 minutes and 40 minutes and the distilled (purified) liquid smoke was significantly increased the total acid in the smoked skipjack fillet until the 8th day. The high total acid is due to the contribution of organic acid compounds containing lactic acid bacteria (LAB), so that it will play a role in converting sugar levels into pyruvic acid which is then hydrolyzed into organic acids and broken down into lactic acid which causes a sour taste (Hardianto & Yunianta, 2015; Bora & Gasong, 2021).

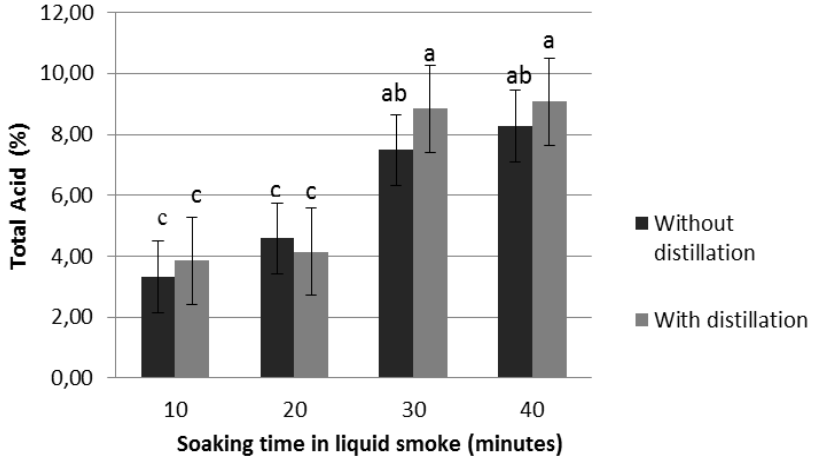

Figure 3. The effect of the combination of soaking time and type of liquid smoke on the total acid of smoked skipjack fillet

3.5. Smoked Skipjack Fillet Fat Content

Analysis of variance showed that there was an interaction effect between the treatment duration of soaking and the type of liquid smoke ($P<0.05$) on the fat content of the smoked skipjack fillet, as shown in Figure 4. From the two treatments, namely the duration of soaking with different types of liquid smoke, which were tested on fillets. Smoked skipjack tuna is known that the soaking time of 30 minutes and 40 minutes in distilled (purified) liquid smoke produces the best fat content of 4.85% and 4.92% and tends not to be significantly different from the soaking time of 20 minutes in the same liquid smoke (4.65%), but higher than those of other treatments.

The high fat content in liquid smoked skipjack fillet (4.85% and 4.92%) occurs because the distilled liquid smoke is more effective containing higher levels of phenol and acid (Table 1), so that it is absorbed more by the skipjack fillet through soaking. long (30 minutes and 40 minutes). This will affect the number of microbes in fish fillets which will slow down protein decomposition.

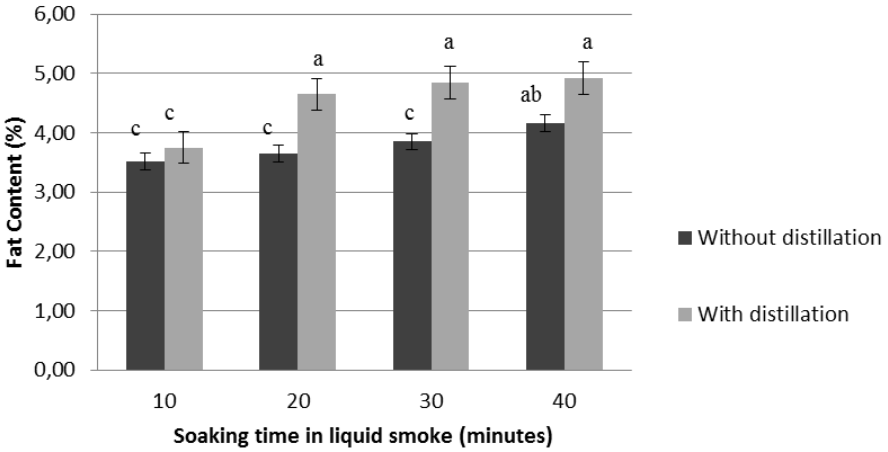

Figure 4. The effect of the combination of soaking time and type of liquid smoke on the fat content of smoked skipjack fillet

According to (Ayudiarti & Sari, 2010) there are two main groups of compounds in liquid smoke that are known to have bactericidal or bacteriostatic effects, namely phenol derivatives and organic acids. Phenol derivatives and organic acids in liquid smoke may play a role in inhibiting the decay process by suppressing the growth of existing spoilage bacteria (Hartono *et al*., 2016).

3.6. Smoked Skipjack Fillet Protein Content

Measurement of protein levels is important because protein is a very important food substance for the body. The source of protein contained in skipjack fillet comes from skipjack tuna as a raw material. Analysis of variance showed that there was a significant interaction effect ($P<0.05$) between the treatment duration of soaking and the type of liquid smoke on the protein content of smoked skipjack fillet. The average protein content of smoked skipjack fillet (calculated based on overall weight or wet basis) is presented in Figure 5. From the two treatments, namely the duration of soaking and the type of smoke tested, it was found that the treatment of soaking time of 20 minutes, 30 minutes and 40 minutes in distilled liquid smoke had higher protein content, namely 17.65%, 18.04% and 17, respectively. 87% and significantly different from those other treatments which showed lower protein content. The high percentage of smoked skipjack fillet protein content after 8 days of storage was due to the low percentage of water content. Protein content has a close relationship with the percentage of water content of a material. The lower the percentage of water content in an ingredient will result in the percentage of protein increasing. This is in accordance with Alinti (2018), who stated that water loss will cause the percentage of protein and fat to increase. The decrease in water content in the oven process can increase the protein content in processed products. The lower the water content in the product, the higher the protein content in fish. The results of this study are in line with the research of Nusaibah *et al*. (2014) which showed that the protein content of smoked fish using the liquid smoke method was 2.5% higher than those of direct smoking. This can be seen from the smoked skipjack fillet using distilled liquid smoke method which has a drier texture than that of fillets treated in smoke without distillation. This relates to the quality of liquid smoke, where distilled liquid smoke has a better quality than liquid smoke without distillation.

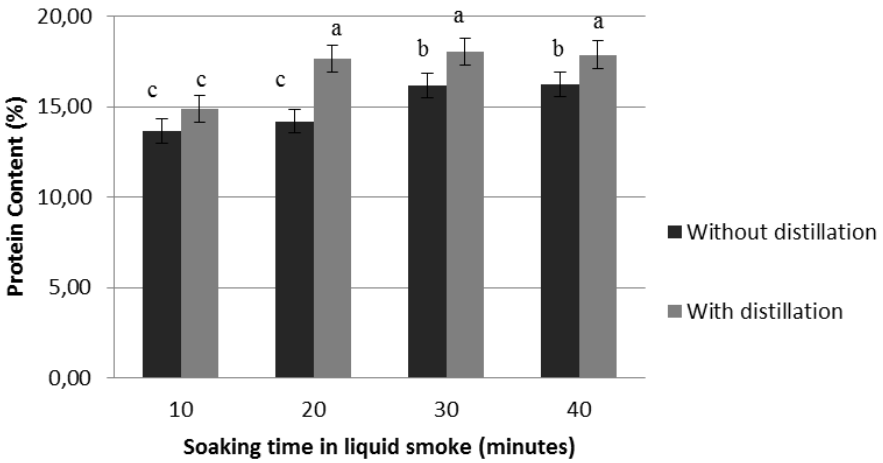

Figure 5. The effect of the combination of soaking time and type of liquid smoke on the protein content of smoked skipjack fillet

3.6. Phenol Content of Smoked Skipjack Fillet

Phenol is one component of smoke that is very influential on the durability that is safe for consumption. Analysis of variance showed that there was a significant interaction effect ($P<0.05$) between the treatment duration of soaking and the type of liquid smoke on the phenol content of the smoked skipjack fillet, as shown in Figure 6.

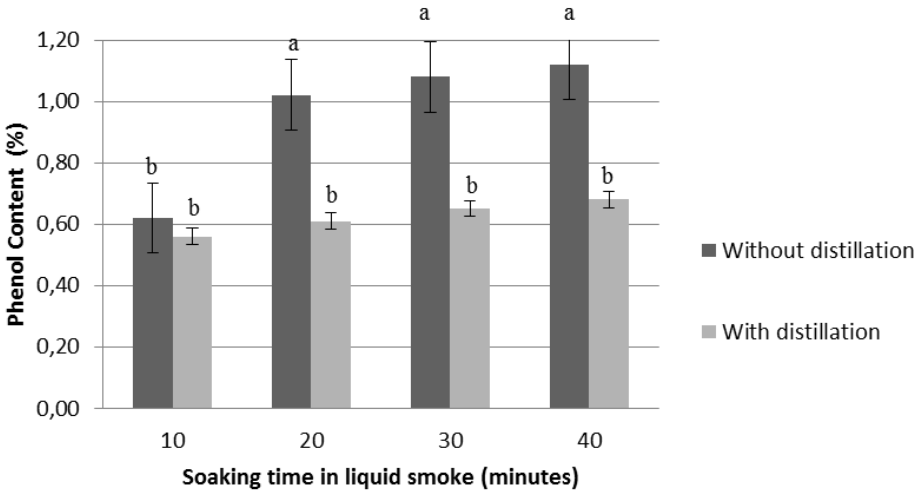

Figure 6. The effect of the combination of soaking time and type of liquid smoke on the fenol content of smoked skipjack fillet

Based on Figure 6, it can be seen that the average value of phenol content between the soaking time and types of liquid smoke is significantly different. The lowest average phenol content was obtained in the treatment duration of soaking (40, 30, 20 and 10 minutes) in the type of liquid smoke by distillation, respectively 0.68%, 0.65%, 0.61% and 0 ,56% and not significantly different from the treatment of 10 minutes of soaking in liquid smoke without distillation (0.62%). However, the treatment of soaking time of 20, 30 and 40 minutes in liquid smoke without distillation showed higher phenol content, namely 1.02%, 1.08% and 1.12%, respectively. Based on the analysis data on the phenol content (Figure 6), it can be concluded that the use of liquid smoke from Kusambi wood with distillation and soaking time of 20, 30 and 40 minutes has the lowest phenol content and is still within safe limits for consumption. The safe limit of phenol levels for consumption is 0.02 - 1.00% (Mardyaningsih et al. (2016)

4. CONCLUSIONS AND SUGGESTIONS

The use of liquid smoke in fumigation is an effective solution to produce quality and uniform products. In general, the quality of the liquid smoke of the Kusambi wood in this study is in accordance with the SNI standard. The treatment duration of soaking and the type of liquid smoke of Kusambi wood gave a significant interaction effect on the quality of liquid smoked skipjack fillet after 8 days of storage at room temperature. The results showed that based on the quality of liquid smoked skipjack fillet after 8 days of storage, it was known that the treatment of soaking 30 minutes and 40 minutes in distilled liquid smoke was the best treatment and met the quality requirements of SNI. This is based on the value of water content (48.46% and 49.67%), pH (4.04 and 3.96), total acid (8.84% and 9.07%, fat content (4.85%) and 4.92%), protein content (18.04% and 17.83%) and phenol content in smoked skipjack fillet (0.65% and 0.61%).

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