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Characteristics of *Julung Julung* Smoked Fish Fillets (*Hemiramphus* sp.) using Liquid Smoke from Corn Cobs Waste

Abstract

Corn cobs can be utilized as a shell ingredient for liquid smoke. Liquid smoke can be used for the smoked fish industry in North Sulawesi, Indonesia. However, the utilization of liquid smoke for the smoked fish industry has yet to be optimal. This study aimed to obtain the best smoking method for *julung julung* fish fillets (*Hemiramphus* sp.) using liquid smoke from corn cob waste. The treatment in this study was to apply liquid smoke from corn cob waste in the smoking process, which was compared with the conventional method (using shell, coconut wood, and corn cob). Parameters to determine the quality of smoked fish were total volatile bases (TVB), moisture, water activity, pH, phenol levels, polycyclic aromatic hydrocarbon (PAH), and sensory assessment. The results showed that the TVB of smoked *julung julung* fish fillets ranged from 19.83 – 32.27 mg N/100g. The moisture ranged from 14.10 – 30.57%. Water activity ranged from 0.46 – 0.82. pH ranged from 4.60 – 5.81. Phenol levels ranged from 4.42 – 16.11 mg/g. PAH levels are still below the standard required in the Indonesian National Standard. Panelists rated neutral to really like the appearance, aroma, taste, and texture of smoked fish. From these results, it can be concluded that the best treatment is fresh fillets preheated in a cabinet dryer for 4 hours at 60 – 80 ^{o}C and then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated in a cabinet dryer for 4 hours at 90 ^{o}C.

Keywords: Coconut, Indonesia, Shell, North Sulawesi, Wood

Introduction

Corn is the second most widely grown crop in Indonesia after rice. Indonesia is the 7th corn producer in the world, with more than 12 million tons of corn produced in 2020 (Nazmi *et al*., 2021). North Sulawesi is one of the contributing regions that produce corn in Indonesia. Considerable corn production will also produce waste, especially corn cobs, which are generally thrown away and burned by most Indonesians (Cahyadi *et al*., 2021).

One of the corn cobs was used to make liquid smoke. Previous research reported that corn cobs can produce liquid smoke, a by-product of the pyrolysis of corn cob waste. The yield of liquid smoke from corn cobs is about 28.37%, with a pH value of 3.5 (Sriharti *et al*., 2020). The particle size of corn cobs affects the yield of liquid smoke, with smaller particle sizes resulting in higher yields (Aladin *et al*., 2018). Another study reported a phenolic content of 335 mg/L in liquid smoke produced from corn cobs (Swastawati *et al*., 2007).

Liquid smoke is a natural product made from the condensation of smoke from burning wood (Andy *et al*., 2021). Liquid smoke is commonly used as a flavoring in food to provide a smoked flavor without the food undergoing the actual smoking process (Sari *et al*., 2006). Using liquid smoke in food will save time, energy, and labor and reduce production costs (Krah *et al*., 2019). In addition, using liquid smoke in food can speed up and standardize the smoking process, adding flavor and microbiological safety

while reducing production time (Indiarto *et al*., 2020). Generally, various types of hardwood were used to manufacture liquid smoke (Diatmika *et al*., 2019). The quality of the liquid smoke produced may vary depending on the type of wood used (Budaraga *et al*., 2016). In addition to imparting specific flavors and aromas to foods (Maulina *et al*., 2020), liquid smoke is reported to be antimicrobial (Septana *et al*., 2020) and antioxidant (Budaraga & Putra, 2021).

The conventional smoking processes generally use hardwood as fuel, such as melon wood (Umar *et al*., 2018), mangrove wood (Cissoko *et al*., 2020), teak wood (Daramola *et al*., 2020), coconut shells, and coconut husks (Nugroho *et al*., 2018). Corn cobs have also been reported to be used in the fish smoking process (Sukowati, 2023). According to literature, traditional fish smoking in North Sulawesi, Indonesia, uses smoke from burning coconut shells or wood charcoal (Landangkasiang *et al*., 2017; Primalasari *et al*., 2019; Saediman *et al*., 2021). Smoked fish processing has been known for a long time because of its traditional processing techniques, simplicity, ease of implementation, and low cost (Islamiyah, 2021). In North Sulawesi, Indonesia, smoked fish commonly known as *fufu* is traditionally made from skipjack (*Katsuwonus pelamis*); *julung julung* (*Hemiramphus far*) (Berhimpon *et al*., 2018); and scad fish (*Decapterus* spp.) known as *pinekuhe* (Ansar & Ijong, 2021). The smoked fish product of *julung julung* is a typical product from North Sulawesi and Gorontalo, commonly called *roa* or *galavea* (Azis & Akolo, 2020).

The study's reported that the conventional smoking process has disadvantages such as smoking time, concentration of carcinogenic substances, temperature, and inconsistent product quality (Racovita *et al*., 2020). Conventional smoking of food products has been shown to produce carcinogenic components such as polycyclic aromatic hydrocarbons (Jinadasa *et al*., 2020). Benzo(a)pyrene ($C_{20}H_{12}$) is one of the carcinogenic compounds produced from traditional smoking products (Jinadasa *et al*., 2020). One method to reduce carcinogenic compounds in smoked fish products is to use liquid smoke in the smoking process (Nithin *et al*., 2020; Xin *et al*., 2021).

The utilization of liquid smoke in the fish smoking industry in North Sulawesi is very likely to be developed. North Sulawesi was reported to have both large-scale and domestic fish-smoking industries (Dotulong *et al*., 2018; Primalasari *et al*., 2019). Making liquid smoke is relatively simple, so it will be readily accepted by fish-smoking industry players (Ali & Al Fiqri, 2020). Previous research reported that using liquid smoke in smoked fish products resulted in good quality in terms of appearance, flavor, and aroma (Rizal *et al*., 2020). In addition, using liquid smoke in smoked fish products can add nutritional value and durability, making it possible to reach a wider market area (Ali *et al*., 2021).

Previous research reported that smoked fish dipped in liquid smoke for 15 minutes had a total plate count value of 4.7×10^4 CFU/g on day six and moisture content below 60% during storage (Suroso *et al*., 2018). Dipping fish in liquid smoke with a concentration of 15% and a dipping time of 60 minutes showed a bacterial colony of 2.12×10^2 CFU/g (Ali *et al*., 2021). The Indonesian National Standard on

smoked fish requires a maximum total plate count of 5×10^4 CFU/g and a maximum moisture content of 60% (Indonesia Standardization Agency, 2013). Another study reported that smoked fish processed with liquid smoke had a moisture content of 47.63%, phenol content of 12.62%, and pH of 4.8 (Berhimpon *et al*., 2018). The results of these studies indicate that liquid smoke is good to apply to smoked fish products. This study characterized smoked fish fillets of *julung julung* (*Hemiramphus* sp.) produced with liquid smoke from corn cobs waste. The study was conducted by dipping the fish fillets of *julung julung* with liquid smoke, with drying and steaming treatments on the fish fillets. This study aims to obtain the best smoking method for fish fillets using liquid smoke from corn cobs waste based on the assessment of total volatile bases, moisture content, water activity, pH value, phenol content, polycyclic aromatic hydrocarbon content, and sensory assessment.

Materials and methods

The main materials of the study were *julung julung* fish (*Hemiramphus* sp.), corn cob (*Zea mays* L.) from Gangga Island in Likupang, North Sulawesi, Indonesia. This study includes the preparation of liquid smoke from corn cobs waste, preparing fish (weeding and filleting), and treating fish fillets by dip in liquid smoke. Fresh *julung julung* fish was obtained from traditional fishermen in Manado, North Sulawesi, Indonesia (Fig. 1). The fish was placed in a cool box and given ice in a ratio of 1:1 (ice:fish) (Mishra *et al*., 2021). Then, the fish was washed, weeded, and filleted. The cleaned fillets were dipped in a 5% salt solution for 30 minutes. The concentration of liquid smoke from corn cob waste was 0.8% (Berhimpon *et al*., 2018).

Fig. 1. *Julung julung* (*Hemiramphus* sp.)

The treatment in this study was applying liquid smoke from corn cobs waste to the smoking process of *julung julung* fish fillets, which was compared with the conventional method (using coconut wood, coconut shells, and corn cobs).

(A) : Fresh fillets were dipped in liquid smoke for 20 minutes and then heated in a cabinet dryer for 4 hours at 90 ºC.

(B) : Fresh fillets pre-heated in a cabinet dryer for 4 hours at 60 – 80 ºC, and then dipped in liquid smoke for 20 minutes. After that, fillets were re-heated in a cabinet dryer for 4 hours at 90 ºC.

(C) : Fresh fillets were steamed for 30 minutes and then dipped in liquid smoke for 20 minutes. After that, fillets were heated in a cabinet dryer for 4 hours at 90 ºC.

(D) : Fresh fillets were smoked with the conventional method using wood and coconut shell for 6 hours at 90 ºC.

(E) : Fresh fillets were smoked with the conventional method using corncob for 6 hours at 90 ºC.

Liquid smoke manufacturing process

Making liquid smoke refers to modified research (Aladin *et al*., 2018). Modifications were made to the equipment used in the pyrolysis process. Dry corn cobs were weighed to determine fuel efficiency. Corn cobs were gradually burned into the combustion furnace. The furnace was closed to prevent smoke from escaping from the tank. The smoke generated from the combustion flows through a pipe connected to a storage tank covered with ice cubes. The smoke that passes through the pipe will become cold, so condensation occurs, turning the smoke into liquid. The smoke that has been formed is collected into a container attached to the end of the pipe. The liquid smoke obtained is then allowed to settle the tar formed for three weeks and filtered to obtain clear liquid smoke.

Total volatile bases assay

Total volatile base (TVB) assay refers to research by Tambunan & Chamidah (2021). TVB assay aims to determine the amount of volatile base compounds formed from protein breakdown—the principle of TVB analysis, namely by evaporating volatile base compounds at room temperature for 24 hours. The compound is then bound with boric acid and titrated with an HCl solution. After the boric acid solution was stored in the inner chamber of the Conway cell containing the blank (V_0) and the filtrate (V_1), the boric acid solution was titrated with 0.02 N HCl so that the boric acid solution changed color to pink. TVB levels in the smoked fish meat were expressed as mg N/100g.

$$TVB\left(\mathrm{mg}\frac{\mathrm{N}}{\mathrm{100g}}\right) = \frac{(V_1 - V_0) \times N.HCl \times dilution \times 100}{sample\ weight\ (g)}$$

Moisture content assay

The moisture content by method of Indonesia National Standard (Indonesia Standardization Agency, 2006a). An empty porcelain cup is weighed (A). Then, the sample was weighed to ± 2 g in a porcelain cup of known mass (B). The samples were dried in an oven at 105°C for 20 hours. The samples were then cooled in a desiccator, after which the samples in the cup were weighed (C). Moisture content was expressed as %.

$$Moisture\ (\%) = \frac{B\ (g) - C\ (g)}{B(g) - A\ (g)} \times 100\%$$

Water activity (Aw) assay

Water activity assay used an Aw meter (Rotronic, HygroPalm 23-AW-A). The Aw meter was set up at room temperature for 2 hours. The water activity of smoked fish was measured by placing the sample into a sample container and conditioning it for 30-60 minutes. The Aw meter sensor is contacted with the sample in the container. Then the water activity (Aw) value can be read on the Aw meter panel (Saputra *et al*., 2014).

pH assay

The sample weighed as much as 30 g, then 400 mL of distilled water was added at 28 ºC. The sample was homogenized with a magnetic stirrer to be evenly stirred. The pH meter electrode was inserted into the sample solution. The data taken in this test is the pH value read by the pH meter (Hanna Instruments HI99192) (Lekahena & Jamin, 2018).

Phenol level assay

Samples were extracted by adding 1 mL ethanol p.a., 5 mL distilled water, and 0.5 mL Folin Ciocalteu 50%, homogenized, and allowed to stand for 5 minutes. Then added, 1 mL of 5% Na_2CO_3 and left in the dark for ± 60 minutes with gallic acid as a standard. The absorbance value was read on a spectrophotometer (Thermo Scientific Genesys 50) with a wavelength of 725 nm. Total phenolics were expressed as mg/g (Indonesia Standardization Agency, 2004; Muliadin *et al*., 2022).

$$Total\ Phenol\ (\frac{mg}{g}) = \frac{Gallic\ acid\ equivalence\ \left(\frac{mg}{L}\right) \times Volume\ (L)}{Sample\ weight\ (g)}$$

Polycyclic aromatic hydrocarbon (PAH) assay

PAH content testing was carried out based on the instructions Basak *et al*. (2010). A 5 g sample was weighed and collected in a 100 ml flask. Then 5 mL of 50% KOH solution, 75 mL of methanol, 1 mL

of internal standard solution (9,10 dimethylantracene), and some boiling stones were added. After that, the mixture was boiled for 4 hours in a soxhelet. The liquid phase was transferred to a separatory funnel and extracted with 100 mL of n-hexane with shaking for 3 min. The MeOH:KOH phase was drained and discarded. The combined n-hexane phase was rinsed twice, each with 50 mL of H_2O-MeOH (8:1) and 50 mL of water. The methanol-water and water phases were dried and discarded. The organic phase was concentrated in a rotary evaporator (40°C) to a volume of about 10 mL. The concentrated extract was dried under nitrogen gas.

After that, the silica gel column was cleaned by dissolving 15 g of silica gel in n-hexane and transferred to a column (200 mm long; diameter: 12 mm). Then, 1 g of anhydrous sodium sulfate was added. The concentrated sample extract was transferred to the column and eluted with 1 mL of n-hexane. A stock solution containing 0.1 mg mL^{-1} of 9,10 dimethylantracene was dissolved in n-hexane and stored at 4°C in a volumetric flask (with a glass stopper) wrapped in aluminum foil to avoid possible light degradation. Working standard solutions were prepared from the stock solutions. Analysis of PAH compounds used a Hewlett-Packard 1100 HPLC equipped with an Agilent-110 fluorescence detector. The injection volume was 10 µL. Mobile phase gradient (acetonitrile-water): 80% acetonitrile + 20% water with a flow rate of 1 ml min^{-1} and a wavelength of 270 nm. The external standard mixture was used for PAH concentration calculation.

Sensory assessment assay

Sensory assessment assay refers to Indonesia National Standard (Indonesia Standardization Agency, 2006b). The sensory assessment was based on a hedonic scale (Table 1). Smoked fish samples were placed on a plastic plate with a glass of water, coded, and presented to 30 panelists randomly under light. The parameters observed were appearance, aroma, taste, and texture. Panelists rated acceptance of the samples on a scale of 1 – 9.

Table 1. Score sheet of hedonic assessment

Specification	Score
Really like it	9
Really like	8
Like	7
Rather like	6
Neutral	5
Rather dislike	4
do not like	3
Very dislike	2
Really don't like it	1

Data analysis

Data analysis was performed using Statistical Product and Service Solutions (SPSS) version 20.0. Statistical analysis of data was performed based on one-way analysis of variance (ANOVA) using a significance level of ($P<0.05$). Specific group differences were determined using Duncan's test.

Results and discussion

Liquid smoke production was carried out using a simple liquid smoke distillation device. This process consumed 17 kg of corn cobs and 12 kg of crushed ice. Liquid smoke from corn cobs is more accessible than coconut shells or wood (Maulina & Karo, 2021). Using coconut shells or wood as fuel will form a flame, thus reducing the volume of smoke in the furnace (Kabir Ahmad *et al*., 2022). The smoked *julung julung* were analyzed for TVB, moisture content, water activity, pH value, phenol content, PAH content, and sensory assessment.

Total volatile bases

The total volatile base (TVB) value is measured to determine the quality of the smoked fish. TVB value is one of the parameters used to determine the decline in fish quality and measures the amount of volatile base compounds formed due to protein degradation (Castro *et al*., 2006). Volatile bases formed in fish muscle tissue mainly consist of ammonia, trimethylamine (TMA), and dimethylamine (DMA) (VELP Scientifica, 2013). TVB value is an essential characteristic for the quality assessment of seafood products and is emerging as the most commonly used chemical parameter to assess the palatability of seafood (Rasulu *et al*., 2020). The results of *total volatile bases* on smoked fish fillets of *julung julung* are presented in Fig. 2.

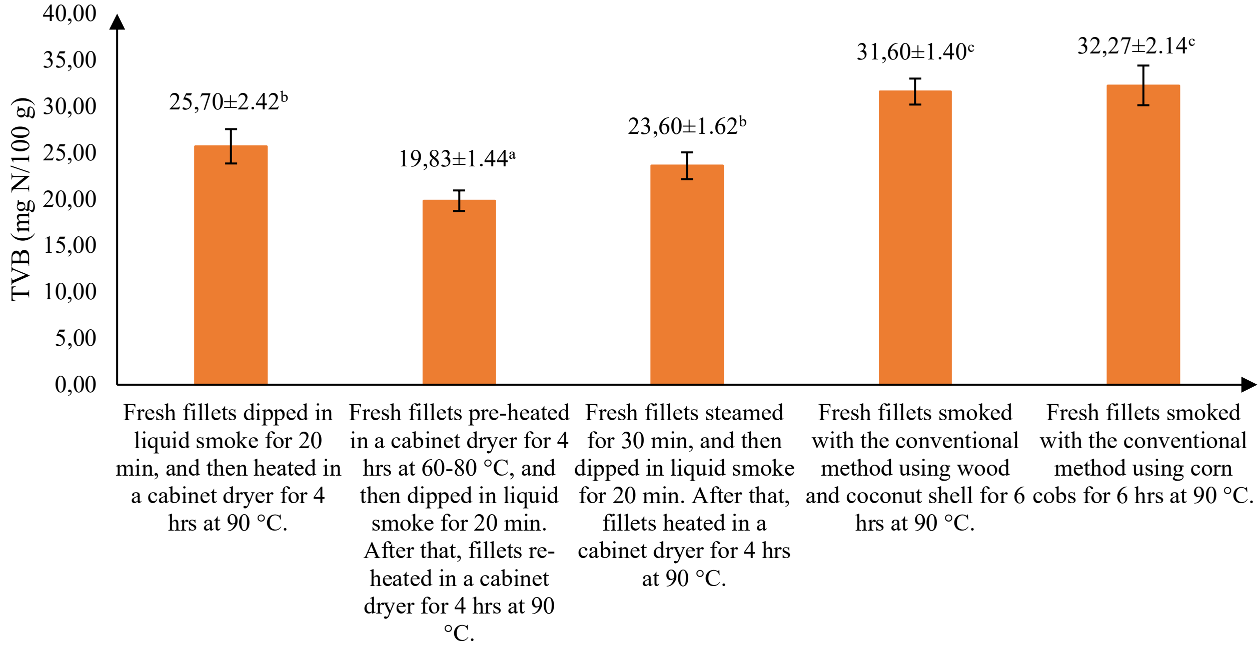

Fig. 2. Total volatile bases of *julung julung* smoked fillet

Fig. 2 shows the analysis of the variance of smoked fish fillets with different smoking method treatments affecting changes in TVB content ($p<0.05$). The TVB content of smoked fish fillets ranged from 19.83 – 32.27 mg N/100g, with the lowest TVB value in treatment B. The treatment of the smoking method with liquid smoke from corn cob waste (treatments A, B, and C) had lower TVB levels when compared to the conventional smoking method (treatments D and E). A literature search has not found why liquid smoking has a lower TVB value than conventional smoking methods. However, one possibility is that the liquid smoke smoking method allows the smoke components to be absorbed into the fish meat better than the conventional smoking method. Better absorption of liquid smoke phenol components in fish meat can inhibit and control microbial growth that causes a decrease in the quality of smoked fish (Santoso *et al*., 2015). A literature study showed that the average TVB value of smoked mackerel (*Euthynnus affinis*) treated with liquid smoke addition ranged from 24.63 – 28.38 mg N/100g (Hardianto & Yunianta, 2015). Another study reported smoked mackerel with a smoking process using rubber wood liquid smoke, producing smoked fish products with lower TVB values (Suroso *et al*., 2018). Duncan's analysis showed a difference between treatments B, A – C, and D – E on the TVB value of smoked fish fillets. The analysis of variance indicates that the drying process of smoked fish fillets before immersion into liquid smoke can maintain the quality of smoked fish (Kaparang *et al*., 2013). Duncan's analysis also showed that treatments A – C and D – E had no difference in TVB value, indicating that the moisture content between the two treatments was not different. TVB value is closely related to moisture content (Rasulu *et al*., 2020).

The European standard (European Market Observatory for Fisheries and Aquaculture, 2020) states that the limit range of TVB value for smoked fish is 25 – 35 mg N/100 g. All treatments' TVB value of the smoked fish fillets still met the European standard. Previous studies reported TVB values of fresh fish fillets of 8.70 ± 0.86 mgN/100g (Moosavi-Nasab *et al*., 2021) and 12.94 ± 0.92 mg N/100g (Bouzgarrou *et al*., 2020). Messina *et al*. (2021) reported that smoked fish fillets that underwent two drying processes had a TVB value of <20 mg N/100g. Another study also reported the TVB value of smoked fish fillet samples with hot smoking and cold smoking, which amounted to 17.80 ± 0.17 and 18.95 ± 0.20 mg N/100g, respectively (El-Lahamy *et al*., 2019). Aydin *et al*. (2020) reported a TVB value of smoked fish with hot smoking of 11.82 ± 0.05 mg N/100g.

Moisture content

Moisture content is an important parameter to determine the quality of smoked fish produced. The moisture content contained in smoked fish can affect the shelf life of smoked fish because water in food is a medium for microbes to grow (Kaban *et al*., 2019). The smoking process was reported to reduce the moisture content of fresh fish to a range between 9 and 17% (Sikoki & Aminigo, 2002). The moisture content of smoked fish fillets is presented in Fig. 3.

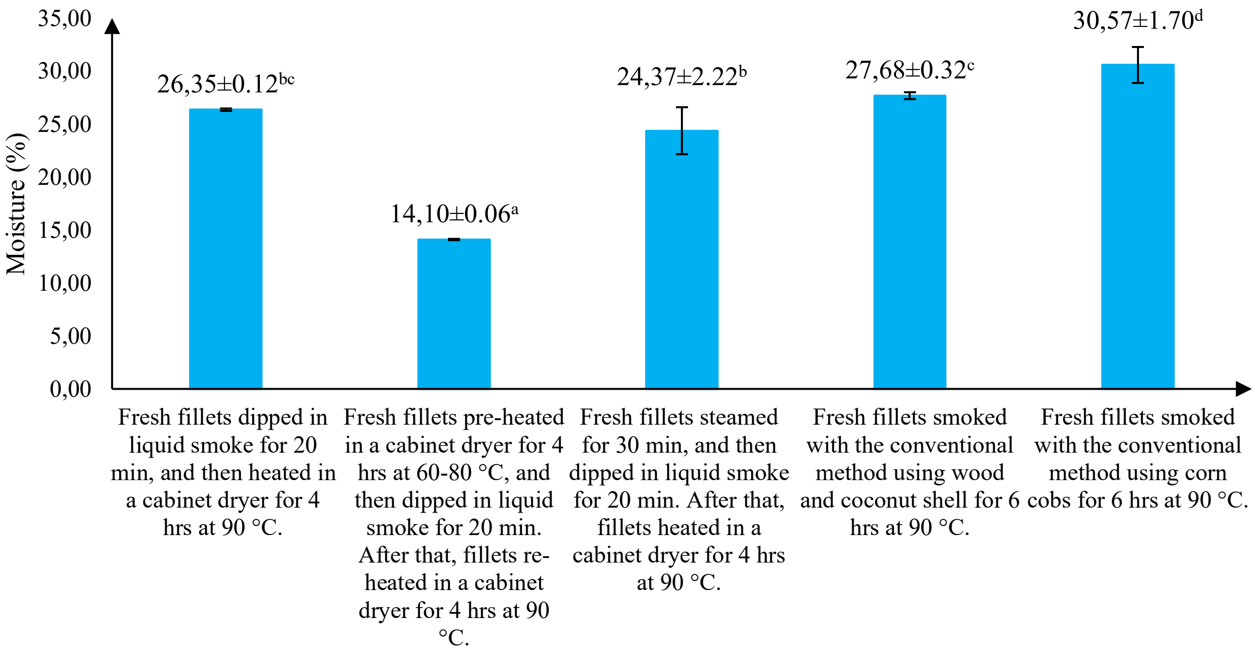

Fig. 3. Moisture content of *julung julung* smoked fillet

Fig. 3 shows the analysis of the variance of smoked *julung julung* fish fillets with different smoking methods treatment affects moisture content ($p<0.05$). The moisture content of smoked *julung julung* fish fillets ranged from 14.10 – 30.57%, with the lowest moisture content in treatment B. The

moisture content value in smoked fish products from all treatments still meets the Indonesian National Standard No. 2725:2013 on smoked fish, which is a maximum of 60% (Indonesia Standardization Agency, 2013). Darianto *et al*. (2018) reported that the smoking process can reduce the moisture content of fish to below 40%, which can help preserve it longer. The treatment of the smoking method with liquid smoke with corn cob (Treatment A, B, C) has a lower moisture content when compared to the treatment of the conventional smoking method (Treatment D and E). This result is because the smoking chamber is not fully enclosed in the conventional smoking method, so the heat generated could be more optimal. Suboptimal heat can increase moisture content and cause the moisture content of smoked fish to decrease only slightly (Amos & Paulina, 2017). Whereas in the liquid smoke smoking method, temperature and humidity can be controlled better so that the moisture content of the product can be reduced efficiently (Salindeho & Lumoindong, 2017).

Duncan's analysis showed a difference between treatment B, treatment A – C, and treatment D – E on the moisture content of smoked fish fillets. The results of the variance study indicate that the drying process of smoked fish fillets before the dip in liquid smoke in treatment B can help reduce the moisture content. This study's results are from previous research, which also reported a significant decrease in moisture content in smoked fish products with two times drying treatment (Messina *et al*., 2021). Duncan's analysis also showed that treatments A and C were not different because steaming in treatment C will increase the moisture content of the product (Salmatia *et al*., 2020). The conventional smoking method with corn cob fuel has the highest moisture content; this result is the conventional smoking process with corn cobs, and the heat generated is lower than smoking using wood (Asmara *et al*., 2022).

Water activity (Aw)

Water activity is one of the essential parameters in the quality of smoked fish. Water activity (Aw) is expressed as the ratio of the vapor pressure in the food to the vapor pressure of pure water, and it predicts whether water tends to move from the food product into the cells of microorganisms that may be present. A well-smoked fish has a water activity of <0.50 and a moisture content between 15 and 25% to inhibit the growth of pathogenic microorganisms in smoked fish products (Mondo *et al*., 2020). According to British Columbia Centre for Disease Control (2013), the maximum water activity value for smoked fish is 0.97. Water activity correlates with the moisture content of a smoked fish product and is two important factors affecting food safety and quality (Fitri *et al*., 2022). The water activity values of smoked fish fillets can be seen in Fig. 4.

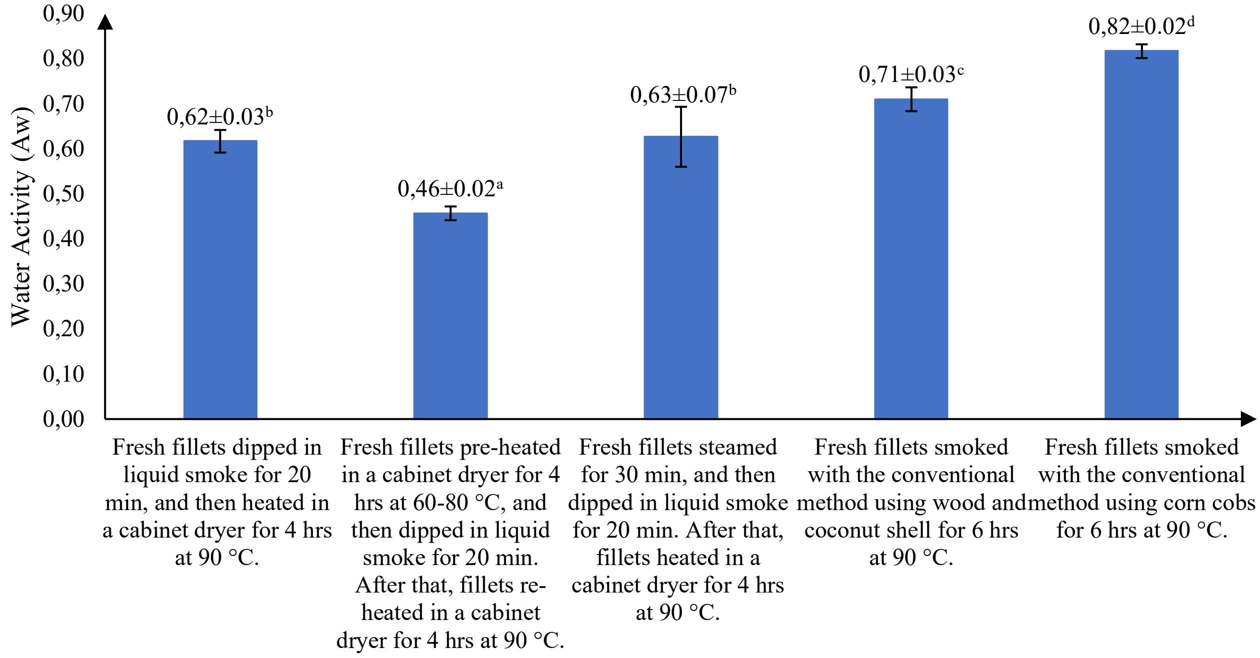

Fig. 4. Water activity of *julung julung* smoked fillet

Fig. 4 shows the analysis of the variance of smoked fish fillet *julung julung* with different smoking method treatments affecting water activity ($p<0.05$). The water activity value of smoked fish fillet ranged from 0.46 – 0.82, with the lowest water activity in treatment B. The treatment of the smoking method with corn cob liquid smoke (Treatment A, B, C) had a lower water content when compared to the treatment of the conventional smoking method (treatment D and E).

The water activity value of fish meat can vary depending on the cooking method, temperature, and duration of heating (Oliveira *et al*., 2017; Zhang *et al*., 2023). Therefore, the factor of fish fillet treatment before immersion in liquid smoke caused the difference in water activity value in treatment B compared to treatments A and C. The water activity value is directly proportional to the water content value of each treatment. The heating process can reduce the moisture content of fish meat (Kiczorowska *et al*., 2019), thus affecting the water activity of fish meat (Gómez *et al*., 2020).

pH

The pH value of smoked fish is an essential factor affecting its quality. The pH value will decrease with increasing smoking time (Baten *et al*., 2020b). The analysis of the variance of smoked fish fillets *julung julung* with different smoking method treatments affects the pH value ($p<0.05$). The pH of smoked fish fillet ranged from 4.60 to 5.81 (Fig. 5). The treatment of the smoking method with corn cob liquid smoke (treatments A, B, and C) had a lower pH value when compared to the conventional smoking method (treatments D and E). Swastawati *et al*. (2022) also reported that the pH value of smoked barracuda fish with the liquid smoke method was significantly lower than that of the conventional

method. Another study also reported that using liquid smoke can reduce pH caused by the condensation of organic acids in the smoking process (Puke & Galoburda, 2020). The pH value of smoked fish correlates with the phenol content contained in the smoke component, and an increase in phenol content causes a decrease in pH (Berhimpon *et al*., 2018).

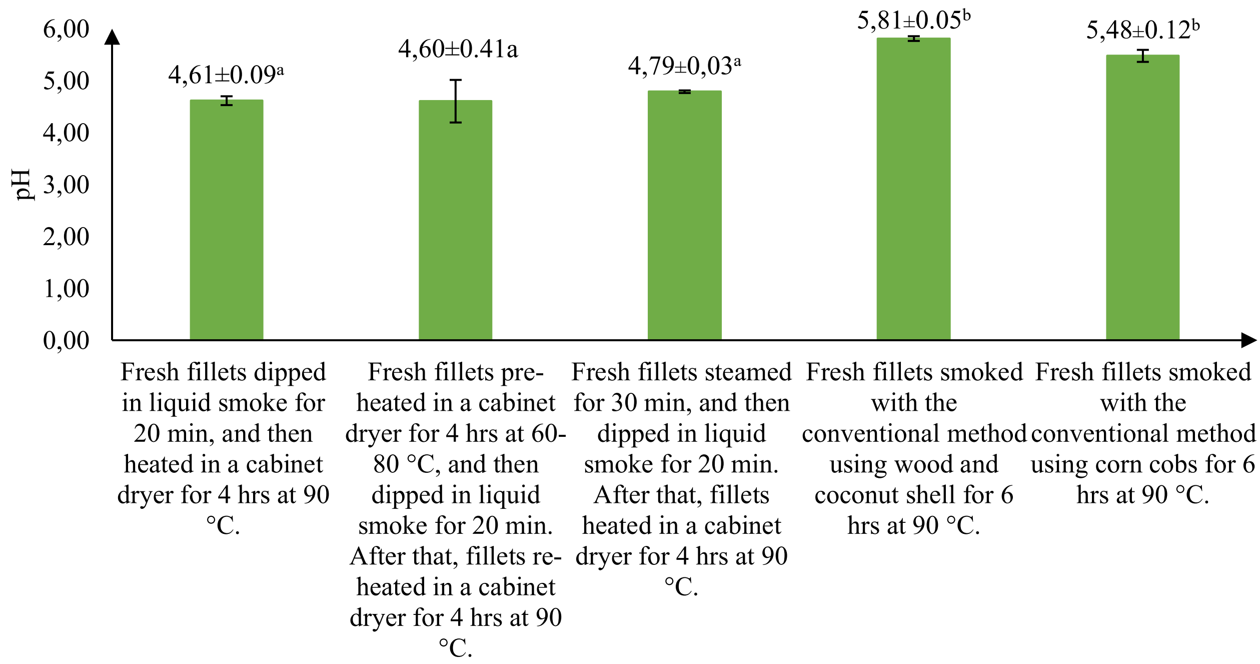

Fig. 5. pH of *julung julung* smoked fillet

Phenol level

Phenol is a compound found in wood smoke. Phenol compounds enter food through diffusion and capillary action, affecting taste, color, and aroma and extending shelf life (Remy *et al*., 2016). In addition, phenol compounds are also reported to act as antioxidants that can prevent rancidity in fish meat (Sérot *et al*., 2004). Leksono *et al*. (2020) also noted that the higher the phenol level in smoke, the stronger the flavor and aroma of smoked fish. The analysis of the variance of smoked fish fillets julung julung with different smoking method treatments affecting phenol level ($p<0.05$). The phenol level of smoked fish fillet ranged from 4.42 to 16.11 mg/g (Fig. 6). The treatment of the smoking method with corn cob liquid smoke (treatments A, B, and C) had higher phenol level when compared to the conventional smoking method (treatments D and E).

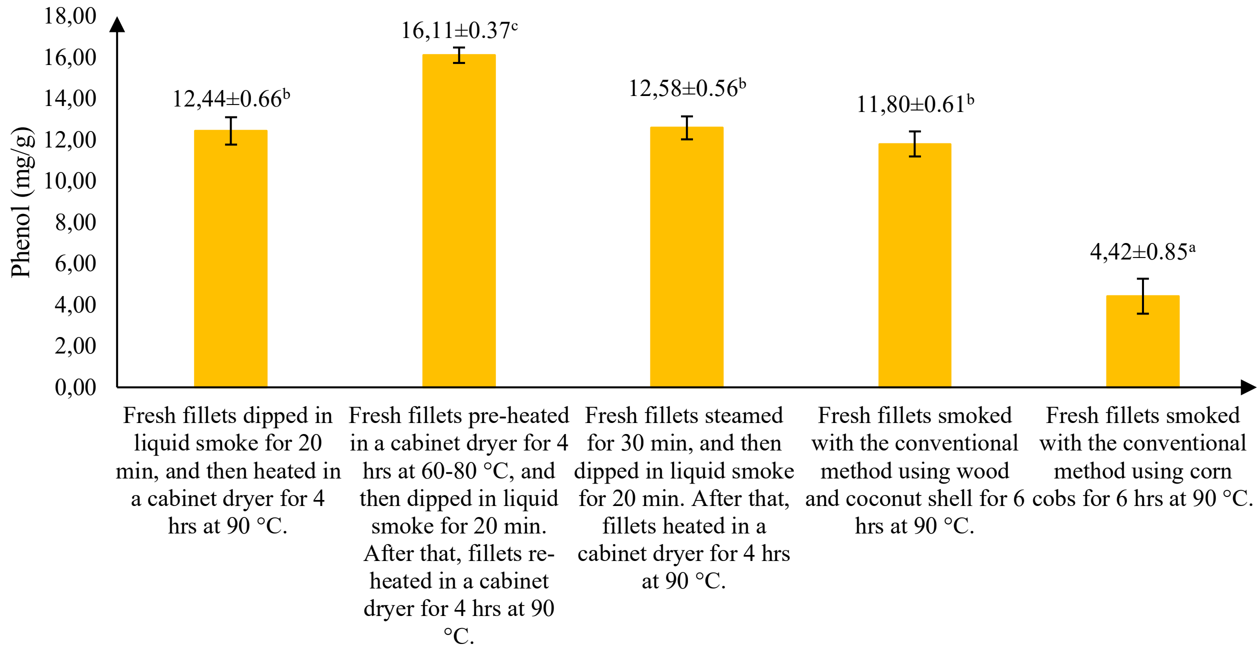

Fig. 6. Phenol level of *julung julung* smoked fillet

Duncan's analysis showed phenol levels in treatments A, C, and D were not different. Previous research reported that the drying treatment of fish meat can increase phenolic compounds in smoked fish products (Sérot *et al*., 2004). The high phenol content in treatment B is thought to be because the pre-heated process causes the fish meat to lose water so that when dipped in liquid smoke, the fish fillets absorb a large amount of liquid smoke. Previous studies have reported that when the fish surface is dried, there is less smoke condensation than products smoked at lower temperatures. The results of this study indicate that a dry fish surface allows for better absorption of smoke components (Belichovska *et al*., 2019).

The study reported phenol levels in liquid smoke from corn cob waste of 0.335 mg/g (Swastawati *et al*., 2007) and 2.55% (Leviyani *et al*., 2019). The phenol level of smoked fish with the conventional method of smoking with corn cobs is lower due to the high acid content, which can potentially reduce the phenol content of smoked fish (Swastawati *et al*., 2012). Anggraini & Nurhazisa (2017) reported that the phenol content of liquid smoke from coconut shells was 3.04%, while liquid smoke from corn cob was 1.38%.

Polycyclic aromatic hydrocarbon (PAH) levels

Polycyclic aromatic hydrocarbons are a group of organic compounds formed by incomplete combustion, such as wood. Therefore, the type of fuel used in the fish smoking process affects the PAH content of smoked fish (Jinadasa *et al*., 2020). One of the carcinogenic PAHs, benzo(a)pyrene, is a carcinogenic marker in smoked fish products (Stołyhwo & Sikorski, 2005). Polycyclic aromatic hydrocarbon (PAH) levels in smoked fish fillets can be seen in Table 2.

Table 2. Polycyclic aromatic hydrocarbon levels in *julung julung* smoked fillet

Polycyclic Aromatic Hydrocarbon	Smoked *Julung Julung* fish (µg/kg) A	B	C	D	E	Corn Cob Liquid Smoke 0.8%(µg/kg)
Benzo(a)pyrene	nd	nd	nd	1.5	0.2	0.5
Benzo(b)fluoranthene	1.2	1.0	1.2	2	1.8	2.5
Benzo(a)anthracene	nd	nd	nd	0.5	nd	0.2
Benzo(g)perylene	0.4	0.2	0.4	1.0	0.8	1.0

nd = not detected

Table 2 shows that the PAH levels in smoked fish are still below the standard required in the Indonesian National Standard No. 2725 of 2013 on smoked fish, which is benzo(a)pyrene max 5 µg/kg (Indonesia Standardization Agency, 2013). The polycyclic aromatic hydrocarbon compound regulated in the Indonesian National Standard is only benzo(a)pyrene. Stołyhwo & Sikorski (2005) reported benzo(a)pyrene levels in smoked fish with hot and cold smoking processes ranging from 0.05 to about 60 µg/kg. Other studies have reported benzo(b,k)fluoranthene levels in salmon and rainbow trout ranging from 1.83 to 9.55 µg/kg, while benzo(g,h)perylene levels in salmon were 0.44 µg/kg (Basak *et al*., 2010). Berhimpon *et al*. (2018) also reported benzo(a)pyrene levels in smoked skipjack fish of 0.25 µg/kg. The level of benzo(a)pyrene in smoked barracuda was reported to be 0.32 µg/kg, benzo(b)fluoranthene 0.35 µg/kg, benzo(k)fluoranthene 0.21 µg/kg, benzo(a)anthracene 0.44 µg/kg, and benzo(g,h)perylene 2.56 µg/kg (Asamoah *et al*., 2021).

High PAH levels in smoked fish are due to the lignin content of the material used as fuel in the smoking process (Asamoah *et al*., 2021). The lignin content in corn cobs is reported to be about 17 – 21% (Thangavelu *et al*., 2018; Olajuyigbe *et al*., 2019; Gandam *et al*., 2022). The lignin content of coconut shell was approximately 59.5% (Wang & Sarkar, 2018), while that of coconut wood was about 50% on a dry weight basis (Anuchi *et al*., 2022). Lignin is reported to absorb PAH compounds because PAH compounds can bind well with lignin (Oliveira *et al*., 2019). During pyrolysis and gasification processes, lignin components react to produce aromatic tar and coke, which can form PAH compounds (Zhou *et al*., 2014; Kawamoto, 2017). Therefore, PAH compounds in smoked fish smoked using corncobs are lower than those smoked with shells and coconut wood.

Sensory assessment

A sensory assessment is carried out to evaluate the panelist's preference level, including appearance, aroma, taste, and texture. A sensory assessment must determine the quality of the smoked fish product and ensure it meets the applicable standards (Hadanu & Lomo, 2019). The sensory characteristics of smoked fish products affect consumer acceptance and preference. Sensory assessment by panelists helps to identify the sensory attributes that are most appealing to consumers, allowing manufacturers to

customize products according to consumer assessment (Ekelemu *et al*., 2021). Indonesian National Standard No. 2725:2013 regarding smoked fish requires a minimum sensory assessment of 7 (Score 1-9) (Indonesia Standardization Agency, 2013). The results of the panelists' assessment of *julung julung* smoked fillet from each treatment can be seen in Fig. 7.

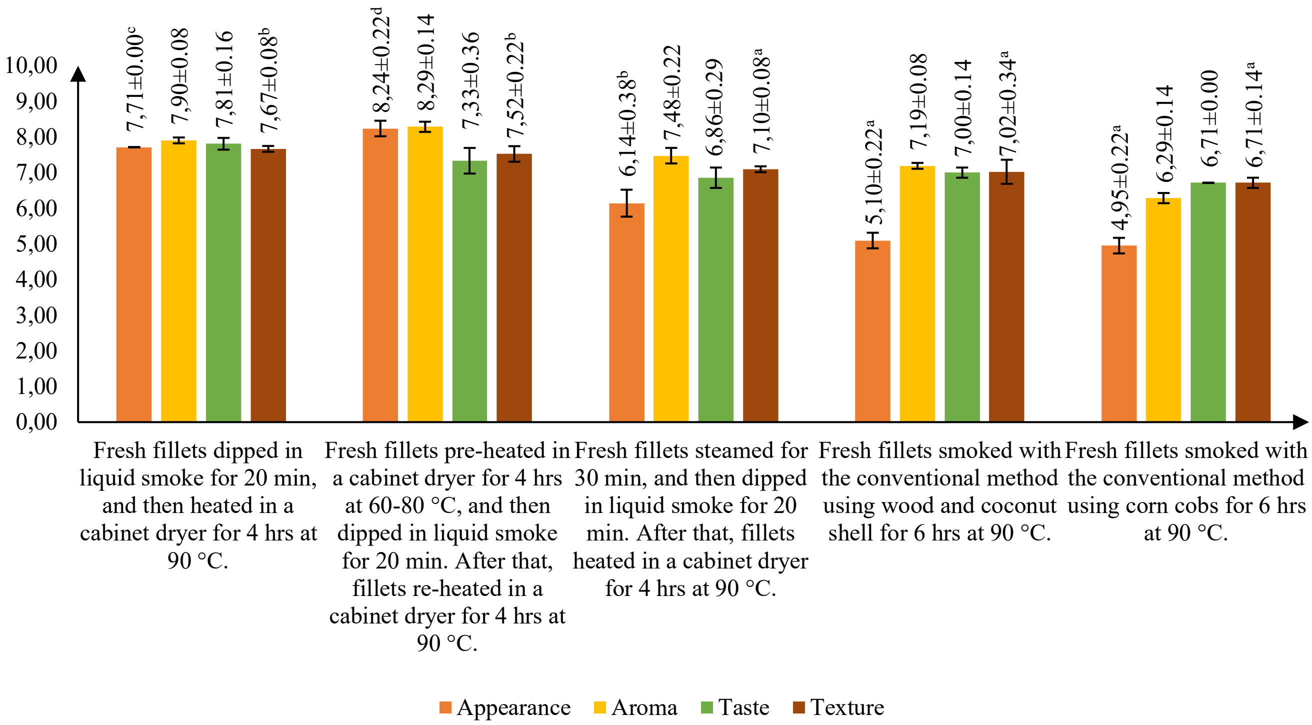

Fig. 7. Sensory assesment of *julung julung* smoked fillet

Appearance: Fig. 7 shows the analysis of the variance of the sensory assessment of the appearance of smoked *julung julung* fish fillets with different smoking method treatments affecting the appearance of smoked fish ($p<0.05$). The panelists' assessment of the appearance of smoked fish fillets ranged from 4.95 to 8.24, with the highest panelists' assessment in treatment B. Based on the requirements of the Indonesian National Standard, only treatments A and B met the minimum panelist assessment requirement of 7. The moisture content factor is thought to have influenced the panelists' assessment of the appearance of smoked fish, so panelists less favored treatment C with steaming. Moisture content can affect the physical properties of fish, such as the appearance and texture of smoked fish (Baten *et al*., 2020a). Smoked fish with high moisture content will make the color of smoked fish look paler (Flick, 2010).

Treatments D and E were less favored by panelists, presumably because conventional smoking methods produce darker products (dark brown), less bright and less shiny. Smoking fish with liquid smoke has smoked fish products with a golden color and clean and shiny surface (Berhimpon *et al*., 2018). Another factor that affects the appearance of smoked fish is the length of the smoking process. The longer the

smoking process, the darker the color will be. The time of the smoking process for smoked fish using the liquid smoke method is shorter than the conventional method (Puke & Galoburda, 2020; Baten *et al*., 2020b). The smoking process can change the color of fish, giving it a distinctive golden color due to the interaction of carbonyls with amino components on the surface of the meat. The color and appearance of smoked fish also positively correlate with phenol content. Phenol compounds in smoke interact with amino acid components in fish meat, producing a distinctive golden color in smoked fish (Montazeri *et al*., 2013).

Aroma: Fig. 7 shows the analysis of the variance of the sensory assessment of the aroma of smoked fish fillets *julung julung* with different smoking method treatments that did not affect the appearance of smoked fish ($p>0.05$). However, the aroma sensory assessment of treatments A, B, C, and D still meets the minimum assessment of the Indonesian National Standard on smoked fish. The sensory assessment of smoked fish aroma was lowest in treatment E. Previous studies have reported that smoking fish with corn cobs as a fuel source resulted in the lowest aroma scores, indicating less aromatic smoked fish (Asmara *et al*., 2022). The aroma and taste of smoked fish are strongly influenced by the time of smoking (Tahir *et al*., 2020). This assessment indicates that the time of the smoking process of 4 and 6 hours in all treatments has not influenced the taste and aroma of smoked fish.

Taste: Fig. 7 shows the analysis of the variance of the sensory assessment of the taste of smoked fish fillets *julung julung* with different smoking method treatments that did not affect the appearance of smoked fish ($p>0.05$). The taste of smoked fish is influenced by the time of the smoking process. Previous studies have reported that longer smoking time can improve the sensory attributes of taste (Baten *et al*., 2020b). This result indicates that the 2-hour time difference between the liquid smoke smoking treatment and the conventional smoking method has not affected the taste of smoked fish. A distinctive smoked taste without bitterness is a criterion for assessing the taste of good quality smoked fish (Sukowati *et al*., 2021).

Texture: Fig. 7 shows the analysis of variance of the sensory assessment of the texture of smoked fish fillet *julung julung* with different smoking method treatments giving effect to the texture of smoked fish ($p<0.05$). Panelists' assessment of the texture of smoked fish *julung julung*, ranged from 6.71 – 7.67, with the highest panelist assessment in treatments A and B. Duncan's test analysis showed that treatments A and B differed from treatments C, D, and E. This result was thought to be because the fish fillets were dipped in liquid smoke, giving a dry, compact, and tender texture. Whereas in treatment C (fish fillets subjected to steaming), the texture of the smoked fillets was rather sticky and not solid. Treatments D and E produced the texture of smoked fish fillets which were less dense and not compact. The texture of smoked fish is negatively correlated with its moisture content. The higher the moisture content in smoked fish, the softer and less firm the texture (Chan *et al*., 2022). High moisture content in smoked fish products results in less dense fish muscle fibers (Chang *et al*., 2021).

A good texture of smoked fish is compact, moderately elastic, and not sticky (Sulistijowati *et al*., 2021). The Indonesian National Standard requires that the texture of smoked fish is dense and compact and the tissue is very tight (Indonesia Standardization Agency, 2013). Based on consumer surveys, the preferred texture of smoked fish is dense, slightly wetter, and easy to chew, and there is a sensation of liquid in the mouth when chewed (Ticoalu *et al*., 2019).

Conclusion

Based on the findings of this study, it can be concluded that treatment B (Fresh fillets pre-heated in a cabinet dryer for 4 hours at 60 – 80 ºC and then dipped in liquid smoke for 20 minutes. After that, the fillets were re-heated in a cabinet dryer for 4 hours at 90 ºC.), when compared to the conventional smoking treatment based on total volatile bases, moisture content, water activity, pH value, phenol level, polycyclic aromatic hydrocarbon content, and sensory assessment. In general, fish smoking dipped in liquid smoke from corn cob produced a better quality of smoked fish than the conventional smoking method. It is necessary to evaluate different smoking times on smoked fish fillets of *julung julung* with liquid smoke method from corn cob.

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Author contributions

Conflicts of interest

The authors declare that there is no conflict of interest.

Highlights

- Liquid smoke from corn cobs can increase total phenols. Fillets dried and soaked in liquid smoke.

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Characteristics of *Julung Julung* Smoked Fish Fillets (*Hemiramphus* sp.) using Liquid Smoke from Corn Cobs Waste

Abstract

Corn cobs can be utilized as a shell ingredient for liquid smoke. Liquid smoke can be used for the smoked fish industry in North Sulawesi, Indonesia. However, the utilization of liquid smoke for the smoked fish industry has yet to be optimal. This study aimed to obtain the best smoking method for *julung julung* fish fillets (*Hemiramphus* sp.) using liquid smoke from corn cob waste. The treatment in this study was to apply liquid smoke from corn cob waste in the smoking process, which was compared with the conventional method (using shell, coconut wood, and corn cob). Parameters to determine the quality of smoked fish were total volatile bases (TVB), moisture, water activity, pH, phenol levels, polycyclic aromatic hydrocarbon (PAH), and sensory assessment. The results showed that the TVB of smoked *julung julung* fish fillets ranged from 19.83 – 32.27 mg N/100g. The moisture ranged from 14.10 – 30.57%. Water activity ranged from 0.46 – 0.82. pH ranged from 4.60 – 5.81. Phenol levels ranged from 4.42 – 16.11 mg/g. PAH levels are still below the standard required in the Indonesian National Standard. Panelists rated neutral to really like the appearance, aroma, taste, and texture of smoked fish. From these results, it can be concluded that the best treatment is fresh fillets preheated in a cabinet dryer for 4 hours at 60 – 80 ^{o}C and then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated in a cabinet dryer for 4 hours at 90 ^{o}C.

Keywords: Coconut, Indonesia, Shell, North Sulawesi, Wood

Introduction

Corn is the second most widely grown crop in Indonesia after rice. Indonesia is the 7th corn producer in the world, with more than 12 million tons of corn produced in 2020 (Nazmi *et al*., 2021). North Sulawesi is one of the contributing regions that produce corn in Indonesia. Considerable corn production will also produce waste, especially corn cobs, which are generally thrown away and burned by most Indonesians (Cahyadi *et al*., 2021).

One of the corn cobs was used to make liquid smoke. Previous research reported that corn cobs can produce liquid smoke, a by-product of the pyrolysis of corn cob waste. The yield of liquid smoke from corn cobs is about 28.37%, with a pH value of 3.5 (Sriharti *et al*., 2020). The particle size of corn cobs affects the yield of liquid smoke, with smaller particle sizes resulting in higher yields (Aladin *et al*., 2018). Another study reported a phenolic content of 335 mg/L in liquid smoke produced from corn cobs (Swastawati *et al*., 2007).

Liquid smoke is a natural product made from the condensation of smoke from burning wood (Andy *et al*., 2021). Liquid smoke is commonly used as a flavoring in food to provide a smoked flavor without the food undergoing the actual smoking process (Sari *et al*., 2006). Using liquid smoke in food will save time, energy, and labor and reduce production costs (Krah *et al*., 2019). In addition, using liquid smoke in food can speed up and standardize the smoking process, adding flavor and microbiological safety

while reducing production time (Indiarto *et al*., 2020). Generally, various types of hardwood were used to manufacture liquid smoke (Diatmika *et al*., 2019). The quality of the liquid smoke produced may vary depending on the type of wood used (Budaraga *et al*., 2016). In addition to imparting specific flavors and aromas to foods (Maulina *et al*., 2020), liquid smoke is reported to be antimicrobial (Septana *et al*., 2020) and antioxidant (Budaraga & Putra, 2021).

The conventional smoking processes generally use hardwood as fuel, such as melon wood (Umar *et al*., 2018), mangrove wood (Cissoko *et al*., 2020), teak wood (Daramola *et al*., 2020), coconut shells, and coconut husks (Nugroho *et al*., 2018). Corn cobs have also been reported to be used in the fish smoking process (Sukowati, 2023). According to literature, traditional fish smoking in North Sulawesi, Indonesia, uses smoke from burning coconut shells or wood charcoal (Landangkasiang *et al*., 2017; Primalasari *et al*., 2019; Saediman *et al*., 2021). Smoked fish processing has been known for a long time because of its traditional processing techniques, simplicity, ease of implementation, and low cost (Islamiyah, 2021). In North Sulawesi, Indonesia, smoked fish commonly known as *fufu* is traditionally made from skipjack (*Katsuwonus pelamis*); *julung julung* (*Hemiramphus far*) (Berhimpon *et al*., 2018); and scad fish (*Decapterus* spp.) known as *pinekuhe* (Ansar & Ijong, 2021). The smoked fish product of *julung julung* is a typical product from North Sulawesi and Gorontalo, commonly called *roa* or *galavea* (Azis & Akolo, 2020).

The study's reported that the conventional smoking process has disadvantages such as smoking time, concentration of carcinogenic substances, temperature, and inconsistent product quality (Racovita *et al*., 2020). Conventional smoking of food products has been shown to produce carcinogenic components such as polycyclic aromatic hydrocarbons (Jinadasa *et al*., 2020). Benzo(a)pyrene ($C_{20}H_{12}$) is one of the carcinogenic compounds produced from traditional smoking products (Jinadasa *et al*., 2020). One method to reduce carcinogenic compounds in smoked fish products is to use liquid smoke in the smoking process (Nithin *et al*., 2020; Xin *et al*., 2021).

The utilization of liquid smoke in the fish smoking industry in North Sulawesi is very likely to be developed. North Sulawesi was reported to have both large-scale and domestic fish-smoking industries (Dotulong *et al*., 2018; Primalasari *et al*., 2019). Making liquid smoke is relatively simple, so it will be readily accepted by fish-smoking industry players (Ali & Al Fiqri, 2020). Previous research reported that using liquid smoke in smoked fish products resulted in good quality in terms of appearance, flavor, and aroma (Rizal *et al*., 2020). In addition, using liquid smoke in smoked fish products can add nutritional value and durability, making it possible to reach a wider market area (Ali *et al*., 2021).

Previous research reported that smoked fish dipped in liquid smoke for 15 minutes had a total plate count value of 4.7×10^4 CFU/g on day six and moisture content below 60% during storage (Suroso *et al*., 2018). Dipping fish in liquid smoke with a concentration of 15% and a dipping time of 60 minutes showed a bacterial colony of 2.12×10^2 CFU/g (Ali *et al*., 2021). The Indonesian National Standard on

smoked fish requires a maximum total plate count of 5×10^4 CFU/g and a maximum moisture content of 60% (Indonesia Standardization Agency, 2013). Another study reported that smoked fish processed with liquid smoke had a moisture content of 47.63%, phenol content of 12.62%, and pH of 4.8 (Berhimpon *et al*., 2018). The results of these studies indicate that liquid smoke is good to apply to smoked fish products. This study characterized smoked fish fillets of *julung julung* (*Hemiramphus* sp.) produced with liquid smoke from corn cobs waste. The study was conducted by dipping the fish fillets of *julung julung* with liquid smoke, with drying and steaming treatments on the fish fillets. This study aims to obtain the best smoking method for fish fillets using liquid smoke from corn cobs waste based on the assessment of total volatile bases, moisture content, water activity, pH value, phenol content, polycyclic aromatic hydrocarbon content, and sensory assessment.

Materials and methods

The main materials of the study were *julung julung* fish (*Hemiramphus* sp.), corn cob (*Zea mays* L.) from Gangga Island in Likupang, North Sulawesi, Indonesia. This study includes the preparation of liquid smoke from corn cobs waste, preparing fish (weeding and filleting), and treating fish fillets by dip in liquid smoke. Fresh *julung julung* fish was obtained from traditional fishermen in Manado, North Sulawesi, Indonesia (Fig. 1). The fish was placed in a cool box and given ice in a ratio of 1:1 (ice:fish) (Mishra *et al*., 2021). Then, the fish was washed, weeded, and filleted. The cleaned fillets were dipped in a 5% salt solution for 30 minutes. The concentration of liquid smoke from corn cob waste was 0.8% (Berhimpon *et al*., 2018).

Fig. 1. *Julung julung* (*Hemiramphus* sp.)

The treatment in this study was applying liquid smoke from corn cobs waste to the smoking process of *julung julung* fish fillets, which was compared with the conventional method (using coconut wood, coconut shells, and corn cobs).

(A) : Fresh fillets were dipped in liquid smoke for 20 minutes and then heated in a cabinet dryer for 4 hours at 90 ºC.

(B) : Fresh fillets pre-heated in a cabinet dryer for 4 hours at 60 – 80 ºC, and then dipped in liquid smoke for 20 minutes. After that, fillets were re-heated in a cabinet dryer for 4 hours at 90 ºC.

(C) : Fresh fillets were steamed for 30 minutes and then dipped in liquid smoke for 20 minutes. After that, fillets were heated in a cabinet dryer for 4 hours at 90 ºC.

(D) : Fresh fillets were smoked with the conventional method using wood and coconut shell for 6 hours at 90 ºC.

(E) : Fresh fillets were smoked with the conventional method using corncob for 6 hours at 90 ºC.

Liquid smoke manufacturing process

Making liquid smoke refers to modified research (Aladin *et al*., 2018). Modifications were made to the equipment used in the pyrolysis process. Dry corn cobs were weighed to determine fuel efficiency. Corn cobs were gradually burned into the combustion furnace. The furnace was closed to prevent smoke from escaping from the tank. The smoke generated from the combustion flows through a pipe connected to a storage tank covered with ice cubes. The smoke that passes through the pipe will become cold, so condensation occurs, turning the smoke into liquid. The smoke that has been formed is collected into a container attached to the end of the pipe. The liquid smoke obtained is then allowed to settle the tar formed for three weeks and filtered to obtain clear liquid smoke.

Total volatile bases assay

Total volatile base (TVB) assay refers to research by Tambunan & Chamidah (2021). TVB assay aims to determine the amount of volatile base compounds formed from protein breakdown—the principle of TVB analysis, namely by evaporating volatile base compounds at room temperature for 24 hours. The compound is then bound with boric acid and titrated with an HCl solution. After the boric acid solution was stored in the inner chamber of the Conway cell containing the blank (V_0) and the filtrate (V_1), the boric acid solution was titrated with 0.02 N HCl so that the boric acid solution changed color to pink. TVB levels in the smoked fish meat were expressed as mg N/100g.

$$TVB\left(\mathrm{mg}\frac{\mathrm{N}}{100\mathrm{g}}\right) = \frac{(V_1 - V_0) \times N.HCl \times dilution \times 100}{sample\ weight\ (g)}$$

Moisture content assay

The moisture content by method of Indonesia National Standard (Indonesia Standardization Agency, 2006a). An empty porcelain cup is weighed (A). Then, the sample was weighed to ± 2 g in a porcelain cup of known mass (B). The samples were dried in an oven at 105°C for 20 hours. The samples were then cooled in a desiccator, after which the samples in the cup were weighed (C). Moisture content was expressed as %.

$$Moisture\ (\%) = \frac{B\ (g) - C\ (g)}{B(g) - A\ (g)} \times 100\%$$

Water activity (Aw) assay

Water activity assay used an Aw meter (Rotronic, HygroPalm 23-AW-A). The Aw meter was set up at room temperature for 2 hours. The water activity of smoked fish was measured by placing the sample into a sample container and conditioning it for 30-60 minutes. The Aw meter sensor is contacted with the sample in the container. Then the water activity (Aw) value can be read on the Aw meter panel (Saputra *et al*., 2014).

pH assay

The sample weighed as much as 30 g, then 400 mL of distilled water was added at 28 ºC. The sample was homogenized with a magnetic stirrer to be evenly stirred. The pH meter electrode was inserted into the sample solution. The data taken in this test is the pH value read by the pH meter (Hanna Instruments HI99192) (Lekahena & Jamin, 2018).

Phenol level assay

Samples were extracted by adding 1 mL ethanol p.a., 5 mL distilled water, and 0.5 mL Folin Ciocalteu 50%, homogenized, and allowed to stand for 5 minutes. Then added, 1 mL of 5% Na_2CO_3 and left in the dark for ± 60 minutes with gallic acid as a standard. The absorbance value was read on a spectrophotometer (Thermo Scientific Genesys 50) with a wavelength of 725 nm. Total phenolics were expressed as mg/g (Indonesia Standardization Agency, 2004; Muliadin *et al*., 2022).

$$Total\ Phenol\ (\frac{mg}{g}) = \frac{Gallic\ acid\ equivalence\ \left(\frac{mg}{L}\right) \times Volume\ (L)}{Sample\ weight\ (g)}$$

Polycyclic aromatic hydrocarbon (PAH) assay

PAH content testing was carried out based on the instructions Basak *et al*. (2010). A 5 g sample was weighed and collected in a 100 ml flask. Then 5 mL of 50% KOH solution, 75 mL of methanol, 1 mL

of internal standard solution (9,10 dimethylantracene), and some boiling stones were added. After that, the mixture was boiled for 4 hours in a soxhelet. The liquid phase was transferred to a separatory funnel and extracted with 100 mL of n-hexane with shaking for 3 min. The MeOH:KOH phase was drained and discarded. The combined n-hexane phase was rinsed twice, each with 50 mL of H_2O-MeOH (8:1) and 50 mL of water. The methanol-water and water phases were dried and discarded. The organic phase was concentrated in a rotary evaporator (40°C) to a volume of about 10 mL. The concentrated extract was dried under nitrogen gas.

After that, the silica gel column was cleaned by dissolving 15 g of silica gel in n-hexane and transferred to a column (200 mm long; diameter: 12 mm). Then, 1 g of anhydrous sodium sulfate was added. The concentrated sample extract was transferred to the column and eluted with 1 mL of n-hexane. A stock solution containing 0.1 mg mL^{-1} of 9,10 dimethylantracene was dissolved in n-hexane and stored at 4°C in a volumetric flask (with a glass stopper) wrapped in aluminum foil to avoid possible light degradation. Working standard solutions were prepared from the stock solutions. Analysis of PAH compounds used a Hewlett-Packard 1100 HPLC equipped with an Agilent-110 fluorescence detector. The injection volume was 10 µL. Mobile phase gradient (acetonitrile-water): 80% acetonitrile + 20% water with a flow rate of 1 ml min^{-1} and a wavelength of 270 nm. The external standard mixture was used for PAH concentration calculation.

Sensory assessment assay

Sensory assessment assay refers to Indonesia National Standard (Indonesia Standardization Agency, 2006b). The sensory assessment was based on a hedonic scale (Table 1). Smoked fish samples were placed on a plastic plate with a glass of water, coded, and presented to 30 panelists randomly under light. The parameters observed were appearance, aroma, taste, and texture. Panelists rated acceptance of the samples on a scale of 1 – 9.

Table 1. Score sheet of hedonic assessment

Specification	Score
Really like it	9
Really like	8
Like	7
Rather like	6
Neutral	5
Rather dislike	4
do not like	3
Very dislike	2
Really don't like it	1

Data analysis

Data analysis was performed using Statistical Product and Service Solutions (SPSS) version 20.0. Statistical analysis of data was performed based on one-way analysis of variance (ANOVA) using a significance level of ($P<0.05$). Specific group differences were determined using Duncan's test.

Results and discussion

Liquid smoke production was carried out using a simple liquid smoke distillation device. This process consumed 17 kg of corn cobs and 12 kg of crushed ice. Liquid smoke from corn cobs is more accessible than coconut shells or wood (Maulina & Karo, 2021). Using coconut shells or wood as fuel will form a flame, thus reducing the volume of smoke in the furnace (Kabir Ahmad *et al*., 2022). The smoked *julung julung* were analyzed for TVB, moisture content, water activity, pH value, phenol content, PAH content, and sensory assessment.

Total volatile bases

The total volatile base (TVB) value is measured to determine the quality of the smoked fish. TVB value is one of the parameters used to determine the decline in fish quality and measures the amount of volatile base compounds formed due to protein degradation (Castro *et al*., 2006). Volatile bases formed in fish muscle tissue mainly consist of ammonia, trimethylamine (TMA), and dimethylamine (DMA) (VELP Scientifica, 2013). TVB value is an essential characteristic for the quality assessment of seafood products and is emerging as the most commonly used chemical parameter to assess the palatability of seafood (Rasulu *et al*., 2020). The results of *total volatile bases* on smoked fish fillets of *julung julung* are presented in Fig. 2.

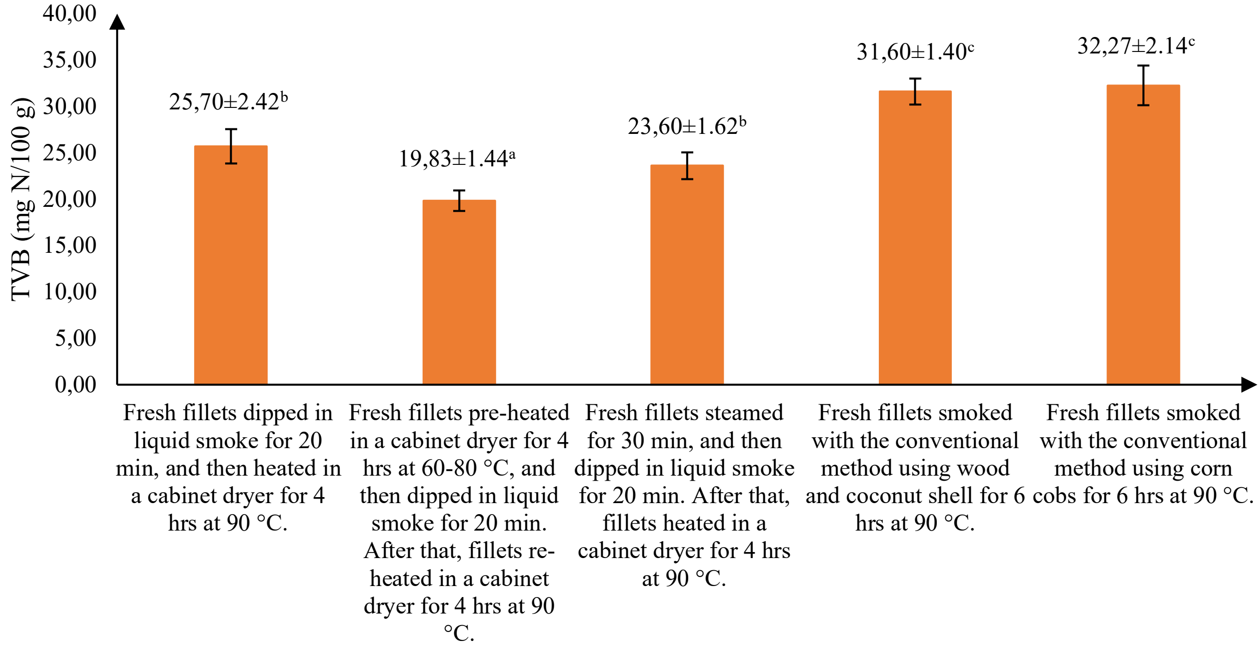

Fig. 2. Total volatile bases of *julung julung* smoked fillet

Fig. 2 shows the analysis of the variance of smoked fish fillets with different smoking method treatments affecting changes in TVB content ($p<0.05$). The TVB content of smoked fish fillets ranged from 19.83 – 32.27 mg N/100g, with the lowest TVB value in treatment B. The treatment of the smoking method with liquid smoke from corn cob waste (treatments A, B, and C) had lower TVB levels when compared to the conventional smoking method (treatments D and E). A literature search has not found why liquid smoking has a lower TVB value than conventional smoking methods. However, one possibility is that the liquid smoke smoking method allows the smoke components to be absorbed into the fish meat better than the conventional smoking method. Better absorption of liquid smoke phenol components in fish meat can inhibit and control microbial growth that causes a decrease in the quality of smoked fish (Santoso *et al*., 2015). A literature study showed that the average TVB value of smoked mackerel (*Euthynnus affinis*) treated with liquid smoke addition ranged from 24.63 – 28.38 mg N/100g (Hardianto & Yunianta, 2015). Another study reported smoked mackerel with a smoking process using rubber wood liquid smoke, producing smoked fish products with lower TVB values (Suroso *et al*., 2018). Duncan's analysis showed a difference between treatments B, A – C, and D – E on the TVB value of smoked fish fillets. The analysis of variance indicates that the drying process of smoked fish fillets before immersion into liquid smoke can maintain the quality of smoked fish (Kaparang *et al*., 2013). Duncan's analysis also showed that treatments A – C and D – E had no difference in TVB value, indicating that the moisture content between the two treatments was not different. TVB value is closely related to moisture content (Rasulu *et al*., 2020).

The European standard (European Market Observatory for Fisheries and Aquaculture, 2020) states that the limit range of TVB value for smoked fish is 25 – 35 mg N/100 g. All treatments' TVB value of the smoked fish fillets still met the European standard. Previous studies reported TVB values of fresh fish fillets of 8.70 ± 0.86 mgN/100g (Moosavi-Nasab *et al*., 2021) and 12.94 ± 0.92 mg N/100g (Bouzgarrou *et al*., 2020). Messina *et al*. (2021) reported that smoked fish fillets that underwent two drying processes had a TVB value of <20 mg N/100g. Another study also reported the TVB value of smoked fish fillet samples with hot smoking and cold smoking, which amounted to 17.80 ± 0.17 and 18.95 ± 0.20 mg N/100g, respectively (El-Lahamy *et al*., 2019). Aydin *et al*. (2020) reported a TVB value of smoked fish with hot smoking of 11.82 ± 0.05 mg N/100g.

Moisture content

Moisture content is an important parameter to determine the quality of smoked fish produced. The moisture content contained in smoked fish can affect the shelf life of smoked fish because water in food is a medium for microbes to grow (Kaban *et al*., 2019). The smoking process was reported to reduce the moisture content of fresh fish to a range between 9 and 17% (Sikoki & Aminigo, 2002). The moisture content of smoked fish fillets is presented in Fig. 3.

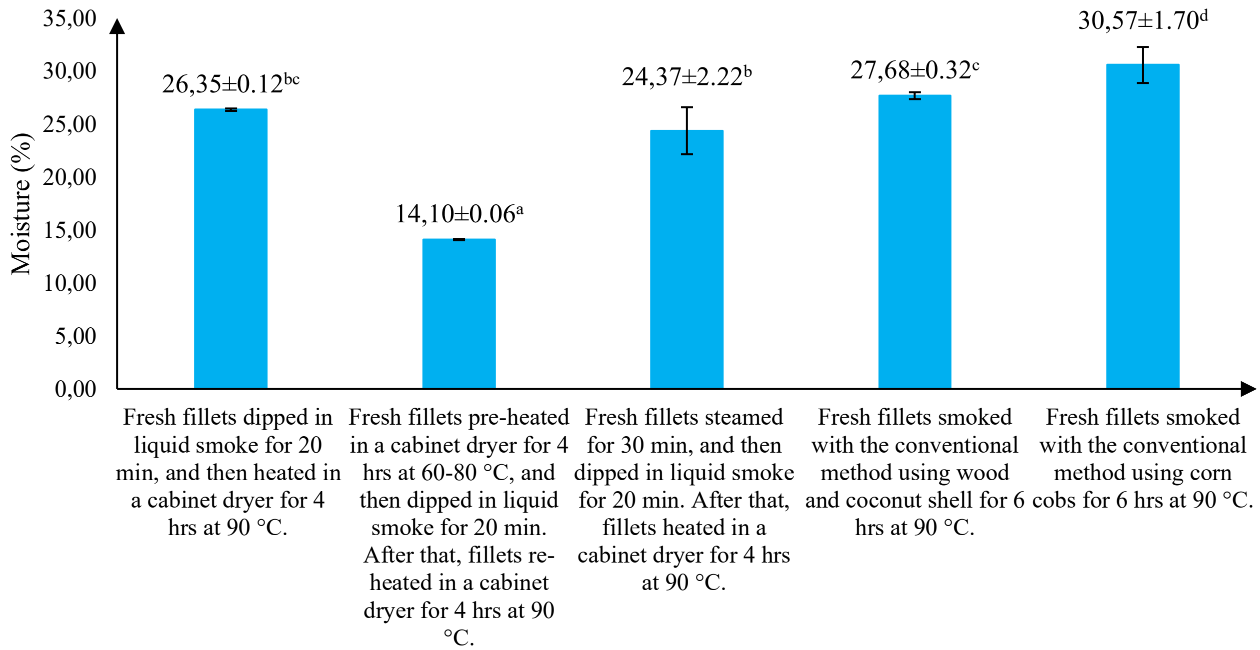

Fig. 3. Moisture content of *julung julung* smoked fillet

Fig. 3 shows the analysis of the variance of smoked *julung julung* fish fillets with different smoking methods treatment affects moisture content ($p<0.05$). The moisture content of smoked *julung julung* fish fillets ranged from 14.10 – 30.57%, with the lowest moisture content in treatment B. The

moisture content value in smoked fish products from all treatments still meets the Indonesian National Standard No. 2725:2013 on smoked fish, which is a maximum of 60% (Indonesia Standardization Agency, 2013). Darianto *et al*. (2018) reported that the smoking process can reduce the moisture content of fish to below 40%, which can help preserve it longer. The treatment of the smoking method with liquid smoke with corn cob (Treatment A, B, C) has a lower moisture content when compared to the treatment of the conventional smoking method (Treatment D and E). This result is because the smoking chamber is not fully enclosed in the conventional smoking method, so the heat generated could be more optimal. Suboptimal heat can increase moisture content and cause the moisture content of smoked fish to decrease only slightly (Amos & Paulina, 2017). Whereas in the liquid smoke smoking method, temperature and humidity can be controlled better so that the moisture content of the product can be reduced efficiently (Salindeho & Lumoindong, 2017).

Duncan's analysis showed a difference between treatment B, treatment A – C, and treatment D – E on the moisture content of smoked fish fillets. The results of the variance study indicate that the drying process of smoked fish fillets before the dip in liquid smoke in treatment B can help reduce the moisture content. This study's results are from previous research, which also reported a significant decrease in moisture content in smoked fish products with two times drying treatment (Messina *et al*., 2021). Duncan's analysis also showed that treatments A and C were not different because steaming in treatment C will increase the moisture content of the product (Salmatia *et al*., 2020). The conventional smoking method with corn cob fuel has the highest moisture content; this result is the conventional smoking process with corn cobs, and the heat generated is lower than smoking using wood (Asmara *et al*., 2022).

Water activity (Aw)

Water activity is one of the essential parameters in the quality of smoked fish. Water activity (Aw) is expressed as the ratio of the vapor pressure in the food to the vapor pressure of pure water, and it predicts whether water tends to move from the food product into the cells of microorganisms that may be present. A well-smoked fish has a water activity of <0.50 and a moisture content between 15 and 25% to inhibit the growth of pathogenic microorganisms in smoked fish products (Mondo *et al*., 2020). According to British Columbia Centre for Disease Control (2013), the maximum water activity value for smoked fish is 0.97. Water activity correlates with the moisture content of a smoked fish product and is two important factors affecting food safety and quality (Fitri *et al*., 2022). The water activity values of smoked fish fillets can be seen in Fig. 4.

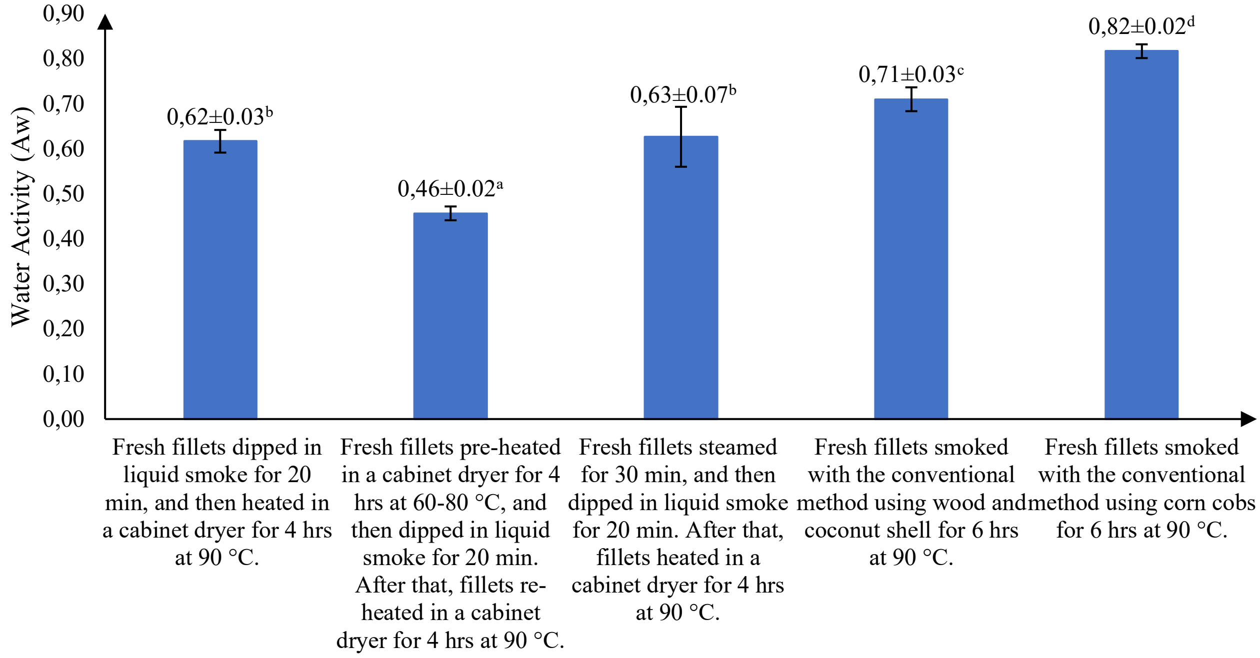

Fig. 4. Water activity of *julung julung* smoked fillet

Fig. 4 shows the analysis of the variance of smoked fish fillet *julung julung* with different smoking method treatments affecting water activity ($p<0.05$). The water activity value of smoked fish fillet ranged from 0.46 – 0.82, with the lowest water activity in treatment B. The treatment of the smoking method with corn cob liquid smoke (Treatment A, B, C) had a lower water content when compared to the treatment of the conventional smoking method (treatment D and E).

The water activity value of fish meat can vary depending on the cooking method, temperature, and duration of heating (Oliveira *et al*., 2017; Zhang *et al*., 2023). Therefore, the factor of fish fillet treatment before immersion in liquid smoke caused the difference in water activity value in treatment B compared to treatments A and C. The water activity value is directly proportional to the water content value of each treatment. The heating process can reduce the moisture content of fish meat (Kiczorowska *et al*., 2019), thus affecting the water activity of fish meat (Gómez *et al*., 2020).

pH

The pH value of smoked fish is an essential factor affecting its quality. The pH value will decrease with increasing smoking time (Baten *et al*., 2020b). The analysis of the variance of smoked fish fillets *julung julung* with different smoking method treatments affects the pH value ($p<0.05$). The pH of smoked fish fillet ranged from 4.60 to 5.81 (Fig. 5). The treatment of the smoking method with corn cob liquid smoke (treatments A, B, and C) had a lower pH value when compared to the conventional smoking method (treatments D and E). Swastawati *et al*. (2022) also reported that the pH value of smoked barracuda fish with the liquid smoke method was significantly lower than that of the conventional

method. Another study also reported that using liquid smoke can reduce pH caused by the condensation of organic acids in the smoking process (Puke & Galoburda, 2020). The pH value of smoked fish correlates with the phenol content contained in the smoke component, and an increase in phenol content causes a decrease in pH (Berhimpon *et al*., 2018).

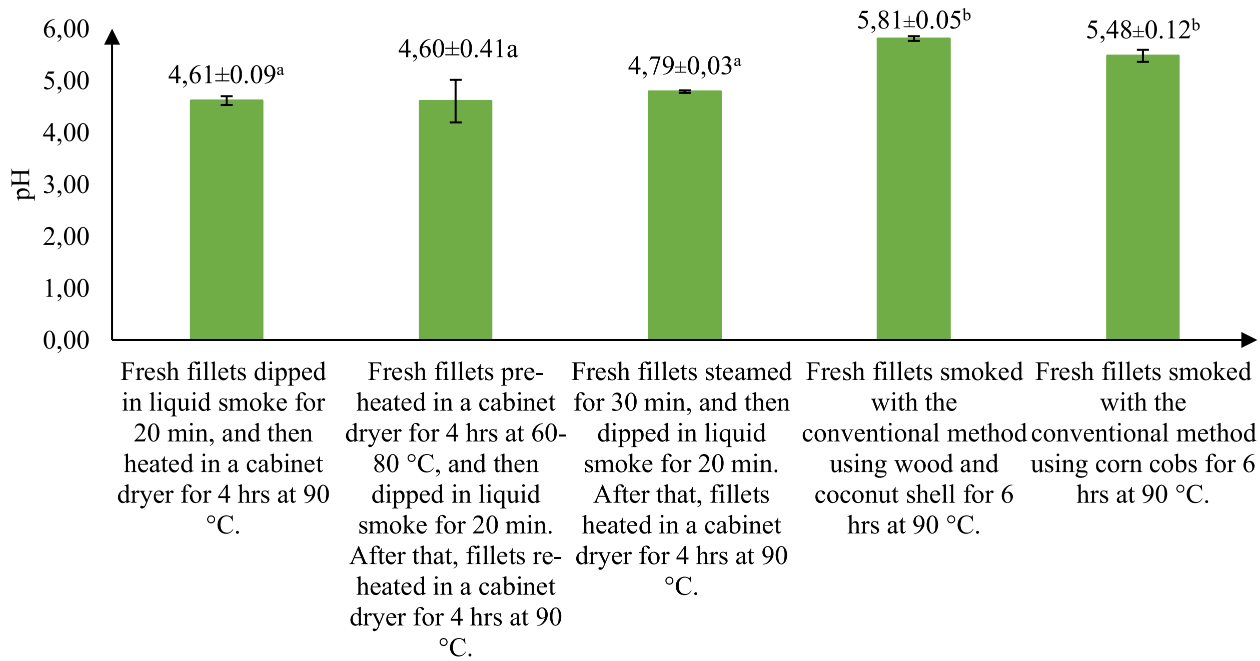

Fig. 5. pH of *julung julung* smoked fillet

Phenol level

Phenol is a compound found in wood smoke. Phenol compounds enter food through diffusion and capillary action, affecting taste, color, and aroma and extending shelf life (Remy *et al*., 2016). In addition, phenol compounds are also reported to act as antioxidants that can prevent rancidity in fish meat (Sérot *et al*., 2004). Leksono *et al*. (2020) also noted that the higher the phenol level in smoke, the stronger the flavor and aroma of smoked fish. The analysis of the variance of smoked fish fillets julung julung with different smoking method treatments affecting phenol level ($p<0.05$). The phenol level of smoked fish fillet ranged from 4.42 to 16.11 mg/g (Fig. 6). The treatment of the smoking method with corn cob liquid smoke (treatments A, B, and C) had higher phenol level when compared to the conventional smoking method (treatments D and E).

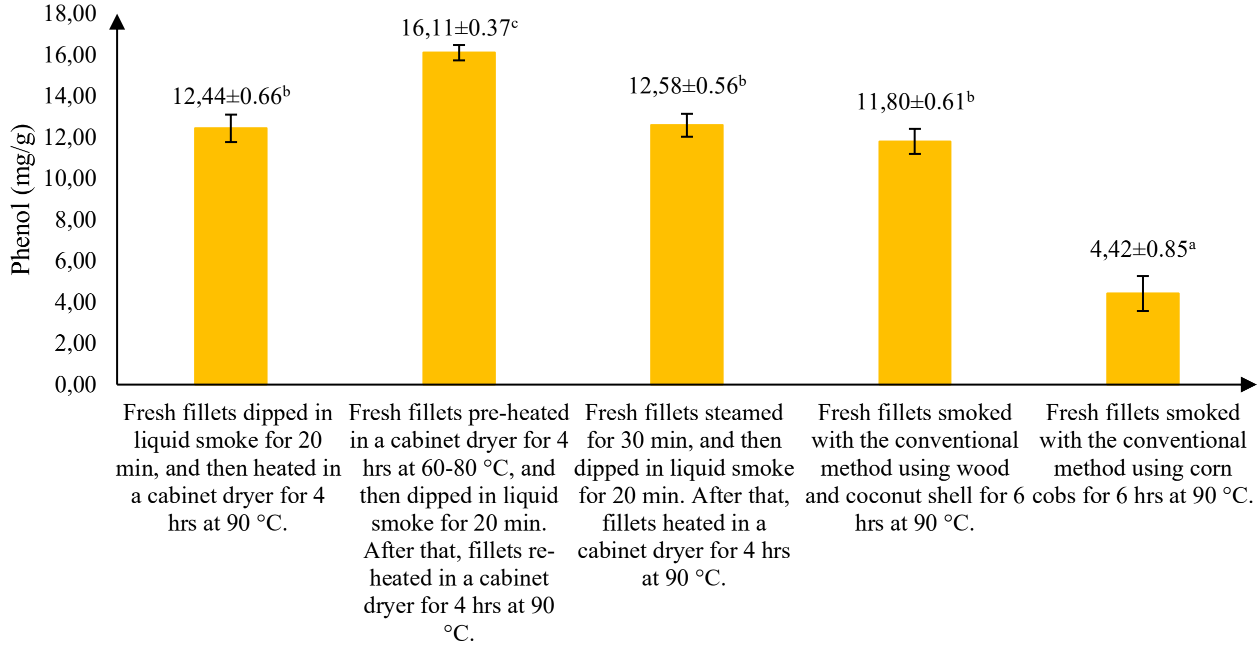

Fig. 6. Phenol level of *julung julung* smoked fillet

Duncan's analysis showed phenol levels in treatments A, C, and D were not different. Previous research reported that the drying treatment of fish meat can increase phenolic compounds in smoked fish products (Sérot *et al*., 2004). The high phenol content in treatment B is thought to be because the pre-heated process causes the fish meat to lose water so that when dipped in liquid smoke, the fish fillets absorb a large amount of liquid smoke. Previous studies have reported that when the fish surface is dried, there is less smoke condensation than products smoked at lower temperatures. The results of this study indicate that a dry fish surface allows for better absorption of smoke components (Belichovska *et al*., 2019).

The study reported phenol levels in liquid smoke from corn cob waste of 0.335 mg/g (Swastawati *et al*., 2007) and 2.55% (Leviyani *et al*., 2019). The phenol level of smoked fish with the conventional method of smoking with corn cobs is lower due to the high acid content, which can potentially reduce the phenol content of smoked fish (Swastawati *et al*., 2012). Anggraini & Nurhazisa (2017) reported that the phenol content of liquid smoke from coconut shells was 3.04%, while liquid smoke from corn cob was 1.38%.

Polycyclic aromatic hydrocarbon (PAH) levels

Polycyclic aromatic hydrocarbons are a group of organic compounds formed by incomplete combustion, such as wood. Therefore, the type of fuel used in the fish smoking process affects the PAH content of smoked fish (Jinadasa *et al*., 2020). One of the carcinogenic PAHs, benzo(a)pyrene, is a carcinogenic marker in smoked fish products (Stołyhwo & Sikorski, 2005). Polycyclic aromatic hydrocarbon (PAH) levels in smoked fish fillets can be seen in Table 2.

Table 2. Polycyclic aromatic hydrocarbon levels in *julung julung* smoked fillet

Polycyclic Aromatic Hydrocarbon	Smoked *Julung Julung* fish (µg/kg)					Corn Cob Liquid Smoke 0.8%(µg/kg)
	A	B	C	D	E	
Benzo(a)pyrene	nd	nd	nd	1.5	0.2	0.5
Benzo(b)fluoranthene	1.2	1.0	1.2	2	1.8	2.5
Benzo(a)anthracene	nd	nd	nd	0.5	nd	0.2
Benzo(g)perylene	0.4	0.2	0.4	1.0	0.8	1.0

nd = not detected

Table 2 shows that the PAH levels in smoked fish are still below the standard required in the Indonesian National Standard No. 2725 of 2013 on smoked fish, which is benzo(a)pyrene max 5 µg/kg (Indonesia Standardization Agency, 2013). The polycyclic aromatic hydrocarbon compound regulated in the Indonesian National Standard is only benzo(a)pyrene. Stołyhwo & Sikorski (2005) reported benzo(a)pyrene levels in smoked fish with hot and cold smoking processes ranging from 0.05 to about 60 µg/kg. Other studies have reported benzo(b,k)fluoranthene levels in salmon and rainbow trout ranging from 1.83 to 9.55 µg/kg, while benzo(g,h)perylene levels in salmon were 0.44 µg/kg (Basak *et al*., 2010). Berhimpon *et al*. (2018) also reported benzo(a)pyrene levels in smoked skipjack fish of 0.25 µg/kg. The level of benzo(a)pyrene in smoked barracuda was reported to be 0.32 µg/kg, benzo(b)fluoranthene 0.35 µg/kg, benzo(k)fluoranthene 0.21 µg/kg, benzo(a)anthracene 0.44 µg/kg, and benzo(g,h)perylene 2.56 µg/kg (Asamoah *et al*., 2021).

High PAH levels in smoked fish are due to the lignin content of the material used as fuel in the smoking process (Asamoah *et al*., 2021). The lignin content in corn cobs is reported to be about 17 – 21% (Thangavelu *et al*., 2018; Olajuyigbe *et al*., 2019; Gandam *et al*., 2022). The lignin content of coconut shell was approximately 59.5% (Wang & Sarkar, 2018), while that of coconut wood was about 50% on a dry weight basis (Anuchi *et al*., 2022). Lignin is reported to absorb PAH compounds because PAH compounds can bind well with lignin (Oliveira *et al*., 2019). During pyrolysis and gasification processes, lignin components react to produce aromatic tar and coke, which can form PAH compounds (Zhou *et al*., 2014; Kawamoto, 2017). Therefore, PAH compounds in smoked fish smoked using corncobs are lower than those smoked with shells and coconut wood.

Sensory assessment

A sensory assessment is carried out to evaluate the panelist's preference level, including appearance, aroma, taste, and texture. A sensory assessment must determine the quality of the smoked fish product and ensure it meets the applicable standards (Hadanu & Lomo, 2019). The sensory characteristics of smoked fish products affect consumer acceptance and preference. Sensory assessment by panelists helps to identify the sensory attributes that are most appealing to consumers, allowing manufacturers to

customize products according to consumer assessment (Ekelemu *et al*., 2021). Indonesian National Standard No. 2725:2013 regarding smoked fish requires a minimum sensory assessment of 7 (Score 1-9) (Indonesia Standardization Agency, 2013). The results of the panelists' assessment of *julung julung* smoked fillet from each treatment can be seen in Fig. 7.

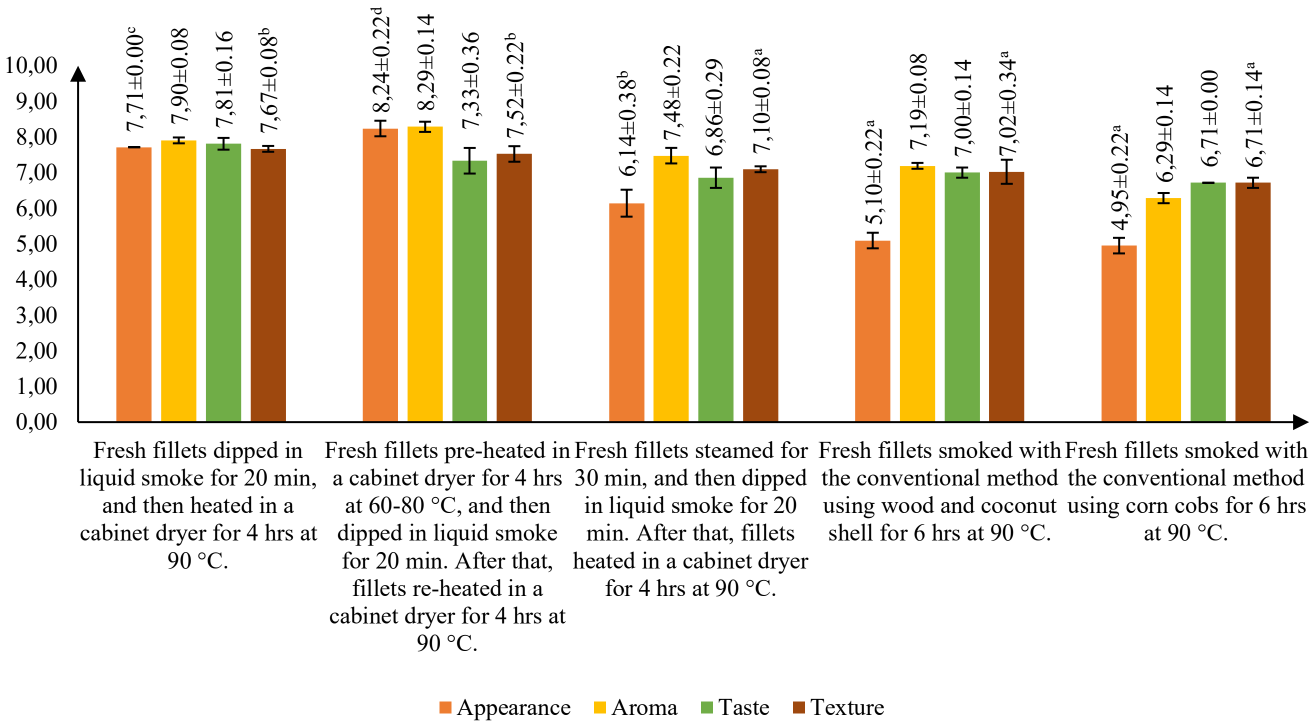

Fig. 7. Sensory assesment of *julung julung* smoked fillet

Appearance: Fig. 7 shows the analysis of the variance of the sensory assessment of the appearance of smoked *julung julung* fish fillets with different smoking method treatments affecting the appearance of smoked fish ($p<0.05$). The panelists' assessment of the appearance of smoked fish fillets ranged from 4.95 to 8.24, with the highest panelists' assessment in treatment B. Based on the requirements of the Indonesian National Standard, only treatments A and B met the minimum panelist assessment requirement of 7. The moisture content factor is thought to have influenced the panelists' assessment of the appearance of smoked fish, so panelists less favored treatment C with steaming. Moisture content can affect the physical properties of fish, such as the appearance and texture of smoked fish (Baten *et al*., 2020a). Smoked fish with high moisture content will make the color of smoked fish look paler (Flick, 2010).

Treatments D and E were less favored by panelists, presumably because conventional smoking methods produce darker products (dark brown), less bright and less shiny. Smoking fish with liquid smoke has smoked fish products with a golden color and clean and shiny surface (Berhimpon *et al*., 2018). Another factor that affects the appearance of smoked fish is the length of the smoking process. The longer the

smoking process, the darker the color will be. The time of the smoking process for smoked fish using the liquid smoke method is shorter than the conventional method (Puke & Galoburda, 2020; Baten *et al*., 2020b). The smoking process can change the color of fish, giving it a distinctive golden color due to the interaction of carbonyls with amino components on the surface of the meat. The color and appearance of smoked fish also positively correlate with phenol content. Phenol compounds in smoke interact with amino acid components in fish meat, producing a distinctive golden color in smoked fish (Montazeri *et al*., 2013).

Aroma: Fig. 7 shows the analysis of the variance of the sensory assessment of the aroma of smoked fish fillets *julung julung* with different smoking method treatments that did not affect the appearance of smoked fish ($p>0.05$). However, the aroma sensory assessment of treatments A, B, C, and D still meets the minimum assessment of the Indonesian National Standard on smoked fish. The sensory assessment of smoked fish aroma was lowest in treatment E. Previous studies have reported that smoking fish with corn cobs as a fuel source resulted in the lowest aroma scores, indicating less aromatic smoked fish (Asmara *et al*., 2022). The aroma and taste of smoked fish are strongly influenced by the time of smoking (Tahir *et al*., 2020). This assessment indicates that the time of the smoking process of 4 and 6 hours in all treatments has not influenced the taste and aroma of smoked fish.

Taste: Fig. 7 shows the analysis of the variance of the sensory assessment of the taste of smoked fish fillets *julung julung* with different smoking method treatments that did not affect the appearance of smoked fish ($p>0.05$). The taste of smoked fish is influenced by the time of the smoking process. Previous studies have reported that longer smoking time can improve the sensory attributes of taste (Baten *et al*., 2020b). This result indicates that the 2-hour time difference between the liquid smoke smoking treatment and the conventional smoking method has not affected the taste of smoked fish. A distinctive smoked taste without bitterness is a criterion for assessing the taste of good quality smoked fish (Sukowati *et al*., 2021).

Texture: Fig. 7 shows the analysis of variance of the sensory assessment of the texture of smoked fish fillet *julung julung* with different smoking method treatments giving effect to the texture of smoked fish ($p<0.05$). Panelists' assessment of the texture of smoked fish *julung julung*, ranged from 6.71 – 7.67, with the highest panelist assessment in treatments A and B. Duncan's test analysis showed that treatments A and B differed from treatments C, D, and E. This result was thought to be because the fish fillets were dipped in liquid smoke, giving a dry, compact, and tender texture. Whereas in treatment C (fish fillets subjected to steaming), the texture of the smoked fillets was rather sticky and not solid. Treatments D and E produced the texture of smoked fish fillets which were less dense and not compact. The texture of smoked fish is negatively correlated with its moisture content. The higher the moisture content in smoked fish, the softer and less firm the texture (Chan *et al*., 2022). High moisture content in smoked fish products results in less dense fish muscle fibers (Chang *et al*., 2021).

A good texture of smoked fish is compact, moderately elastic, and not sticky (Sulistijowati *et al*., 2021). The Indonesian National Standard requires that the texture of smoked fish is dense and compact and the tissue is very tight (Indonesia Standardization Agency, 2013). Based on consumer surveys, the preferred texture of smoked fish is dense, slightly wetter, and easy to chew, and there is a sensation of liquid in the mouth when chewed (Ticoalu *et al*., 2019).

Conclusion

Based on the findings of this study, it can be concluded that treatment B (Fresh fillets pre-heated in a cabinet dryer for 4 hours at 60 – 80 ºC and then dipped in liquid smoke for 20 minutes. After that, the fillets were re-heated in a cabinet dryer for 4 hours at 90 ºC.), when compared to the conventional smoking treatment based on total volatile bases, moisture content, water activity, pH value, phenol level, polycyclic aromatic hydrocarbon content, and sensory assessment. In general, fish smoking dipped in liquid smoke from corn cob produced a better quality of smoked fish than the conventional smoking method. It is necessary to evaluate different smoking times on smoked fish fillets of *julung julung* with liquid smoke method from corn cob.

Acknowledgement (bold)

Author contributions

Conflicts of interest

The authors declare that there is no conflict of interest.

Highlights

- Liquid smoke from corn cobs can increase total phenols. Fillets dried and soaked in liquid smoke.

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Reviewer 1#

Detailed comments;

Line 179. Change Data analysis to Statistical analysis.

State replicates for all tests.

In figures just put Duncan's letters on the bars. There is no need to mention the numbers on the bars.

Provide the proper abbreviations for the treatments in figures, tables as well as the manuscript text.

Duncan's letters are misattributed, the first letters must show the biggest numbers. Also, Duncan's letters should be added in Table 2.

Sensory evaluation data should be present as a radar plot.

Delete methods from the conclusion section.

The last line of the abstract and conclusion should be revised and the best method mentioned.

Characteristics of *Julung Julung* Smoked Fish Fillets (*Hemiramphus* sp.) using Liquid Smoke from Corn Cobs Waste

Abstract

Corn cobs can be utilized as a shell ingredient for liquid smoke. Liquid smoke can be used for the smoked fish industry in North Sulawesi, Indonesia. However, the utilization of liquid smoke for the smoked fish industry has yet to be optimal. This study aimed to obtain the best smoking method for *julung julung* fish fillets (*Hemiramphus* sp.) using liquid smoke from corn cob waste. The treatment in this study was to apply liquid smoke from corn cob waste in the smoking process, which was compared with the conventional method (using shell, coconut wood, and corn cob). Parameters to determine the quality of smoked fish were total volatile bases (TVB), moisture, water activity, pH, phenol levels, polycyclic aromatic hydrocarbon (PAH), and sensory assessment. The results showed that the TVB of smoked *julung julung* fish fillets ranged from 19.83 – 32.27 mg N/100g. The moisture ranged from 14.10 – 30.57%. Water activity ranged from 0.46 – 0.82. pH ranged from 4.60 – 5.81. Phenol levels ranged from 4.42 – 16.11 mg/g. PAH levels are still below the standard required in the Indonesian National Standard. Panelists rated neutral to really like the appearance, aroma, taste, and texture of smoked fish. From these results, it can be concluded that the best treatment is fresh fillets preheated in a cabinet dryer for 4 hours at 60 – 80 ºC and then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated in a cabinet dryer for 4 hours at 90 ºC.

Keywords: ~~Coconut~~, Indonesia, ~~Shell~~, North Sulawesi, Wood

Commented [u1]: Corn cobs
Liquid smoke

Introduction

Corn is the second most widely grown crop in Indonesia after rice. Indonesia is the 7th corn producer in the world, with more than 12 million tons of corn produced in 2020 (Nazmi *et al*., 2021). North Sulawesi is one of the contributing regions that produce corn in Indonesia. Considerable corn production will also produce waste, especially corn cobs, which are generally thrown away and burned by most Indonesians (Cahyadi *et al*., 2021).

One of the corn cobs was used to make liquid smoke. Previous research reported that corn cobs can produce liquid smoke, a by-product of the pyrolysis of corn cob waste. The yield of liquid smoke from corn cobs is about 28.37%, with a pH value of 3.5 (Sriharti *et al*., 2020). The particle size of corn cobs affects the yield of liquid smoke, with smaller particle sizes resulting in higher yields (Aladin *et al*., 2018). Another study reported a phenolic content of 335 mg/L in liquid smoke produced from corn cobs (Swastawati *et al*., 2007).

Liquid smoke is a natural product made from the condensation of smoke from burning wood (Andy *et al*., 2021). Liquid smoke is commonly used as a flavoring in food to provide a smoked flavor without the food undergoing the actual smoking process (Sari *et al*., 2006). Using liquid smoke in food will save time, energy, and labor and reduce production costs (Krah *et al*., 2019). In addition, using liquid smoke in food can speed up and standardize the smoking process, adding flavor and microbiological safety

while reducing production time (Indiarto *et al*., 2020). Generally, various types of hardwood were used to manufacture liquid smoke (Diatmika *et al*., 2019). The quality of the liquid smoke produced may vary depending on the type of wood used (Budaraga *et al*., 2016). In addition to imparting specific flavors and aromas to foods (Maulina *et al*., 2020), liquid smoke is reported to be antimicrobial (Septana *et al*., 2020) and antioxidant (Budaraga & Putra, 2021).

The conventional smoking processes generally use hardwood as fuel, such as melon wood (Umar *et al*., 2018), mangrove wood (Cissoko *et al*., 2020), teak wood (Daramola *et al*., 2020), coconut shells, and coconut husks (Nugroho *et al*., 2018). Corn cobs have also been reported to be used in the fish smoking process (Sukowati, 2023). According to literature, traditional fish smoking in North Sulawesi, Indonesia, uses smoke from burning coconut shells or wood charcoal (Landangkasiang *et al*., 2017; Primalasari *et al*., 2019; Saediman *et al*., 2021). Smoked fish processing has been known for a long time because of its traditional processing techniques, simplicity, ease of implementation, and low cost (Islamiyah, 2021). In North Sulawesi, Indonesia, smoked fish commonly known as *fufu* is traditionally made from skipjack (*Katsuwonus pelamis*); *julung julung* (*Hemiramphus far*) (Berhimpon *et al*., 2018); and scad fish (*Decapterus* spp.) known as *pinekuhe* (Ansar & Ijong, 2021). The smoked fish product of *julung julung* is a typical product from North Sulawesi and Gorontalo, commonly called *roa* or *galavea* (Azis & Akolo, 2020).

The study's reported that the conventional smoking process has disadvantages such as smoking time, concentration of carcinogenic substances, temperature, and inconsistent product quality (Racovita *et al*., 2020). Conventional smoking of food products has been shown to produce carcinogenic components such as polycyclic aromatic hydrocarbons (Jinadasa *et al*., 2020). Benzo(a)pyrene ($C_{20}H_{12}$) is one of the carcinogenic compounds produced from traditional smoking products (Jinadasa *et al*., 2020). One method to reduce carcinogenic compounds in smoked fish products is to use liquid smoke in the smoking process (Nithin *et al*., 2020; Xin *et al*., 2021).

The utilization of liquid smoke in the fish smoking industry in North Sulawesi is very likely to be developed. North Sulawesi was reported to have both large-scale and domestic fish-smoking industries (Dotulong *et al*., 2018; Primalasari *et al*., 2019). Making liquid smoke is relatively simple, so it will be readily accepted by fish-smoking industry players (Ali & Al Fiqri, 2020). Previous research reported that using liquid smoke in smoked fish products resulted in good quality in terms of appearance, flavor, and aroma (Rizal *et al*., 2020). In addition, using liquid smoke in smoked fish products can add nutritional value and durability, making it possible to reach a wider market area (Ali *et al*., 2021).

Previous research reported that smoked fish dipped in liquid smoke for 15 minutes had a total plate count value of 4.7×10^4 CFU/g on day six and moisture content below 60% during storage (Suroso *et al*., 2018). Dipping fish in liquid smoke with a concentration of 15% and a dipping time of 60 minutes showed a bacterial colony of 2.12×10^2 CFU/g (Ali *et al*., 2021). The Indonesian National Standard on

smoked fish requires a maximum total plate count of 5×10^4 CFU/g and a maximum moisture content of 60% (Indonesia Standardization Agency, 2013). Another study reported that smoked fish processed with liquid smoke had a moisture content of 47.63%, phenol content of 12.62%, and pH of 4.8 (Berhimpon *et al*., 2018). ~~The results of these studies indicate that liquid smoke is good to apply to smoked fish products.~~ This study characterized smoked fish fillets of *julung julung* (*Hemiramphus* sp.) produced with liquid smoke from corn cobs waste. The study was conducted by dipping the fish fillets of *julung julung* with liquid smoke, with drying and steaming treatments on the fish fillets. This study aims to obtain the best smoking method for fish fillets using liquid smoke from corn cobs waste based on the assessment of total volatile bases, moisture content, water activity, pH value, phenol content, polycyclic aromatic hydrocarbon content, and sensory assessment.

Materials and methods

The main materials of the study were *julung julung* fish (*Hemiramphus* sp.), corn cob (*Zea mays* L.) from Gangga Island in Likupang, North Sulawesi, Indonesia. This study includes the preparation of liquid smoke from corn cobs waste, preparing fish (weeding and filleting), and treating fish fillets by dip in liquid smoke. Fresh *julung julung* fish was obtained from traditional fishermen in Manado, North Sulawesi, Indonesia (Fig. 1). The fish was placed in a cool box and given ice in a ratio of 1:1 (ice:fish) (Mishra *et al*., 2021). Then, the fish was washed, weeded, and filleted. The cleaned fillets were dipped in a 5% salt solution for 30 minutes. The concentration of liquid smoke from corn cob waste was 0.8% (Berhimpon *et al*., 2018).

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Fig. 1. *Julung julung* (*Hemiramphus* sp.)

The treatment in this study was applying liquid smoke from corn cobs waste to the smoking process of *julung julung* fish fillets, which was compared with the conventional method (using coconut wood, coconut shells, and corn cobs).

(A) : Fresh fillets were dipped in liquid smoke for 20 minutes and then heated in a cabinet dryer for 4 hours at 90 ^{o}C.

(B) : Fresh fillets pre-heated in a cabinet dryer for 4 hours at 60 – 80 ^{o}C, and then dipped in liquid smoke for 20 minutes. After that, fillets were re-heated in a cabinet dryer for 4 hours at 90 ^{o}C.

(C) : Fresh fillets were steamed for 30 minutes and then dipped in liquid smoke for 20 minutes. After that, fillets were heated in a cabinet dryer for 4 hours at 90 ^{o}C.

(D) : Fresh fillets were smoked with the conventional method using wood and coconut shell for 6 hours at 90 ^{o}C.

(E) : Fresh fillets were smoked with the conventional method using corncob for 6 hours at 90 ^{o}C.

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Liquid smoke manufacturing process

Making liquid smoke refers to modified research (Aladin *et al.*, 2018). Modifications were made to the equipment used in the pyrolysis process. Dry corn cobs were weighed to determine fuel efficiency. Corn cobs were gradually burned into the combustion furnace. The furnace was closed to prevent smoke from escaping from the tank. The smoke generated from the combustion flows through a pipe connected to a storage tank covered with ice cubes. The smoke that passes through the pipe will become cold, so condensation occurs, turning the smoke into liquid. The smoke that has been formed is collected into a container attached to the end of the pipe. The liquid smoke obtained is then allowed to settle the tar formed for three weeks and filtered to obtain clear liquid smoke.

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Total volatile bases assay

Total volatile base (TVB) assay refers to research by Tambunan & Chamidah (2021). TVB assay aims to determine the amount of volatile base compounds formed from protein breakdown—the principle of TVB analysis, namely by evaporating volatile base compounds at room temperature for 24 hours. The compound is then bound with boric acid and titrated with an HCl solution. After the boric acid solution was stored in the inner chamber of the Conway cell containing the blank (V_0) and the filtrate (V_1), the boric acid solution was titrated with 0.02 N HCl so that the boric acid solution changed color to pink. TVB levels in the smoked fish meat were expressed as mg N/100g.

$$TVB\left(\mathrm{mg}\frac{\mathrm{N}}{100\mathrm{g}}\right) = \frac{(V_1 - V_0) \times N.HCl \times dilution \times 100}{sample\ weight\ (g)}$$

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Moisture content assay

The moisture content by method of Indonesia National Standard (Indonesia Standardization Agency, 2006a). An empty porcelain cup is weighed (A). Then, the sample was weighed to ± 2 g in a porcelain cup of known mass (B). The samples were dried in an oven at 105°C for 20 hours. The samples were then cooled in a desiccator, after which the samples in the cup were weighed (C). Moisture content was expressed as %.

$$Moisture\ (\%) = \frac{B\ (g) - C\ (g)}{B(g) - A\ (g)} \times 100\%$$

Water activity (Aw) assay

Water activity assay used an Aw meter (Rotronic, HygroPalm 23-AW-A). The Aw meter was set up at room temperature for 2 hours. The water activity of smoked fish was measured by placing the sample into a sample container and conditioning it for 30-60 minutes. The Aw meter sensor is contacted with the sample in the container. Then the water activity (Aw) value can be read on the Aw meter panel (Saputra *et al*., 2014).

pH assay

The sample weighed as much as 30 g, then 400 mL of distilled water was added at 28 ºC. The sample was homogenized with a magnetic stirrer to be evenly stirred. The pH meter electrode was inserted into the sample solution. The data taken in this test is the pH value read by the pH meter (Hanna Instruments HI99192) (Lekahena & Jamin, 2018).

Phenol level assay

Samples were extracted by adding 1 mL ethanol p.a., 5 mL distilled water, and 0.5 mL Folin Ciocalteu 50%, homogenized, and allowed to stand for 5 minutes. Then added, 1 mL of 5% Na_2CO_3 and left in the dark for ± 60 minutes with gallic acid as a standard. The absorbance value was read on a spectrophotometer (Thermo Scientific Genesys 50) with a wavelength of 725 nm. Total phenolics were expressed as mg/g (Indonesia Standardization Agency, 2004; Muliadin *et al*., 2022).

$$Total\ Phenol\ (\frac{mg}{g}) = \frac{Gallic\ acid\ equivalence\ \left(\frac{mg}{L}\right) \times Volume\ (L)}{Sample\ weight\ (g)}$$

Polycyclic aromatic hydrocarbon (PAH) assay

PAH content testing was carried out based on the instructions Basak *et al*. (2010). A 5 g sample was weighed and collected in a 100 ml flask. Then 5 mL of 50% KOH solution, 75 mL of methanol, 1 mL

of internal standard solution (9,10 dimethylantracene), and some boiling stones were added. After that, the mixture was boiled for 4 hours in a soxhelet. The liquid phase was transferred to a separatory funnel and extracted with 100 mL of n-hexane with shaking for 3 min. The MeOH:KOH phase was drained and discarded. The combined n-hexane phase was rinsed twice, each with 50 mL of H_2O-MeOH (8:1) and 50 mL of water. The methanol-water and water phases were dried and discarded. The organic phase was concentrated in a rotary evaporator (40°C) to a volume of about 10 mL. The concentrated extract was dried under nitrogen gas.

After that, the silica gel column was cleaned by dissolving 15 g of silica gel in n-hexane and transferred to a column (200 mm long; diameter: 12 mm). Then, 1 g of anhydrous sodium sulfate was added. The concentrated sample extract was transferred to the column and eluted with 1 mL of n-hexane. A stock solution containing 0.1 mg mL^{-1} of 9,10 dimethylantracene was dissolved in n-hexane and stored at 4°C in a volumetric flask (with a glass stopper) wrapped in aluminum foil to avoid possible light degradation. Working standard solutions were prepared from the stock solutions. Analysis of PAH compounds used a Hewlett-Packard 1100 HPLC equipped with an Agilent-110 fluorescence detector. The injection volume was 10 µL. Mobile phase gradient (acetonitrile-water): 80% acetonitrile + 20% water with a flow rate of 1 ml min^{-1} and a wavelength of 270 nm. The external standard mixture was used for PAH concentration calculation.

Sensory assessment assay

Sensory assessment assay refers to Indonesia National Standard (Indonesia Standardization Agency, 2006b). The sensory assessment was based on a hedonic scale (Table 1). Smoked fish samples were placed on a plastic plate with a glass of water, coded, and presented to 30 panelists randomly under light. The parameters observed were appearance, aroma, taste, and texture. Panelists rated acceptance of the samples on a scale of 1 – 9.

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Table 1. Score sheet of hedonic assessment

Specification	Score
Really like it	9
Really like	8
Like	7
Rather like	6
Neutral	5
Rather dislike	4
do not like	3
Very dislike	2
Really don't like it	1

Data analysis

Data analysis was performed using Statistical Product and Service Solutions (SPSS) version 20.0. Statistical analysis of data was performed based on one-way analysis of variance (ANOVA) using a significance level of ($P<0.05$). Specific group differences were determined using Duncan's test.

Results and discussion

Liquid smoke production was carried out using a simple liquid smoke distillation device. This process consumed 17 kg of corn cobs and 12 kg of crushed ice. Liquid smoke from corn cobs is more accessible than coconut shells or wood (Maulina & Karo, 2021). Using coconut shells or wood as fuel will form a flame, thus reducing the volume of smoke in the furnace (Kabir Ahmad *et al*., 2022). The smoked *julung julung* were analyzed for TVB, moisture content, water activity, pH value, phenol content, PAH content, and sensory assessment.

Total volatile bases

The total volatile base (TVB) value is measured to determine the quality of the smoked fish. TVB value is one of the parameters used to determine the decline in fish quality and measures the amount of volatile base compounds formed due to protein degradation (Castro *et al*., 2006). Volatile bases formed in fish muscle tissue mainly consist of ammonia, trimethylamine (TMA), and dimethylamine (DMA) (VELP Scientifica, 2013). TVB value is an essential characteristic for the quality assessment of seafood products and is emerging as the most commonly used chemical parameter to assess the palatability of seafood (Rasulu *et al*., 2020). The results of *total volatile bases* on smoked fish fillets of *julung julung* are presented in Fig. 2.

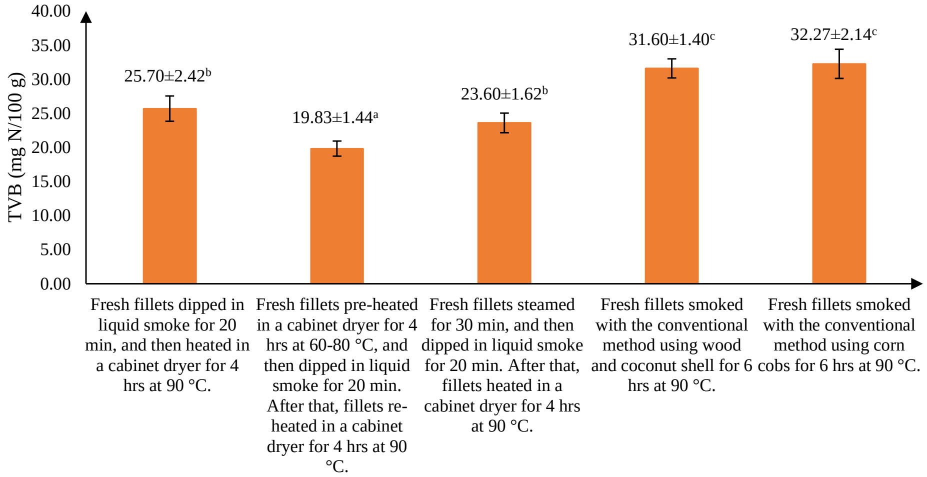

Fig. 2. Total volatile bases of *julung julung* smoked fillet

Fig. 2 shows the analysis of the variance of smoked fish fillets with different smoking method treatments affecting changes in TVB content ($p<0.05$). The TVB content of smoked fish fillets ranged from 19.83 – 32.27 mg N/100g, with the lowest TVB value in treatment B. The treatment of the smoking method with liquid smoke from corn cob waste (treatments A, B, and C) had lower TVB levels when compared to the conventional smoking method (treatments D and E). A literature search has not found why liquid smoking has a lower TVB value than conventional smoking methods. However, one possibility is that the liquid smoke smoking method allows the smoke components to be absorbed into the fish meat better than the conventional smoking method. Better absorption of liquid smoke phenol components in fish meat can inhibit and control microbial growth that causes a decrease in the quality of smoked fish (Santoso *et al*., 2015). A literature study showed that the average TVB value of smoked mackerel (*Euthynnus affinis*) treated with liquid smoke addition ranged from 24.63 – 28.38 mg N/100g (Hardianto & Yunianta, 2015). Another study reported smoked mackerel with a smoking process using rubber wood liquid smoke, producing smoked fish products with lower TVB values (Suroso *et al*., 2018).
Duncan's analysis showed a difference between treatments B, A – C, and D – E on the TVB value of smoked fish fillets. The analysis of variance indicates that the drying process of smoked fish fillets before immersion into liquid smoke can maintain the quality of smoked fish (Kaparang *et al*., 2013). Duncan's analysis also showed that treatments A – C and D – E had no difference in TVB value, indicating that the moisture content between the two treatments was not different. TVB value is closely related to moisture content (Rasulu *et al*., 2020).

The European standard (European Market Observatory for Fisheries and Aquaculture, 2020) states that the limit range of TVB value for smoked fish is 25 – 35 mg N/100 g. All treatments' TVB value of the smoked fish fillets still met the European standard. Previous studies reported TVB values of fresh fish fillets of 8.70 ± 0.86 mgN/100g (Moosavi-Nasab *et al*., 2021) and 12.94 ± 0.92 mg N/100g (Bouzgarrou *et al*., 2020). Messina *et al*. (2021) reported that smoked fish fillets that underwent two drying processes had a TVB value of <20 mg N/100g. Another study also reported the TVB value of smoked fish fillet samples with hot smoking and cold smoking, which amounted to 17.80 ± 0.17 and 18.95 ± 0.20 mg N/100g, respectively (El-Lahamy *et al*., 2019). Aydin *et al*. (2020) reported a TVB value of smoked fish with hot smoking of 11.82 ± 0.05 mg N/100g.

Moisture content

Moisture content is an important parameter to determine the quality of smoked fish produced. The moisture content contained in smoked fish can affect the shelf life of smoked fish because water in food is a medium for microbes to grow (Kaban *et al*., 2019). The smoking process was reported to reduce the moisture content of fresh fish to a range between 9 and 17% (Sikoki & Aminigo, 2002). The moisture content of smoked fish fillets is presented in Fig. 3.

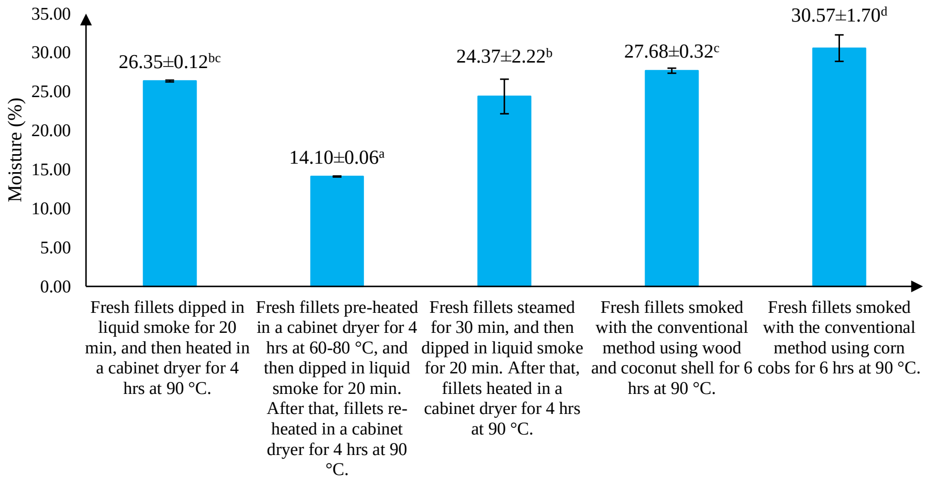

Fig. 3. Moisture content of *julung julung* smoked fillet

Fig. 3 shows the analysis of the variance of smoked *julung julung* fish fillets with different smoking methods treatment affects moisture content ($p<0.05$). The moisture content of smoked *julung julung* fish fillets ranged from 14.10 – 30.57%, with the lowest moisture content in treatment B. The

moisture content value in smoked fish products from all treatments still meets the Indonesian National Standard No. 2725:2013 on smoked fish, which is a maximum of 60% (Indonesia Standardization Agency, 2013). Darianto *et al*. (2018) reported that the smoking process can reduce the moisture content of fish to below 40%, which can help preserve it longer. The treatment of the smoking method with liquid smoke with corn cob (Treatment A, B, C) has a lower moisture content when compared to the treatment of the conventional smoking method (Treatment D and E). This result is because the smoking chamber is not fully enclosed in the conventional smoking method, so the heat generated could be more optimal. Suboptimal heat can increase moisture content and cause the moisture content of smoked fish to decrease only slightly (Amos & Paulina, 2017). Whereas in the liquid smoke smoking method, temperature and humidity can be controlled better so that the moisture content of the product can be reduced efficiently (Salindeho & Lumoindong, 2017).

Duncan's analysis showed a difference between treatment B, treatment A – C, and treatment D – E on the moisture content of smoked fish fillets. The results of the variance study indicate that the drying process of smoked fish fillets before the dip in liquid smoke in treatment B can help reduce the moisture content. This study's results are from previous research, which also reported a significant decrease in moisture content in smoked fish products with two times drying treatment (Messina *et al*., 2021). Duncan's analysis also showed that treatments A and C were not different because steaming in treatment C will increase the moisture content of the product (Salmatia *et al*., 2020). The conventional smoking method with corn cob fuel has the highest moisture content; this result is the conventional smoking process with corn cobs, and the heat generated is lower than smoking using wood (Asmara *et al*., 2022).

Water activity (Aw)

Water activity is one of the essential parameters in the quality of smoked fish. Water activity (Aw) is expressed as the ratio of the vapor pressure in the food to the vapor pressure of pure water, and it predicts whether water tends to move from the food product into the cells of microorganisms that may be present. A well-smoked fish has a water activity of <0.50 and a moisture content between 15 and 25% to inhibit the growth of pathogenic microorganisms in smoked fish products (Mondo *et al*., 2020). According to British Columbia Centre for Disease Control (2013), the maximum water activity value for smoked fish is 0.97. Water activity correlates with the moisture content of a smoked fish product and is two important factors affecting food safety and quality (Fitri *et al*., 2022). The water activity values of smoked fish fillets can be seen in Fig. 4.

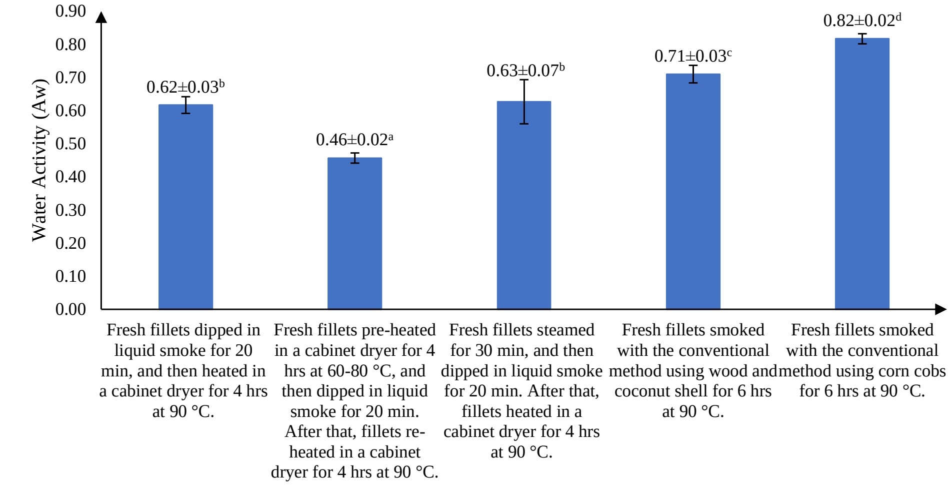

Fig. 4. Water activity of *julung julung* smoked fillet

Fig. 4 shows the analysis of the variance of smoked fish fillet *julung julung* with different smoking method treatments affecting water activity ($p<0.05$). The water activity value of smoked fish fillet ranged from 0.46 – 0.82, with the lowest water activity in treatment B. The treatment of the smoking method with corn cob liquid smoke (Treatment A, B, C) had a lower water content when compared to the treatment of the conventional smoking method (treatment D and E).

The water activity value of fish meat can vary depending on the cooking method, temperature, and duration of heating (Oliveira *et al*., 2017; Zhang *et al*., 2023). Therefore, the factor of fish fillet treatment before immersion in liquid smoke caused the difference in water activity value in treatment B compared to treatments A and C. The water activity value is directly proportional to the water content value of each treatment. The heating process can reduce the moisture content of fish meat (Kiczorowska *et al*., 2019), thus affecting the water activity of fish meat (Gómez *et al*., 2020).

pH

The pH value of smoked fish is an essential factor affecting its quality. The pH value will decrease with increasing smoking time (Baten *et al*., 2020b). The analysis of the variance of smoked fish fillets *julung julung* with different smoking method treatments affects the pH value ($p<0.05$). The pH of smoked fish fillet ranged from 4.60 to 5.81 (Fig. 5). The treatment of the smoking method with corn cob liquid smoke (treatments A, B, and C) had a lower pH value when compared to the conventional smoking method (treatments D and E). Swastawati *et al*. (2022) also reported that the pH value of smoked barracuda fish with the liquid smoke method was significantly lower than that of the conventional

method. Another study also reported that using liquid smoke can reduce pH caused by the condensation of organic acids in the smoking process (Puke & Galoburda, 2020). The pH value of smoked fish correlates with the phenol content contained in the smoke component, and an increase in phenol content causes a decrease in pH (Berhimpon *et al*., 2018).

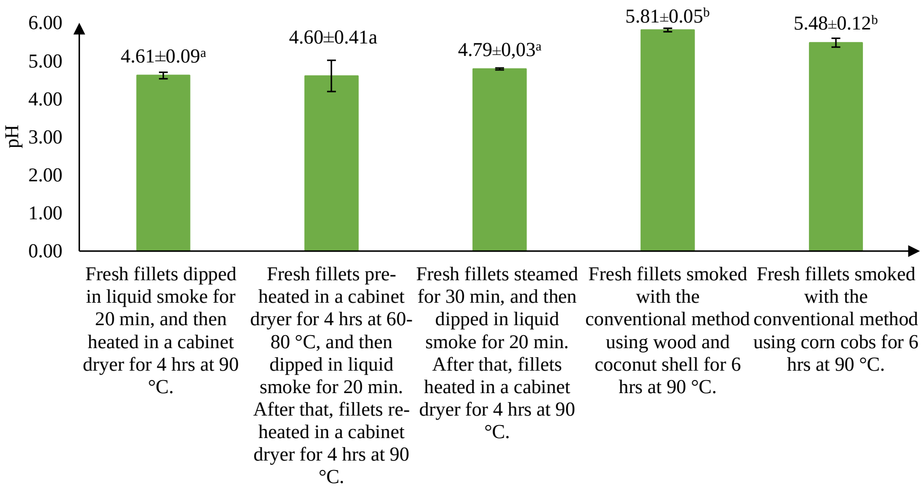

Fig. 5. pH of *julung julung* smoked fillet

Phenol level

Phenol is a compound found in wood smoke. Phenol compounds enter food through diffusion and capillary action, affecting taste, color, and aroma and extending shelf life (Remy *et al*., 2016). In addition, phenol compounds are also reported to act as antioxidants that can prevent rancidity in fish meat (Sérot *et al*., 2004). Leksono *et al*. (2020) also noted that the higher the phenol level in smoke, the stronger the flavor and aroma of smoked fish. The analysis of the variance of smoked fish fillets julung julung with different smoking method treatments affecting phenol level ($p<0.05$). The phenol level of smoked fish fillet ranged from 4.42 to 16.11 mg/g (Fig. 6). The treatment of the smoking method with corn cob liquid smoke (treatments A, B, and C) had higher phenol level when compared to the conventional smoking method (treatments D and E).

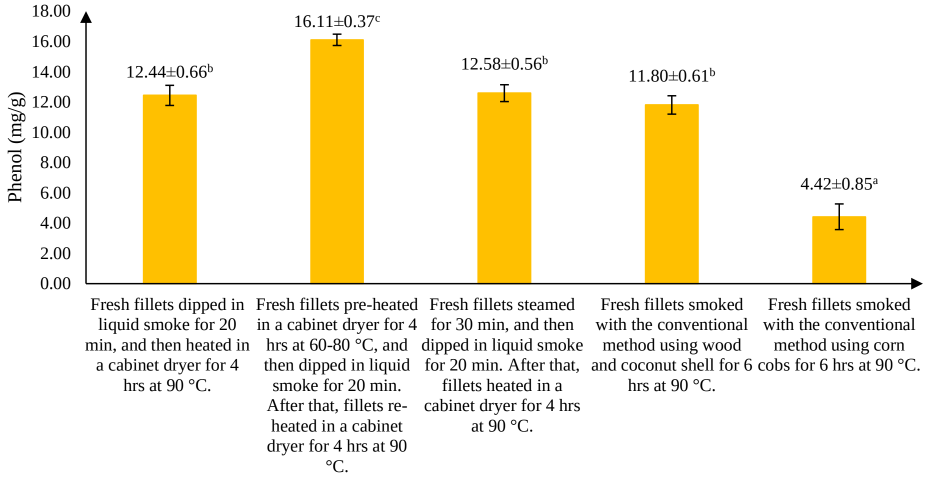

Fig. 6. Phenol level of *julung julung* smoked fillet

Duncan's analysis showed phenol levels in treatments A, C, and D were not different. Previous research reported that the drying treatment of fish meat can increase phenolic compounds in smoked fish products (Sérot *et al*., 2004). The high phenol content in treatment B is thought to be because the pre-heated process causes the fish meat to lose water so that when dipped in liquid smoke, the fish fillets absorb a large amount of liquid smoke. Previous studies have reported that when the fish surface is dried, there is less smoke condensation than products smoked at lower temperatures. The results of this study indicate that a dry fish surface allows for better absorption of smoke components (Belichovska *et al*., 2019).

The study reported phenol levels in liquid smoke from corn cob waste of 0.335 mg/g (Swastawati *et al*., 2007) and 2.55% (Leviyani *et al*., 2019). The phenol level of smoked fish with the conventional method of smoking with corn cobs is lower due to the high acid content, which can potentially reduce the phenol content of smoked fish (Swastawati *et al*., 2012). Anggraini & Nurhazisa (2017) reported that the phenol content of liquid smoke from coconut shells was 3.04%, while liquid smoke from corn cob was 1.38%.

Polycyclic aromatic hydrocarbon (PAH) levels

Polycyclic aromatic hydrocarbons are a group of organic compounds formed by incomplete combustion, such as wood. Therefore, the type of fuel used in the fish smoking process affects the PAH content of smoked fish (Jinadasa *et al*., 2020). One of the carcinogenic PAHs, benzo(a)pyrene, is a carcinogenic marker in smoked fish products (Stołyhwo & Sikorski, 2005). Polycyclic aromatic hydrocarbon (PAH) levels in smoked fish fillets can be seen in Table 2.

Table 2. Polycyclic aromatic hydrocarbon levels in *julung julung* smoked fillet

Polycyclic Aromatic Hydrocarbon	Smoked *Julung Julung* fish (µg/kg)					Corn Cob Liquid Smoke 0.8%(µg/kg)
	A	B	C	D	E	
Benzo(a)pyrene	nd	nd	nd	1.5	0.2	0.5
Benzo(b)fluoranthene	1.2	1.0	1.2	2	1.8	2.5
Benzo(a)anthracene	nd	nd	nd	0.5	nd	0.2
Benzo(g)perylene	0.4	0.2	0.4	1.0	0.8	1.0

nd = not detected

Table 2 shows that the PAH levels in smoked fish are still below the standard required in the Indonesian National Standard No. 2725 of 2013 on smoked fish, which is benzo(a)pyrene max 5 µg/kg (Indonesia Standardization Agency, 2013). The polycyclic aromatic hydrocarbon compound regulated in the Indonesian National Standard is only benzo(a)pyrene. Stołyhwo & Sikorski (2005) reported benzo(a)pyrene levels in smoked fish with hot and cold smoking processes ranging from 0.05 to about 60 µg/kg. Other studies have reported benzo(b,k)fluoranthene levels in salmon and rainbow trout ranging from 1.83 to 9.55 µg/kg, while benzo(g,h)perylene levels in salmon were 0.44 µg/kg (Basak *et al*., 2010). Berhimpon *et al*. (2018) also reported benzo(a)pyrene levels in smoked skipjack fish of 0.25 µg/kg. The level of benzo(a)pyrene in smoked barracuda was reported to be 0.32 µg/kg, benzo(b)fluoranthene 0.35 µg/kg, benzo(k)fluoranthene 0.21 µg/kg, benzo(a)anthracene 0.44 µg/kg, and benzo(g,h)perylene 2.56 µg/kg (Asamoah *et al*., 2021).

High PAH levels in smoked fish are due to the lignin content of the material used as fuel in the smoking process (Asamoah *et al*., 2021). The lignin content in corn cobs is reported to be about 17 – 21% (Thangavelu *et al*., 2018; Olajuyigbe *et al*., 2019; Gandam *et al*., 2022). The lignin content of coconut shell was approximately 59.5% (Wang & Sarkar, 2018), while that of coconut wood was about 50% on a dry weight basis (Anuchi *et al*., 2022). Lignin is reported to absorb PAH compounds because PAH compounds can bind well with lignin (Oliveira *et al*., 2019). During pyrolysis and gasification processes, lignin components react to produce aromatic tar and coke, which can form PAH compounds (Zhou *et al*., 2014; Kawamoto, 2017). Therefore, PAH compounds in smoked fish smoked using corncobs are lower than those smoked with shells and coconut wood.

Sensory assessment

A sensory assessment is carried out to evaluate the panelist's preference level, including appearance, aroma, taste, and texture. A sensory assessment must determine the quality of the smoked fish product and ensure it meets the applicable standards (Hadanu & Lomo, 2019). The sensory characteristics of smoked fish products affect consumer acceptance and preference. Sensory assessment by panelists helps to identify the sensory attributes that are most appealing to consumers, allowing manufacturers to

customize products according to consumer assessment (Ekelemu *et al*., 2021). Indonesian National Standard No. 2725:2013 regarding smoked fish requires a minimum sensory assessment of 7 (Score 1-9) (Indonesia Standardization Agency, 2013). The results of the panelists' assessment of *julung julung* smoked fillet from each treatment can be seen in Fig. 7.

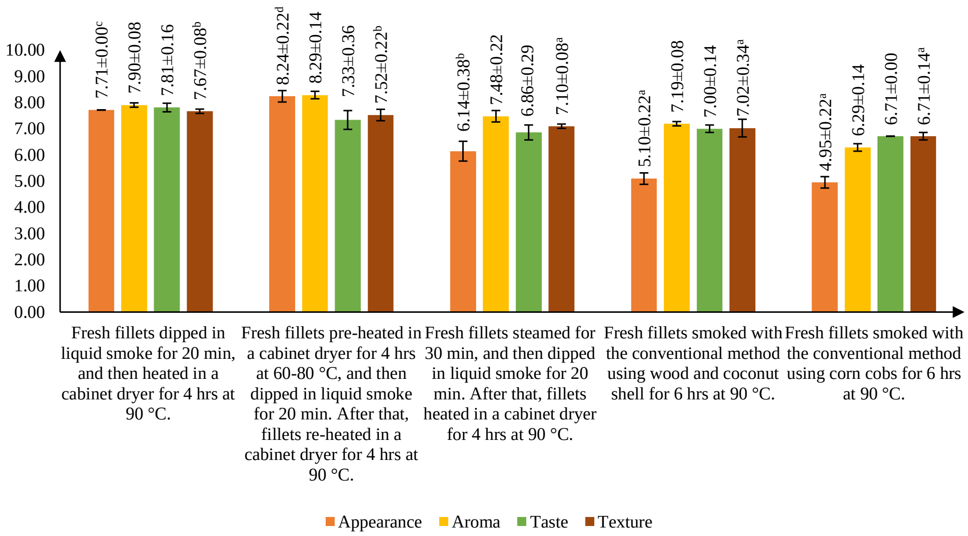

Fig. 7. Sensory assesment of *julung julung* smoked fillet

Appearance: Fig. 7 shows the analysis of the variance of the sensory assessment of the appearance of smoked *julung julung* fish fillets with different smoking method treatments affecting the appearance of smoked fish ($p<0.05$). The panelists' assessment of the appearance of smoked fish fillets ranged from 4.95 to 8.24, with the highest panelists' assessment in treatment B. Based on the requirements of the Indonesian National Standard, only treatments A and B met the minimum panelist assessment requirement of 7. The moisture content factor is thought to have influenced the panelists' assessment of the appearance of smoked fish, so panelists less favored treatment C with steaming. Moisture content can affect the physical properties of fish, such as the appearance and texture of smoked fish (Baten *et al*., 2020a). Smoked fish with high moisture content will make the color of smoked fish look paler (Flick, 2010).

Treatments D and E were less favored by panelists, presumably because conventional smoking methods produce darker products (dark brown), less bright and less shiny. Smoking fish with liquid smoke has smoked fish products with a golden color and clean and shiny surface (Berhimpon *et al*., 2018). Another factor that affects the appearance of smoked fish is the length of the smoking process. The longer the

smoking process, the darker the color will be. The time of the smoking process for smoked fish using the liquid smoke method is shorter than the conventional method (Puke & Galoburda, 2020; Baten *et al*., 2020b). The smoking process can change the color of fish, giving it a distinctive golden color due to the interaction of carbonyls with amino components on the surface of the meat. The color and appearance of smoked fish also positively correlate with phenol content. Phenol compounds in smoke interact with amino acid components in fish meat, producing a distinctive golden color in smoked fish (Montazeri *et al*., 2013).

Aroma: Fig. 7 shows the analysis of the variance of the sensory assessment of the aroma of smoked fish fillets *julung julung* with different smoking method treatments that did not affect the appearance of smoked fish ($p>0.05$). However, the aroma sensory assessment of treatments A, B, C, and D still meets the minimum assessment of the Indonesian National Standard on smoked fish. The sensory assessment of smoked fish aroma was lowest in treatment E. Previous studies have reported that smoking fish with corn cobs as a fuel source resulted in the lowest aroma scores, indicating less aromatic smoked fish (Asmara *et al*., 2022). The aroma and taste of smoked fish are strongly influenced by the time of smoking (Tahir *et al*., 2020). This assessment indicates that the time of the smoking process of 4 and 6 hours in all treatments has not influenced the taste and aroma of smoked fish.

Taste: Fig. 7 shows the analysis of the variance of the sensory assessment of the taste of smoked fish fillets *julung julung* with different smoking method treatments that did not affect the appearance of smoked fish ($p>0.05$). The taste of smoked fish is influenced by the time of the smoking process. Previous studies have reported that longer smoking time can improve the sensory attributes of taste (Baten *et al*., 2020b). This result indicates that the 2-hour time difference between the liquid smoke smoking treatment and the conventional smoking method has not affected the taste of smoked fish. A distinctive smoked taste without bitterness is a criterion for assessing the taste of good quality smoked fish (Sukowati *et al*., 2021).

Texture: Fig. 7 shows the analysis of variance of the sensory assessment of the texture of smoked fish fillet *julung julung* with different smoking method treatments giving effect to the texture of smoked fish ($p<0.05$). Panelists' assessment of the texture of smoked fish *julung julung*, ranged from 6.71 – 7.67, with the highest panelist assessment in treatments A and B. Duncan's test analysis showed that treatments A and B differed from treatments C, D, and E. This result was thought to be because the fish fillets were dipped in liquid smoke, giving a dry, compact, and tender texture. Whereas in treatment C (fish fillets subjected to steaming), the texture of the smoked fillets was rather sticky and not solid. Treatments D and E produced the texture of smoked fish fillets which were less dense and not compact. The texture of smoked fish is negatively correlated with its moisture content. The higher the moisture content in smoked fish, the softer and less firm the texture (Chan *et al*., 2022). High moisture content in smoked fish products results in less dense fish muscle fibers (Chang *et al*., 2021).

A good texture of smoked fish is compact, moderately elastic, and not sticky (Sulistijowati *et al*., 2021). The Indonesian National Standard requires that the texture of smoked fish is dense and compact and the tissue is very tight (Indonesia Standardization Agency, 2013). Based on consumer surveys, the preferred texture of smoked fish is dense, slightly wetter, and easy to chew, and there is a sensation of liquid in the mouth when chewed (Ticoalu *et al*., 2019).

Conclusion

Based on the findings of this study, it can be concluded that treatment B (Fresh fillets pre-heated in a cabinet dryer for 4 hours at 60 – 80 ºC and then dipped in liquid smoke for 20 minutes. After that, the fillets were re-heated in a cabinet dryer for 4 hours at 90 ºC.), when compared to the conventional smoking treatment based on total volatile bases, moisture content, water activity, pH value, phenol level, polycyclic aromatic hydrocarbon content, and sensory assessment. In general, fish smoking dipped in liquid smoke from corn cob produced a better quality of smoked fish than the conventional smoking method. It is necessary to evaluate different smoking times on smoked fish fillets of *julung julung* with liquid smoke method from corn cob.

Acknowledgement (bold)

Author contributions

Conflicts of interest

The authors declare that there is no conflict of interest.

Highlights

- Liquid smoke from corn cobs can increase total phenols. Fillets dried and soaked in liquid smoke.

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Commented [u10]: A lot! Decrease that.

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Characteristics of *Julung Julung* Smoked ~~Fish~~ Fillets (*Hemiramphus* sp.) using Liquid Smoke from Corn Cobs Waste

Abstract

Corn cobs can be utilized as a shell ingredient for liquid smoke. Liquid smoke can be used for the smoked fish industry in North Sulawesi, Indonesia. However, the utilization of liquid smoke for the smoked fish industry has yet to be optimal. This study aimed to obtain the best smoking method for *julung julung* fillets (*Hemiramphus* sp.) using liquid smoke from corn cob waste. The treatment in this study was to apply liquid smoke from corn cob waste in the smoking process, which was compared with the conventional method (using shell, coconut wood, and corn cob). Parameters to determine the quality of smoked fish were total volatile bases (TVB), moisture, water activity, pH, phenol levels, polycyclic aromatic hydrocarbon (PAH), and sensory assessment. The results showed that the TVB of smoked *julung julung* fish fillets ranged from 19.83 – 32.27 mg N/100g. The moisture ranged from 14.10 – 30.57%. Water activity ranged from 0.46 – 0.82. pH ranged from 4.60 – 5.81. Phenol levels ranged from 4.42 – 16.11 mg/g. PAH levels are still below the standard required in the Indonesian National Standard. Panelists rated neutral to really like the appearance, aroma, taste, and texture of smoked fish. From these results, it can be concluded that the best treatment is fresh fillets preheated in a cabinet dryer for 4 hours at 60 – 80 ºC and then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated in a cabinet dryer for 4 hours at 90 ºC.

Keywords: Coconut, Indonesia, Shell, North Sulawesi, Wood

Introduction

Corn is the second most widely grown crop in Indonesia after rice. Indonesia is the 7th corn producer in the world, with more than 12 million tons of corn produced in 2020 (Nazmi *et al*., 2021). North Sulawesi is one of the contributing regions that produce corn in Indonesia. Considerable corn production will also produce waste, especially corn cobs, which are generally thrown away and burned by most Indonesians (Cahyadi *et al*., 2021).

One of the corn cobs was used to make liquid smoke. Previous research reported that corn cobs can produce liquid smoke, a by-product of the pyrolysis of corn cob waste. The yield of liquid smoke from corn cobs is about 28.37%, with a pH value of 3.5 (Sriharti *et al*., 2020). The particle size of corn cobs affects the yield of liquid smoke, with smaller particle sizes resulting in higher yields (Aladin *et al*., 2018). Another study reported a phenolic content of 335 mg/L in liquid smoke produced from corn cobs (Swastawati *et al*., 2007).

Liquid smoke is a natural product made from the condensation of smoke from burning wood (Andy *et al*., 2021). Liquid smoke is commonly used as a flavoring in food to provide a smoked flavor without the food undergoing the actual smoking process (Sari *et al*., 2006). Using liquid smoke in food will save time, energy, and labor and reduce production costs (Krah *et al*., 2019). In addition, using liquid smoke in food can speed up and standardize the smoking process, adding flavor and microbiological safety

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while reducing production time (Indiarto *et al*., 2020). Generally, various types of hardwood were used to manufacture liquid smoke (Diatmika *et al*., 2019). The quality of the liquid smoke produced may vary depending on the type of wood used (Budaraga *et al*., 2016). In addition to imparting specific flavors and aromas to foods (Maulina *et al*., 2020), liquid smoke is reported to be antimicrobial (Septana *et al*., 2020) and antioxidant (Budaraga & Putra, 2021).

The conventional smoking processes generally use hardwood as fuel, such as melon wood (Umar *et al*., 2018), mangrove wood (Cissoko *et al*., 2020), teak wood (Daramola *et al*., 2020), coconut shells, and coconut husks (Nugroho *et al*., 2018). Corn cobs have also been reported to be used in the fish smoking process (Sukowati, 2023). According to literature, traditional fish smoking in North Sulawesi, Indonesia, uses smoke from burning coconut shells or wood charcoal (Landangkasiang *et al*., 2017; Primalasari *et al*., 2019; Saediman *et al*., 2021). Smoked fish processing has been known for a long time because of its traditional processing techniques, simplicity, ease of implementation, and low cost (Islamiyah, 2021). In North Sulawesi, Indonesia, smoked fish commonly known as *fufu* is traditionally made from skipjack (*Katsuwonus pelamis*); *julung julung* (*Hemiramphus far*) (Berhimpon *et al*., 2018); and scad fish (*Decapterus* spp.) known as *pinekuhe* (Ansar & Ijong, 2021). The smoked fish product of *julung julung* is a typical product from North Sulawesi and Gorontalo, commonly called *roa* or *galavea* (Azis & Akolo, 2020).

The study's reported that the conventional smoking process has disadvantages such as smoking time, concentration of carcinogenic substances, temperature, and inconsistent product quality (Racovita *et al*., 2020). Conventional smoking of food products has been shown to produce carcinogenic components such as polycyclic aromatic hydrocarbons (Jinadasa *et al*., 2020). Benzo(a)pyrene ($C_{20}H_{12}$) is one of the carcinogenic compounds produced from traditional smoking products (Jinadasa *et al*., 2020). One method to reduce carcinogenic compounds in smoked fish products is to use liquid smoke in the smoking process (Nithin *et al*., 2020; Xin *et al*., 2021).

The utilization of liquid smoke in the fish smoking industry in North Sulawesi is very likely to be developed. North Sulawesi was reported to have both large-scale and domestic fish-smoking industries (Dotulong *et al*., 2018; Primalasari *et al*., 2019). Making liquid smoke is relatively simple, so it will be readily accepted by fish-smoking industry players (Ali & Al Fiqri, 2020). Previous research reported that using liquid smoke in smoked fish products resulted in good quality in terms of appearance, flavor, and aroma (Rizal *et al*., 2020). In addition, using liquid smoke in smoked fish products can add nutritional value and durability, making it possible to reach a wider market area (Ali *et al*., 2021).

Previous research reported that smoked fish dipped in liquid smoke for 15 minutes had a total plate count value of 4.7×10^4 CFU/g on day six and moisture content below 60% during storage (Suroso *et al*., 2018). Dipping fish in liquid smoke with a concentration of 15% and a dipping time of 60 minutes showed a bacterial colony of 2.12×10^2 CFU/g (Ali *et al*., 2021). The Indonesian National Standard on

smoked fish requires a maximum total plate count of 5×10^4 CFU/g and a maximum moisture content of 60% (Indonesia Standardization Agency, 2013). Another study reported that smoked fish processed with liquid smoke had a moisture content of 47.63%, phenol content of 12.62%, and pH of 4.8 (Berhimpon *et al*., 2018). The results of these studies indicate that liquid smoke is good to apply to smoked fish products. This study characterized smoked fish fillets of *julung julung* (*Hemiramphus* sp.) produced with liquid smoke from corn cobs waste. The study was conducted by dipping the fish fillets of *julung julung* with liquid smoke, with drying and steaming treatments on the fish fillets. This study aims to obtain the best smoking method for fish fillets using liquid smoke from corn cobs waste based on the assessment of total volatile bases, moisture content, water activity, pH value, phenol content, polycyclic aromatic hydrocarbon content, and sensory assessment.

Materials and methods

The main materials of the study were *julung julung* fish (*Hemiramphus* sp.), corn cob (*Zea mays* L.) from Gangga Island in Likupang, North Sulawesi, Indonesia. This study includes the preparation of liquid smoke from corn cobs waste, preparing fish (weeding and filleting), and treating fish fillets by dip in liquid smoke. Fresh *julung julung* fish was obtained from traditional fishermen in Manado, North Sulawesi, Indonesia (Fig. 1). The fish was placed in a cool box and given ice in a ratio of 1:1 (ice:fish) (Mishra *et al*., 2021). Then, the fish was washed, weeded, and filleted. The cleaned fillets were dipped in a 5% salt solution for 30 minutes. The concentration of liquid smoke from corn cob waste was 0.8% (Berhimpon *et al*., 2018).

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Fig. 1. *Julung julung* (*Hemiramphus* sp.)

The treatment in this study was applying liquid smoke from corn cobs waste to the smoking process of *julung julung* fish fillets, which was compared with the conventional method (using coconut wood, coconut shells, and corn cobs).

(A) : Fresh fillets were dipped in liquid smoke for 20 minutes and then heated in a cabinet dryer for 4 hours at 90 °C.

(B) : Fresh fillets pre-heated in a cabinet dryer for 4 hours at 60 – 80 °C, and then dipped in liquid smoke for 20 minutes. After that, fillets were re-heated in a cabinet dryer for 4 hours at 90 °C.

(C) : Fresh fillets were steamed for 30 minutes and then dipped in liquid smoke for 20 minutes. After that, fillets were heated in a cabinet dryer for 4 hours at 90 °C.

(D) : Fresh fillets were smoked with the conventional method using wood and coconut shell for 6 hours at 90 °C.

(E) : Fresh fillets were smoked with the conventional method using corncob for 6 hours at 90 °C.

Liquid smoke manufacturing process

Making liquid smoke refers to modified research (Aladin *et al*., 2018). Modifications were made to the equipment used in the pyrolysis process. Dry corn cobs were weighed to determine fuel efficiency. Corn cobs were gradually burned into the combustion furnace. The furnace was closed to prevent smoke from escaping from the tank. The smoke generated from the combustion flows through a pipe connected to a storage tank covered with ice cubes. The smoke that passes through the pipe will become cold, so condensation occurs, turning the smoke into liquid. The smoke that has been formed is collected into a container attached to the end of the pipe. The liquid smoke obtained is then allowed to settle the tar formed for three weeks and filtered to obtain clear liquid smoke.

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Total volatile bases assay

Total volatile base (TVB) assay refers to research by Tambunan & Chamidah (2021). TVB assay aims to determine the amount of volatile base compounds formed from protein breakdown—the principle of TVB analysis, namely by evaporating volatile base compounds at room temperature for 24 hours. The compound is then bound with boric acid and titrated with an HCl solution. After the boric acid solution was stored in the inner chamber of the Conway cell containing the blank (V_0) and the filtrate (V_1), the boric acid solution was titrated with 0.02 N HCl so that the boric acid solution changed color to pink. TVB levels in the smoked fish meat were expressed as mg N/100g.

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$$TVB\left(\mathrm{mg}\frac{\mathrm{N}}{100\mathrm{g}}\right) = \frac{(V_1 - V_0) \times N.HCl \times dilution \times 100}{sample\ weight\ (g)}$$

Moisture content assay

The moisture content by method of Indonesia National Standard (Indonesia Standardization Agency, 2006a). An empty porcelain cup is weighed (A). Then, the sample was weighed to ± 2 g in a porcelain cup of known mass (B). The samples were dried in an oven at 105°C for 20 hours. The samples were then cooled in a desiccator, after which the samples in the cup were weighed (C). Moisture content was expressed as %.

$$Moisture\ (\%) = \frac{B\ (g) - C\ (g)}{B(g) - A\ (g)} \times 100\%$$

Water activity (Aw) assay

Water activity assay used an Aw meter (Rotronic, HygroPalm 23-AW-A). The Aw meter was set up at room temperature for 2 hours. The water activity of smoked fish was measured by placing the sample into a sample container and conditioning it for 30-60 minutes. The Aw meter sensor is contacted with the sample in the container. Then the water activity (Aw) value can be read on the Aw meter panel (Saputra *et al*., 2014).

pH assay

The sample weighed as much as 30 g, then 400 mL of distilled water was added at 28 ºC. The sample was homogenized with a magnetic stirrer to be evenly stirred. The pH meter electrode was inserted into the sample solution. The data taken in this test is the pH value read by the pH meter (Hanna Instruments HI99192) (Lekahena & Jamin, 2018).

Phenol level assay

Samples were extracted by adding 1 mL ethanol p.a., 5 mL distilled water, and 0.5 mL Folin Ciocalteu 50%, homogenized, and allowed to stand for 5 minutes. Then added, 1 mL of 5% Na_2CO_3 and left in the dark for ± 60 minutes with gallic acid as a standard. The absorbance value was read on a spectrophotometer (Thermo Scientific Genesys 50) with a wavelength of 725 nm. Total phenolics were expressed as mg/g (Indonesia Standardization Agency, 2004; Muliadin *et al*., 2022).

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$$Total\ Phenol\ \left(\frac{mg}{g}\right) = \frac{Gallic\ acid\ equivalence\ \left(\frac{mg}{L}\right) \times Volume\ (L)}{Sample\ weight\ (g)}$$

Polycyclic aromatic hydrocarbon (PAH) assay

PAH content testing was carried out based on the instructions Basak *et al*. (2010). A 5 g sample was weighed and collected in a 100 ml flask. Then 5 mL of 50% KOH solution, 75 mL of methanol, 1 mL

of internal standard solution (9,10 dimethylantracene), and some boiling stones were added. After that, the mixture was boiled for 4 hours in a soxhelet. The liquid phase was transferred to a separatory funnel and extracted with 100 mL of n-hexane with shaking for 3 min. The MeOH:KOH phase was drained and discarded. The combined n-hexane phase was rinsed twice, each with 50 mL of H_2O-MeOH (8:1) and 50 mL of water. The methanol-water and water phases were dried and discarded. The organic phase was concentrated in a rotary evaporator (40°C) to a volume of about 10 mL. The concentrated extract was dried under nitrogen gas.

After that, the silica gel column was cleaned by dissolving 15 g of silica gel in n-hexane and transferred to a column (200 mm long; diameter: 12 mm). Then, 1 g of anhydrous sodium sulfate was added. The concentrated sample extract was transferred to the column and eluted with 1 mL of n-hexane. A stock solution containing 0.1 mg mL^{-1} of 9,10 dimethylantracene was dissolved in n-hexane and stored at 4°C in a volumetric flask (with a glass stopper) wrapped in aluminum foil to avoid possible light degradation. Working standard solutions were prepared from the stock solutions. Analysis of PAH compounds used a Hewlett-Packard 1100 HPLC equipped with an Agilent-110 fluorescence detector. The injection volume was 10 μL. Mobile phase gradient (acetonitrile-water): 80% acetonitrile + 20% water with a flow rate of 1 ml min^{-1} and a wavelength of 270 nm. The external standard mixture was used for PAH concentration calculation.

Sensory assessment assay

Sensory assessment assay refers to Indonesia National Standard (Indonesia Standardization Agency, 2006b). The sensory assessment was based on a hedonic scale (Table 1). Smoked fish samples were placed on a plastic plate with a glass of water, coded, and presented to 30 panelists randomly under light. The parameters observed were appearance, aroma, taste, and texture. Panelists rated acceptance of the samples on a scale of 1 – 9.

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Table 1. Score sheet of hedonic assessment

Specification	Score
Really like it	9
Really like	8
Like	7
Rather like	6
Neutral	5
Rather dislike	4
do not like	3
Very dislike	2
Really don't like it	1

Data analysis

Data analysis was performed using Statistical Product and Service Solutions (SPSS) version 20.0. Statistical analysis of data was performed based on one-way analysis of variance (ANOVA) using a significance level of ($P<0.05$). Specific group differences were determined using Duncan's test.

Results and discussion

Liquid smoke production was carried out using a simple liquid smoke distillation device. This process consumed 17 kg of corn cobs and 12 kg of crushed ice. Liquid smoke from corn cobs is more accessible than coconut shells or wood (Maulina & Karo, 2021). Using coconut shells or wood as fuel will form a flame, thus reducing the volume of smoke in the furnace (Kabir Ahmad *et al*., 2022). The smoked *julung julung* were analyzed for TVB, moisture content, water activity, pH value, phenol content, PAH content, and sensory assessment.

Total volatile bases

The total volatile base (TVB) value is measured to determine the quality of the smoked fish. TVB value is one of the parameters used to determine the decline in fish quality and measures the amount of volatile base compounds formed due to protein degradation (Castro *et al*., 2006). Volatile bases formed in fish muscle tissue mainly consist of ammonia, trimethylamine (TMA), and dimethylamine (DMA) (VELP Scientifica, 2013). TVB value is an essential characteristic for the quality assessment of seafood products and is emerging as the most commonly used chemical parameter to assess the palatability of seafood (Rasulu *et al*., 2020). The results of *total volatile bases* on smoked fish fillets of *julung julung* are presented in Fig. 2.

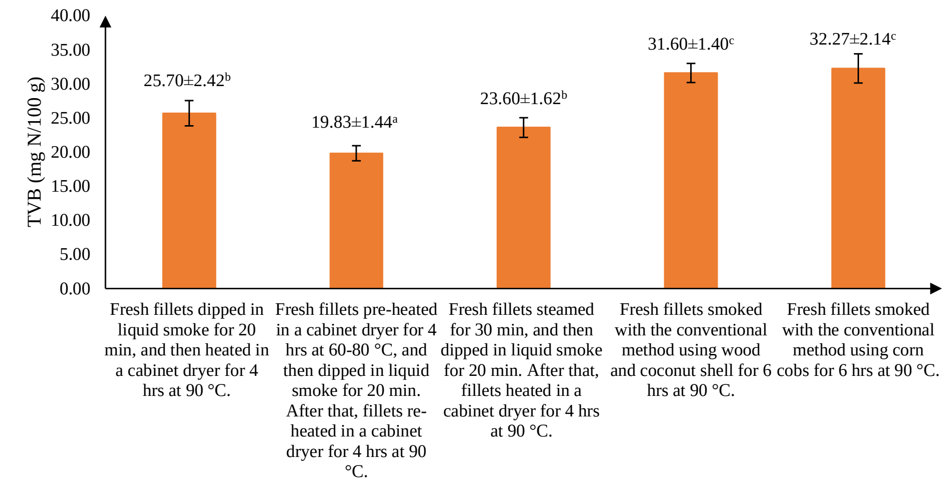

Fig. 2. Total volatile bases of *julung julung* smoked fillet

Fig. 2 shows the analysis of the variance of smoked fish fillets with different smoking method treatments affecting changes in TVB content ($p<0.05$). The TVB content of smoked fish fillets ranged from 19.83 – 32.27 mg N/100g, with the lowest TVB value in treatment B. The treatment of the smoking method with liquid smoke from corn cob waste (treatments A, B, and C) had lower TVB levels when compared to the conventional smoking method (treatments D and E). A literature search has not found why liquid smoking has a lower TVB value than conventional smoking methods. However, one possibility is that the liquid smoke smoking method allows the smoke components to be absorbed into the fish meat better than the conventional smoking method. Better absorption of liquid smoke phenol components in fish meat can inhibit and control microbial growth that causes a decrease in the quality of smoked fish (Santoso *et al*., 2015). A literature study showed that the average TVB value of smoked mackerel (*Euthynnus affinis*) treated with liquid smoke addition ranged from 24.63 – 28.38 mg N/100g (Hardianto & Yunianta, 2015). Another study reported smoked mackerel with a smoking process using rubber wood liquid smoke, producing smoked fish products with lower TVB values (Suroso *et al*., 2018). Duncan's analysis showed a difference between treatments B, A – C, and D – E on the TVB value of smoked fish fillets. The analysis of variance indicates that the drying process of smoked fish fillets before immersion into liquid smoke can maintain the quality of smoked fish (Kaparang *et al*., 2013). Duncan's analysis also showed that treatments A – C and D – E had no difference in TVB value, indicating that the moisture content between the two treatments was not different. TVB value is closely related to moisture content (Rasulu *et al*., 2020).

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The European standard (European Market Observatory for Fisheries and Aquaculture, 2020) states that the limit range of TVB value for smoked fish is 25 – 35 mg N/100 g. All treatments' TVB value of the smoked fish fillets still met the European standard. Previous studies reported TVB values of fresh fish fillets of 8.70 ± 0.86 mgN/100g (Moosavi-Nasab *et al*., 2021) and 12.94 ± 0.92 mg N/100g (Bouzgarrou *et al*., 2020). Messina *et al*. (2021) reported that smoked fish fillets that underwent two drying processes had a TVB value of <20 mg N/100g. Another study also reported the TVB value of smoked fish fillet samples with hot smoking and cold smoking, which amounted to 17.80 ± 0.17 and 18.95 ± 0.20 mg N/100g, respectively (El-Lahamy *et al*., 2019). Aydin *et al*. (2020) reported a TVB value of smoked fish with hot smoking of 11.82 ± 0.05 mg N/100g.

Moisture content

Moisture content is an important parameter to determine the quality of smoked fish produced. The moisture content contained in smoked fish can affect the shelf life of smoked fish because water in food is a medium for microbes to grow (Kaban *et al*., 2019). The smoking process was reported to reduce the moisture content of fresh fish to a range between 9 and 17% (Sikoki & Aminigo, 2002). The moisture content of smoked fish fillets is presented in Fig. 3.

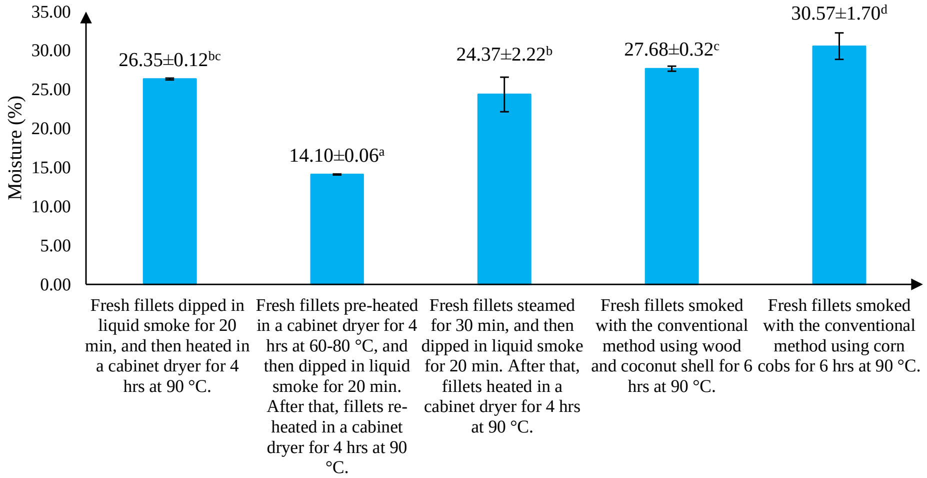

Fig. 3. Moisture content of *julung julung* smoked fillet

Fig. 3 shows the analysis of the variance of smoked *julung julung* fish fillets with different smoking methods treatment affects moisture content ($p<0.05$). The moisture content of smoked *julung julung* fish fillets ranged from 14.10 – 30.57%, with the lowest moisture content in treatment B. The

moisture content value in smoked fish products from all treatments still meets the Indonesian National Standard No. 2725:2013 on smoked fish, which is a maximum of 60% (Indonesia Standardization Agency, 2013). Darianto *et al*. (2018) reported that the smoking process can reduce the moisture content of fish to below 40%, which can help preserve it longer. The treatment of the smoking method with liquid smoke with corn cob (Treatment A, B, C) has a lower moisture content when compared to the treatment of the conventional smoking method (Treatment D and E). This result is because the smoking chamber is not fully enclosed in the conventional smoking method, so the heat generated could be more optimal. Suboptimal heat can increase moisture content and cause the moisture content of smoked fish to decrease only slightly (Amos & Paulina, 2017). Whereas in the liquid smoke smoking method, temperature and humidity can be controlled better so that the moisture content of the product can be reduced efficiently (Salindeho & Lumoindong, 2017).

Duncan's analysis showed a difference between treatment B, treatment A – C, and treatment D – E on the moisture content of smoked fish fillets. The results of the variance study indicate that the drying process of smoked fish fillets before the dip in liquid smoke in treatment B can help reduce the moisture content. This study's results are from previous research, which also reported a significant decrease in moisture content in smoked fish products with two times drying treatment (Messina *et al*., 2021). Duncan's analysis also showed that treatments A and C were not different because steaming in treatment C will increase the moisture content of the product (Salmatia *et al*., 2020). The conventional smoking method with corn cob fuel has the highest moisture content; this result is the conventional smoking process with corn cobs, and the heat generated is lower than smoking using wood (Asmara *et al*., 2022).

Water activity (Aw)

Water activity is one of the essential parameters in the quality of smoked fish. Water activity (Aw) is expressed as the ratio of the vapor pressure in the food to the vapor pressure of pure water, and it predicts whether water tends to move from the food product into the cells of microorganisms that may be present. A well-smoked fish has a water activity of <0.50 and a moisture content between 15 and 25% to inhibit the growth of pathogenic microorganisms in smoked fish products (Mondo *et al*., 2020). According to British Columbia Centre for Disease Control (2013), the maximum water activity value for smoked fish is 0.97. Water activity correlates with the moisture content of a smoked fish product and is two important factors affecting food safety and quality (Fitri *et al*., 2022). The water activity values of smoked fish fillets can be seen in Fig. 4.

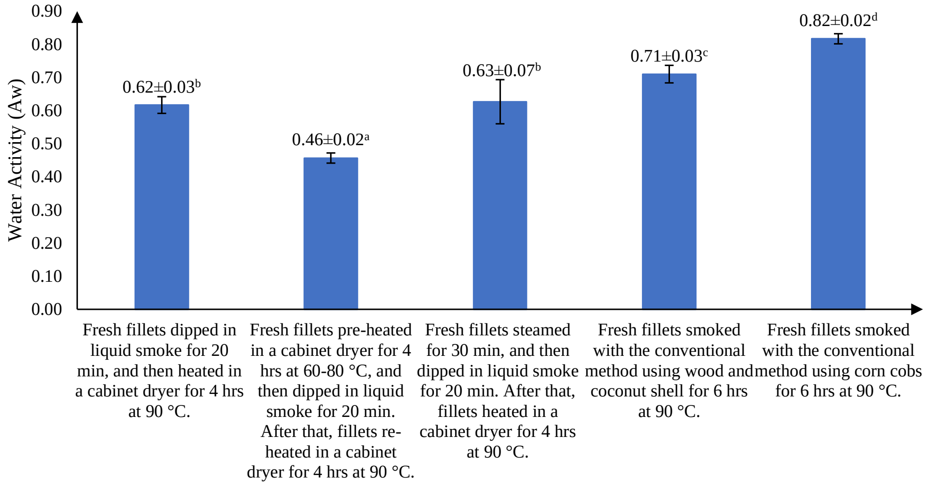

Fig. 4. Water activity of *julung julung* smoked fillet

Fig. 4 shows the analysis of the variance of smoked fish fillet *julung julung* with different smoking method treatments affecting water activity ($p<0.05$). The water activity value of smoked fish fillet ranged from 0.46 – 0.82, with the lowest water activity in treatment B. The treatment of the smoking method with corn cob liquid smoke (Treatment A, B, C) had a lower water content when compared to the treatment of the conventional smoking method (treatment D and E).

The water activity value of fish meat can vary depending on the cooking method, temperature, and duration of heating (Oliveira *et al*., 2017; Zhang *et al*., 2023). Therefore, the factor of fish fillet treatment before immersion in liquid smoke caused the difference in water activity value in treatment B compared to treatments A and C. The water activity value is directly proportional to the water content value of each treatment. The heating process can reduce the moisture content of fish meat (Kiczorowska *et al*., 2019), thus affecting the water activity of fish meat (Gómez *et al*., 2020).

pH

The pH value of smoked fish is an essential factor affecting its quality. The pH value will decrease with increasing smoking time (Baten *et al*., 2020b). The analysis of the variance of smoked fish fillets *julung julung* with different smoking method treatments affects the pH value ($p<0.05$). The pH of smoked fish fillet ranged from 4.60 to 5.81 (Fig. 5). The treatment of the smoking method with corn cob liquid smoke (treatments A, B, and C) had a lower pH value when compared to the conventional smoking method (treatments D and E). Swastawati *et al*. (2022) also reported that the pH value of smoked barracuda fish with the liquid smoke method was significantly lower than that of the conventional

method. Another study also reported that using liquid smoke can reduce pH caused by the condensation of organic acids in the smoking process (Puke & Galoburda, 2020). The pH value of smoked fish correlates with the phenol content contained in the smoke component, and an increase in phenol content causes a decrease in pH (Berhimpon *et al*., 2018).

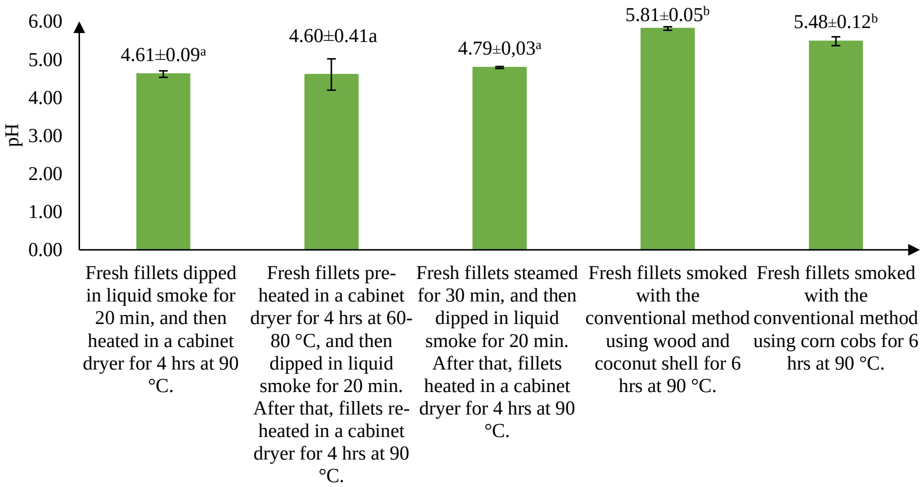

Fig. 5. pH of *julung julung* smoked fillet

Phenol level

Phenol is a compound found in wood smoke. Phenol compounds enter food through diffusion and capillary action, affecting taste, color, and aroma and extending shelf life (Remy *et al*., 2016). In addition, phenol compounds are also reported to act as antioxidants that can prevent rancidity in fish meat (Sérot *et al*., 2004). Leksono *et al*. (2020) also noted that the higher the phenol level in smoke, the stronger the flavor and aroma of smoked fish. The analysis of the variance of smoked fish fillets julung julung with different smoking method treatments affecting phenol level ($p<0.05$). The phenol level of smoked fish fillet ranged from 4.42 to 16.11 mg/g (Fig. 6). The treatment of the smoking method with corn cob liquid smoke (treatments A, B, and C) had higher phenol level when compared to the conventional smoking method (treatments D and E).

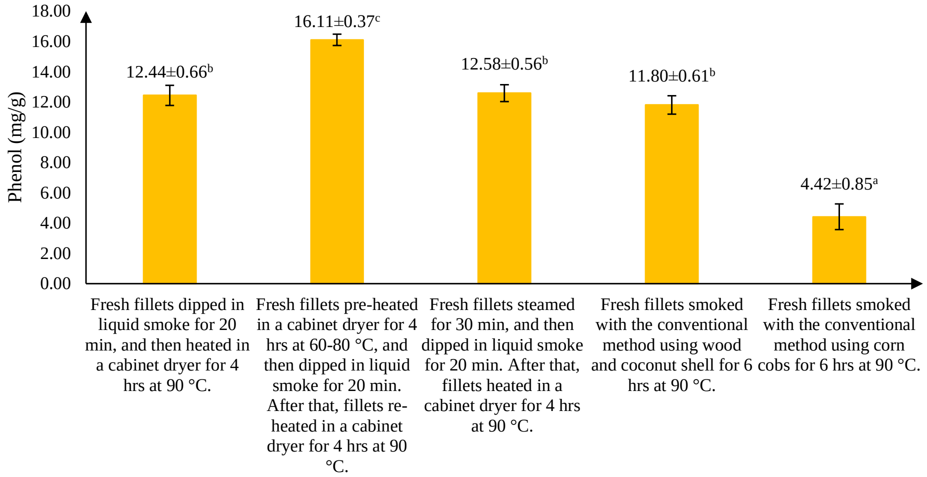

Fig. 6. Phenol level of *julung julung* smoked fillet

Duncan's analysis showed phenol levels in treatments A, C, and D were not different. Previous research reported that the drying treatment of fish meat can increase phenolic compounds in smoked fish products (Sérot *et al*., 2004). The high phenol content in treatment B is thought to be because the pre-heated process causes the fish meat to lose water so that when dipped in liquid smoke, the fish fillets absorb a large amount of liquid smoke. Previous studies have reported that when the fish surface is dried, there is less smoke condensation than products smoked at lower temperatures. The results of this study indicate that a dry fish surface allows for better absorption of smoke components (Belichovska *et al*., 2019).

The study reported phenol levels in liquid smoke from corn cob waste of 0.335 mg/g (Swastawati *et al*., 2007) and 2.55% (Leviyani *et al*., 2019). The phenol level of smoked fish with the conventional method of smoking with corn cobs is lower due to the high acid content, which can potentially reduce the phenol content of smoked fish (Swastawati *et al*., 2012). Anggraini & Nurhazisa (2017) reported that the phenol content of liquid smoke from coconut shells was 3.04%, while liquid smoke from corn cob was 1.38%.

Polycyclic aromatic hydrocarbon (PAH) levels

Polycyclic aromatic hydrocarbons are a group of organic compounds formed by incomplete combustion, such as wood. Therefore, the type of fuel used in the fish smoking process affects the PAH content of smoked fish (Jinadasa *et al*., 2020). One of the carcinogenic PAHs, benzo(a)pyrene, is a carcinogenic marker in smoked fish products (Stołyhwo & Sikorski, 2005). Polycyclic aromatic hydrocarbon (PAH) levels in smoked fish fillets can be seen in Table 2.

Table 2. Polycyclic aromatic hydrocarbon levels in *julung julung* smoked fillet

Polycyclic Aromatic Hydrocarbon	Smoked *Julung Julung* fish (µg/kg) A	B	C	D	E	Corn Cob Liquid Smoke 0.8%(µg/kg)
Benzo(a)pyrene	nd	nd	nd	1.5	0.2	0.5
Benzo(b)fluoranthene	1.2	1.0	1.2	2	1.8	2.5
Benzo(a)anthracene	nd	nd	nd	0.5	nd	0.2
Benzo(g)perylene	0.4	0.2	0.4	1.0	0.8	1.0

nd = not detected

Table 2 shows that the PAH levels in smoked fish are still below the standard required in the Indonesian National Standard No. 2725 of 2013 on smoked fish, which is benzo(a)pyrene max 5 µg/kg (Indonesia Standardization Agency, 2013). The polycyclic aromatic hydrocarbon compound regulated in the Indonesian National Standard is only benzo(a)pyrene. Stołyhwo & Sikorski (2005) reported benzo(a)pyrene levels in smoked fish with hot and cold smoking processes ranging from 0.05 to about 60 µg/kg. Other studies have reported benzo(b,k)fluoranthene levels in salmon and rainbow trout ranging from 1.83 to 9.55 µg/kg, while benzo(g,h)perylene levels in salmon were 0.44 µg/kg (Basak *et al*., 2010). Berhimpon *et al*. (2018) also reported benzo(a)pyrene levels in smoked skipjack fish of 0.25 µg/kg. The level of benzo(a)pyrene in smoked barracuda was reported to be 0.32 µg/kg, benzo(b)fluoranthene 0.35 µg/kg, benzo(k)fluoranthene 0.21 µg/kg, benzo(a)anthracene 0.44 µg/kg, and benzo(g,h)perylene 2.56 µg/kg (Asamoah *et al*., 2021).

High PAH levels in smoked fish are due to the lignin content of the material used as fuel in the smoking process (Asamoah *et al*., 2021). The lignin content in corn cobs is reported to be about 17 – 21% (Thangavelu *et al*., 2018; Olajuyigbe *et al*., 2019; Gandam *et al*., 2022). The lignin content of coconut shell was approximately 59.5% (Wang & Sarkar, 2018), while that of coconut wood was about 50% on a dry weight basis (Anuchi *et al*., 2022). Lignin is reported to absorb PAH compounds because PAH compounds can bind well with lignin (Oliveira *et al*., 2019). During pyrolysis and gasification processes, lignin components react to produce aromatic tar and coke, which can form PAH compounds (Zhou *et al*., 2014; Kawamoto, 2017). Therefore, PAH compounds in smoked fish smoked using corncobs are lower than those smoked with shells and coconut wood.

Sensory assessment

A sensory assessment is carried out to evaluate the panelist's preference level, including appearance, aroma, taste, and texture. A sensory assessment must determine the quality of the smoked fish product and ensure it meets the applicable standards (Hadanu & Lomo, 2019). The sensory characteristics of smoked fish products affect consumer acceptance and preference. Sensory assessment by panelists helps to identify the sensory attributes that are most appealing to consumers, allowing manufacturers to

customize products according to consumer assessment (Ekelemu *et al*., 2021). Indonesian National Standard No. 2725:2013 regarding smoked fish requires a minimum sensory assessment of 7 (Score 1-9) (Indonesia Standardization Agency, 2013). The results of the panelists' assessment of *julung julung* smoked fillet from each treatment can be seen in Fig. 7.

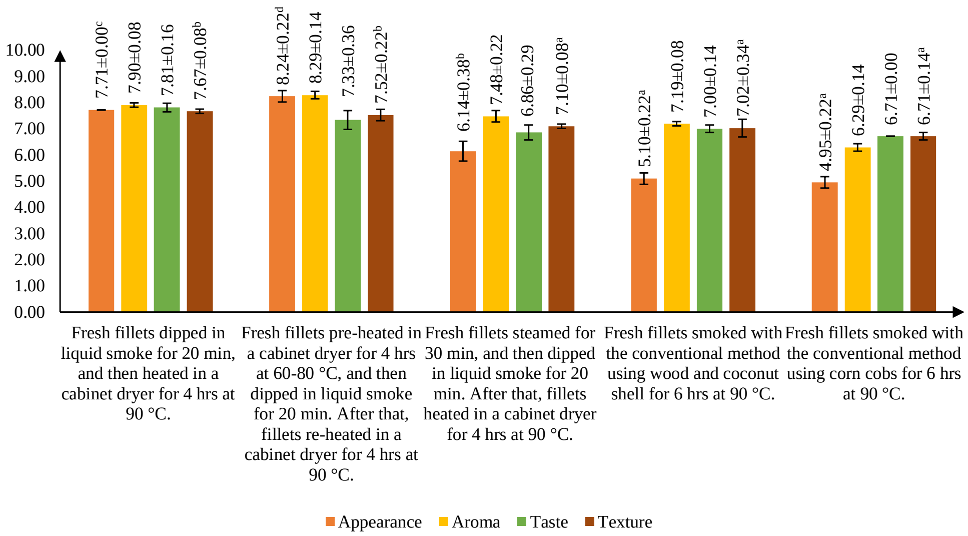

Fig. 7. Sensory assesment of *julung julung* smoked fillet

Appearance: Fig. 7 shows the analysis of the variance of the sensory assessment of the appearance of smoked *julung julung* fish fillets with different smoking method treatments affecting the appearance of smoked fish ($p<0.05$). The panelists' assessment of the appearance of smoked fish fillets ranged from 4.95 to 8.24, with the highest panelists' assessment in treatment B. Based on the requirements of the Indonesian National Standard, only treatments A and B met the minimum panelist assessment requirement of 7. The moisture content factor is thought to have influenced the panelists' assessment of the appearance of smoked fish, so panelists less favored treatment C with steaming. Moisture content can affect the physical properties of fish, such as the appearance and texture of smoked fish (Baten *et al*., 2020a). Smoked fish with high moisture content will make the color of smoked fish look paler (Flick, 2010).

Treatments D and E were less favored by panelists, presumably because conventional smoking methods produce darker products (dark brown), less bright and less shiny. Smoking fish with liquid smoke has smoked fish products with a golden color and clean and shiny surface (Berhimpon *et al*., 2018). Another factor that affects the appearance of smoked fish is the length of the smoking process. The longer the

smoking process, the darker the color will be. The time of the smoking process for smoked fish using the liquid smoke method is shorter than the conventional method (Puke & Galoburda, 2020; Baten *et al*., 2020b). The smoking process can change the color of fish, giving it a distinctive golden color due to the interaction of carbonyls with amino components on the surface of the meat. The color and appearance of smoked fish also positively correlate with phenol content. Phenol compounds in smoke interact with amino acid components in fish meat, producing a distinctive golden color in smoked fish (Montazeri *et al*., 2013).

Aroma: Fig. 7 shows the analysis of the variance of the sensory assessment of the aroma of smoked fish fillets *julung julung* with different smoking method treatments that did not affect the appearance of smoked fish ($p>0.05$). However, the aroma sensory assessment of treatments A, B, C, and D still meets the minimum assessment of the Indonesian National Standard on smoked fish. The sensory assessment of smoked fish aroma was lowest in treatment E. Previous studies have reported that smoking fish with corn cobs as a fuel source resulted in the lowest aroma scores, indicating less aromatic smoked fish (Asmara *et al*., 2022). The aroma and taste of smoked fish are strongly influenced by the time of smoking (Tahir *et al*., 2020). This assessment indicates that the time of the smoking process of 4 and 6 hours in all treatments has not influenced the taste and aroma of smoked fish.

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Taste: Fig. 7 shows the analysis of the variance of the sensory assessment of the taste of smoked fish fillets *julung julung* with different smoking method treatments that did not affect the appearance of smoked fish ($p>0.05$). The taste of smoked fish is influenced by the time of the smoking process. Previous studies have reported that longer smoking time can improve the sensory attributes of taste (Baten *et al*., 2020b). This result indicates that the 2-hour time difference between the liquid smoke smoking treatment and the conventional smoking method has not affected the taste of smoked fish. A distinctive smoked taste without bitterness is a criterion for assessing the taste of good quality smoked fish (Sukowati *et al*., 2021).

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Texture: Fig. 7 shows the analysis of variance of the sensory assessment of the texture of smoked fish fillet *julung julung* with different smoking method treatments giving effect to the texture of smoked fish ($p<0.05$). Panelists' assessment of the texture of smoked fish *julung julung*, ranged from 6.71 – 7.67, with the highest panelist assessment in treatments A and B. Duncan's test analysis showed that treatments A and B differed from treatments C, D, and E. This result was thought to be because the fish fillets were dipped in liquid smoke, giving a dry, compact, and tender texture. Whereas in treatment C (fish fillets subjected to steaming), the texture of the smoked fillets was rather sticky and not solid. Treatments D and E produced the texture of smoked fish fillets which were less dense and not compact. The texture of smoked fish is negatively correlated with its moisture content. The higher the moisture content in smoked fish, the softer and less firm the texture (Chan *et al*., 2022). High moisture content in smoked fish products results in less dense fish muscle fibers (Chang *et al*., 2021).

A good texture of smoked fish is compact, moderately elastic, and not sticky (Sulistijowati *et al*., 2021). The Indonesian National Standard requires that the texture of smoked fish is dense and compact and the tissue is very tight (Indonesia Standardization Agency, 2013). Based on consumer surveys, the preferred texture of smoked fish is dense, slightly wetter, and easy to chew, and there is a sensation of liquid in the mouth when chewed (Ticoalu *et al*., 2019).

Conclusion

Based on the findings of this study, it can be concluded that treatment B (Fresh fillets pre-heated in a cabinet dryer for 4 hours at 60 – 80 ^{o}C and then dipped in liquid smoke for 20 minutes. After that, the fillets were re-heated in a cabinet dryer for 4 hours at 90 ^{o}C.), when compared to the conventional smoking treatment based on total volatile bases, moisture content, water activity, pH value, phenol level, polycyclic aromatic hydrocarbon content, and sensory assessment. In general, fish smoking dipped in liquid smoke from corn cob produced a better quality of smoked fish than the conventional smoking method. It is necessary to evaluate different smoking times on smoked fish fillets of *julung julung* with liquid smoke method from corn cob.

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Acknowledgement (bold)

Author contributions

Conflicts of interest

The authors declare that there is no conflict of interest.

Highlights

- Liquid smoke from corn cobs can increase total phenols. Fillets dried and soaked in liquid smoke.

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3. Bukti respons kepada reviewer terkait revisi manuskrip

Characteristics of *Julung Julung* Smoked Fillets (*Hemiramphus* sp.) using Liquid Smoke from Corn Cobs Waste

Abstract

This study aimed to obtain the best smoking method for *julung julung* fillets (*Hemiramphus* sp.) using liquid smoke from corn cob waste. This study used three different fillet treatment methods (dried and steamed) before liquid smoke application. This study also compared fish smoking between using liquid smoke with conventional methods (using shell, coconut wood, and corn cob). The parameters to determine the quality of smoked fish were Total Volatile Bases (TVB), moisture, water activity, pH, phenol levels, polycyclic aromatic hydrocarbon (PAH), and sensory assessment. The results showed that the TVB of smoked *julung julung* fillets ranged from 19.83 – 32.27 mg N/100g. The moisture ranged from 14.10 – 30.57%. Water activity ranged from 0.46 – 0.82. pH ranged from 4.60 – 5.81. Phenol levels ranged from 4.42 – 16.11 mg/g. PAH levels are still below the standard required in the Indonesian National Standard. Panelists rated neutral to really like the appearance, aroma, taste, and texture of smoked fish. From these research, it can be concluded that treatment B is the best treatment, namely fresh fillets are first heated for 4 hours at 60 – 80 ºC and then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated for 4 hours at 90 ºC.

Keywords: Corn cobs, Indonesia, Liquid smoke, North Sulawesi, Wood

Introduction

Corn is the second most widely grown crop in Indonesia after rice. Indonesia's maize production in 2023 is 14.46 million tons, ranking 9th in the world. (Indonesia Center of Statistic Agency, 2023). North Sulawesi is one of the contributing regions that produce corn in Indonesia. Considerable corn production will also produce waste, especially corn cobs, which are generally thrown away and burned by most Indonesians (Cahyadi *et al*., 2021).

One of the corn cobs was used to make liquid smoke. Previous research reported that corn cobs can produce liquid smoke, a by-product of the pyrolysis of corn cob waste. The yield of liquid smoke from corn cobs is about 28.37%, with a pH value of 3.5 (Sriharti *et al*., 2020). The particle size of corn cobs affects the yield of liquid smoke, with smaller particle sizes resulting in higher yields (Aladin *et al*., 2018). Swastawati et al. (2007) reported the phenolic content in liquid smoke produced from 335 mg/L corn cobs.

Liquid smoke is a natural product made from the condensation of smoke from burning wood (Andy *et al*., 2021). Liquid smoke is commonly used as a flavoring in food to provide a smoked flavor without the food undergoing the actual smoking process (Sari *et al*., 2006). Using liquid smoke in food will save time, energy, and labor, so that can reduce production costs (Krah *et al*., 2019). In addition, using liquid smoke in food can speed up and standardize the smoking process, adding flavor and microbiological safety while reducing production time (Indiarto *et al*., 2020). Generally, various types of hardwood were used to manufacture liquid smoke (Diatmika *et al*., 2019). The quality of the liquid smoke produced

may vary depending on the type of wood used (Budaraga *et al*., 2016). In addition to imparting specific flavors and aromas to foods (Maulina *et al*., 2020), liquid smoke is reported to be antimicrobial (Septana *et al*., 2020) and antioxidant (Budaraga & Putra, 2021).

The conventional smoking processes generally use hardwood as fuel, such as melon wood (Umar *et al*., 2018), mangrove wood (Cissoko *et al*., 2020), teak wood (Daramola *et al*., 2020), coconut shells, and coconut husks (Nugroho *et al*., 2018). Corn cobs have also been reported to be used in the fish smoking process (Sukowati, 2023). According to literature, traditional fish smoking in North Sulawesi, Indonesia, uses smoke from burning coconut shells or wood charcoal (Landangkasiang *et al*., 2017; Primalasari *et al*., 2019; Saediman *et al*., 2021). Smoked fish processing has been known for a long time because of its traditional processing techniques, simplicity, ease of implementation, and low cost (Islamiyah, 2021). In North Sulawesi, Indonesia, smoked fish commonly known as *fufu* is traditionally made from skipjack (*Katsuwonus pelamis*); *julung julung* (*Hemiramphus far*) (Berhimpon *et al*., 2018); and scad fish (*Decapterus* spp.) known as *pinekuhe* (Ansar & Ijong, 2021). The smoked fish product of *julung julung* is a typical product from North Sulawesi and Gorontalo, commonly called *roa* or *galavea* (Azis & Akolo, 2020).

The study's reported that the conventional smoking process has disadvantages such as smoking time, concentration of carcinogenic substances, temperature, and inconsistent product quality (Racovita *et al*., 2020). Conventional smoking of food products has been shown to produce carcinogenic components such as polycyclic aromatic hydrocarbons (Jinadasa *et al*., 2020). Benzo(a)pyrene ($C_{20}H_{12}$) is one of the carcinogenic compounds produced from traditional smoking products (Jinadasa *et al*., 2020). One method to reduce carcinogenic compounds in smoked fish products is to use liquid smoke in the smoking process (Nithin *et al*., 2020; Xin *et al*., 2021).

The utilization of liquid smoke in the fish smoking industry in North Sulawesi is very likely to be developed. North Sulawesi was reported to have both large-scale and domestic fish-smoking industries (Dotulong *et al*., 2018; Primalasari *et al*., 2019). Making liquid smoke is relatively simple, so it will be readily accepted by fish-smoking industry players (Ali & Al Fiqri, 2020). Previous research reported that using liquid smoke in smoked fish products resulted in good quality in terms of appearance, flavor, and aroma (Rizal *et al*., 2020). In addition, using liquid smoke in smoked fish products can add nutritional value and durability, making it possible to reach a wider market area (Ali *et al*., 2021).

Previous research reported that smoked fish dipped in liquid smoke for 15 minutes had a total plate count value of 4.7×10^4 CFU/g on day six and moisture content below 60% during storage (Suroso *et al*., 2018). Dipping fish in liquid smoke with a concentration of 15% and a dipping time of 60 minutes showed a bacterial colony of 2.12×10^2 CFU/g (Ali *et al*., 2021). The Indonesian National Standard on smoked fish requires a maximum total plate count of 5×10^4 CFU/g and a maximum moisture content of 60% (Indonesia Standardization Agency, 2013). Another study reported that smoked fish processed

with liquid smoke had a moisture content of 47.63%, phenol content of 12.62%, and pH of 4.8 (Berhimpon *et al*., 2018). The results of these studies indicate that liquid smoke is good to apply to smoked fish products. This study characterized smoked fish fillets of *julung julung* (*Hemiramphus* sp.) produced with liquid smoke from corn cobs waste. The study was conducted by dipping the fish fillets of *julung julung* with liquid smoke, with drying and steaming treatments on the fish fillets. This study aims to obtain the best smoking method for fish fillets using liquid smoke from corn cobs waste based on the assessment of total volatile bases, moisture content, water activity, pH value, phenol content, polycyclic aromatic hydrocarbon content, and sensory assessment.

Materials and methods

The main materials of the study were *julung julung* fish (*Hemiramphus* sp.), corn cob (*Zea mays* L.) from Gangga Island in Likupang, North Sulawesi, Indonesia. This study includes the preparation of liquid smoke from corn cobs waste, preparing fish (weeding and filleting), and treating fish fillets by dip in liquid smoke. Fresh *julung julung* fish was obtained from traditional fishermen in Manado, North Sulawesi, Indonesia (Fig. 1). The fish was placed in a cool box and given ice in a ratio of 1:1 (ice:fish) (Mishra *et al*., 2021). Then, the fish was washed, cleaned, and filleted. The cleaned fillets were dipped in a 5% salt solution for 30 minutes. The concentration of liquid smoke from corn cob waste was 0.8% (Berhimpon *et al*., 2018).

Commented [WT1]: Because based on the results of Berhimpon *et al*., 2018, the best concentration of liquid acid from corn cobs for fish smoking is at a concentration of 0.8%.

Fig. 1. *Julung julung* (*Hemiramphus* sp.)

The treatment in this study was applying liquid smoke from corn cobs waste to the smoking process of *julung julung* fillets, which was compared with the conventional method (using coconut wood, coconut shells, and corn cobs).

(A) : Fresh fillets were dipped in liquid smoke for 20 minutes and then dried in a cabinet dryer for 4 hours at 90 °C.

(B) : Fresh fillets were preheated in a cabinet dryer for 4 hours at 60 – 80 °C, then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated in a cabinet dryer for 4 hours at 90 °C.

(C) : Fresh fillets were steamed for 30 minutes, then dipped in liquid smoke for 20 minutes. After that, the fillets were dried in a cabinet dryer for 4 hours at 90 °C.

(D) : Fresh fillets were smoked with the conventional method using wood and coconut shell for 6 hours at 90 ºC.

(E) : Fresh fillets were smoked with the conventional method using corncob for 6 hours at 90 ºC.

Commented [WT2]: The treatment in this study was based on the results of Berhimpon et al. (2018) with some modifications, namely smoking fish without liquid smoke (using wood, head, coconut shell, and corn cobs).

Liquid smoke manufacturing process

Making liquid smoke refers to modified research (Aladin *et al*., 2018). Modifications were made to the equipment used in the pyrolysis process. Dry corn cobs were weighed to determine fuel efficiency. Corn cobs are gradually burned into the combustion furnace, at 400 ºC and have a pyrolysis time of about 120 minutes. The furnace was closed to prevent smoke from escaping from the tank. The smoke generated from the combustion flows through a pipe connected to a storage tank covered with ice cubes. The smoke that passes through the pipe will become cold, so condensation occurs, turning the smoke into liquid. The smoke that has been formed is collected into a container attached to the end of the pipe. The liquid smoke obtained is then allowed to settle the tar formed for three weeks and filtered (using Whatman filter paper No. 10) to obtain clear liquid smoke.

Commented [WT3]: Based Aladin *et al*., 2018

Commented [WT4]: charcoal formed from the pyrolysis process

Commented [WT5]: Based Aladin *et al*., 2018

Commented [WT6]: The level of benzopyrene from liquid smoke was evaluated by HPLC. And the benzopyrene in liquid smoke was about 0.5 µg/kg.

Total volatile bases assay

Total Volatile Base (TVB) assay refers to research by Tambunan & Chamidah (2021). TVB assay aims to determine the amount of volatile base compounds formed from protein breakdown—the principle of TVB analysis, namely by evaporating volatile base compounds at room temperature for 24 hours. The compound is then bound with boric acid and titrated with an HCl solution. After the boric acid solution was stored in the inner chamber of the Conway cell containing the blank (V_0) and the filtrate (V_1), the boric acid solution was titrated with 0.02 N HCl so that the boric acid solution changed color to pink. TVB levels in the smoked fish meat were expressed as mg N/100g.

$$TVB\left(\mathrm{mg}\frac{\mathrm{N}}{100\mathrm{g}}\right) = \frac{(V_1 - V_0) \times N.HCl \times dilution \times 100}{sample\ weight\ (g)}$$

Moisture content assay

The moisture content is determined by the Indonesia National Standard (Indonesia Standardization Agency, 2006a). An empty porcelain cup is weighed (A). Then, the sample was weighed to ± 2 g in a porcelain cup of known mass (B). The samples were dried in an oven at 105°C for 20 hours. The samples were then cooled in a desiccator, after which the samples in the cup were weighed (C). Moisture content was expressed as %.

$$Moisture\ (\%) = \frac{B\ (g) - C\ (g)}{B(g) - A\ (g)} \times 100\%$$

Water activity (Aw) assay

Water activity assay used an Aw meter (Rotronic, HygroPalm 23-AW-A). The Aw meter was set up at room temperature for 2 hours. The water activity of smoked fish was measured by placing the sample into a sample container and conditioning it for 30-60 minutes. The Aw meter sensor is contacted with the sample in the container. Then the water activity (Aw) value can be read on the Aw meter panel (Saputra *et al*., 2014).

pH assay

The sample weighed as much as 30 g, then 400 mL of distilled water was added at 28 ºC. The sample was homogenized with a magnetic stirrer to be evenly stirred. The pH meter electrode was inserted into the sample solution. The data taken in this test is the pH value read by the pH meter (Hanna Instruments HI99192) (Lekahena & Jamin, 2018).

Phenol level assay

Samples were extracted by adding 1 mL ethanol p.a., 5 mL distilled water, and 0.5 mL Folin Ciocalteu 50%, homogenized, and allowed to stand for 5 minutes. Then, 1 mL of 5% Na_2CO_3 and left in the dark for ± 60 minutes with gallic acid as a standard. The absorbance value was read on a spectrophotometer (Thermo Scientific Genesys 50) with a wavelength of 725 nm. Total phenolics were expressed as mg/g (Indonesia Standardization Agency, 2004; Muliadin *et al*., 2022).

$$Total\ Phenol\ (\frac{mg}{g}) = \frac{Gallic\ acid\ equivalence\ \left(\frac{mg}{L}\right) \times Volume\ (L)}{Sample\ weight\ (g)}$$

Polycyclic aromatic hydrocarbon (PAH) assay

PAH content testing was carried out based on the instructions Basak *et al*. (2010). A 5 g sample was weighed and collected in a 100 ml flask. Then 5 mL of 50% KOH solution, 75 mL of methanol, 1 mL of internal standard solution (9,10 dimethylantracene), and some boiling stones were added. After that,

the mixture was boiled for 4 hours in a soxhelet. The liquid phase was transferred to a separatory funnel and extracted with 100 mL of n-hexane with shaking for 3 min. The MeOH:KOH phase was drained and discarded. The combined n-hexane phase was rinsed twice, each with 50 mL of H_2O-MeOH (8:1) and 50 mL of water. The methanol-water and water phases were dried and discarded. The organic phase was concentrated in a rotary evaporator (40°C) to a volume of about 10 mL. The concentrated extract was dried under nitrogen gas.

After that, the silica gel column was cleaned by dissolving 15 g of silica gel in n-hexane and transferred to a column (200 mm long; diameter: 12 mm). Then, 1 g of anhydrous sodium sulfate was added. The concentrated sample extract was transferred to the column and eluted with 1 mL of n-hexane. A stock solution containing 0.1 mg mL^{-1} of 9,10 dimethylantracene was dissolved in n-hexane and stored at 4°C in a volumetric flask (with a glass stopper) wrapped in aluminum foil to avoid possible light degradation. Working standard solutions were prepared from the stock solutions. Analysis of PAH compounds used a Hewlett-Packard 1100 HPLC equipped with an Agilent-110 fluorescence detector. The injection volume was 10 µL. Mobile phase gradient (acetonitrile-water): 80% acetonitrile + 20% water with a flow rate of 1 ml min^{-1} and a wavelength of 270 nm. The external standard mixture was used for PAH concentration calculation.

Sensory assessment assay

Sensory assessment assay refers to the Indonesia National Standard (Indonesia Standardization Agency, 2006b). The sensory assessment was based on a hedonic scale (Table 1). Smoked fish samples were placed on a plastic plate with a glass of water, coded, and presented to 30 randomly selected semi-trained panelists (university students who had studied sensory assessment techniques), and the assessment process was carried out under light. The parameters observed were appearance, aroma, taste, and texture. Panelists rated acceptance of the samples on a scale of 1 – 9.

Commented [WT7]: Without preparation, karena sampel ikan asap yang digunakan masih layak dikonsumsi (dibawah 24 jam sejak pengasapan)

Table 1. Score sheet of hedonic assessment

Specification	Score
Really like it	9
Really like	8
Like	7
Rather like	6
Neutral	5
Rather dislike	4
do not like	3
Very dislike	2
Really don't like it	1

Statistical analysis

Statistical analysis was performed using Statistical Product and Service Solutions (SPSS) version 20.0. Statistical analysis of data was performed based on one-way analysis of variance (ANOVA) using a significance level of ($P<0.05$). Specific group differences were determined using Duncan's test. The TVB, total phenol, pH, moisture, water activity, and sensory assessment parameters were evaluated with three replicates.

Results and discussion

Liquid smoke production was carried out using a simple liquid smoke distillation device. This process consumed 17 kg of corn cobs and 12 kg of crushed ice. Liquid smoke from corn cobs is more accessible than coconut shells or wood (Maulina & Karo, 2021). Using coconut shells or wood as fuel will form a flame, thus reducing the volume of smoke in the furnace (Kabir Ahmad *et al*., 2022). The smoked *julung julung* were analyzed for TVB, moisture content, water activity, pH value, phenol content, PAH content, and sensory assessment.

Total volatile bases

The total volatile base (TVB) value is measured to determine the quality of the smoked fish. TVB value is one of the parameters used to determine the decline in fish quality and measures the amount of volatile base compounds formed due to protein degradation (Castro *et al*., 2006). Volatile bases formed in fish muscle tissue mainly consist of ammonia, trimethylamine (TMA), and dimethylamine (DMA) (VELP Scientifica, 2013). TVB value is an essential characteristic for the quality assessment of seafood products and is emerging as the most commonly used chemical parameter to assess the palatability of seafood (Rasulu *et al*., 2020). The results of *total volatile bases* on smoked fish fillets of *julung julung* are presented in Fig. 2.

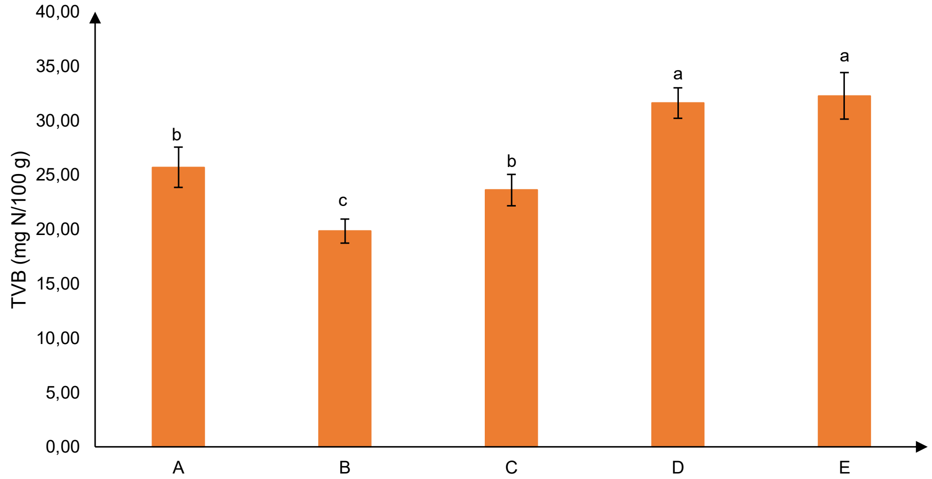

Fig. 2. Total volatile bases of *julung julung* smoked fillet

Fig. 2 shows the analysis of the variance of smoked fish fillets with different smoking method treatments affecting changes in TVB content ($p<0.05$). The TVB content of smoked fish fillets ranged from 19.83 – 32.27 mg N/100g, with the lowest TVB value in treatment B. The treatment of the smoking method with liquid smoke from corn cob waste (treatments A, B, and C) had lower TVB levels when compared to the conventional smoking method (treatments D and E). A literature search has not found why liquid smoking has a lower TVB value than conventional smoking methods. However, one possibility is that the liquid smoke smoking method allows the smoke components to be absorbed into the fish meat better than the conventional smoking method. Better absorption of liquid smoke phenol components in fish meat can inhibit and control microbial growth that causes a decrease in the quality of smoked fish (Santoso *et al*., 2015). A literature study showed that the average TVB value of smoked mackerel (*Euthynnus affinis*) treated with liquid smoke addition ranged from 24.63 – 28.38 mg N/100g (Hardianto & Yunianta, 2015). Another study reported smoked mackerel with a smoking process using rubber wood liquid smoke, producing smoked fish products with lower TVB values (Suroso *et al*., 2018). The statistical analysis showed a difference between treatments B, A – C, and D – E on the TVB value of smoked fish fillets. The analysis of variance indicates that the drying process of smoked fish fillets before immersion into liquid smoke can maintain the quality of smoked fish (Kaparang *et al*., 2013). The statistical analysis also showed that treatments A – C and D – E had no difference in TVB value, indicating that the moisture content between the two treatments was not different. TVB value is closely related to moisture content (Rasulu *et al*., 2020).

The European standard (European Market Observatory for Fisheries and Aquaculture, 2020) states that the limit range of TVB value for smoked fish is 25 – 35 mg N/100 g. All treatments' TVB value of the smoked fish fillets still met the European standard. Previous studies reported TVB values of fresh fish fillets of 8.70 ± 0.86 mgN/100g (Moosavi-Nasab *et al*., 2021) and 12.94 ± 0.92 mg N/100g (Bouzgarrou *et al*., 2020). Messina *et al*. (2021) reported that smoked fish fillets that underwent two drying processes had a TVB value of <20 mg N/100g. Another study also reported the TVB value of smoked fish fillet samples with hot smoking and cold smoking, which amounted to 17.80 ± 0.17 and 18.95 ± 0.20 mg N/100g, respectively (El-Lahamy *et al*., 2019). Aydin *et al*. (2020) reported a TVB value of smoked fish with hot smoking of 11.82 ± 0.05 mg N/100g.

Moisture content

Moisture content is an important parameter to determine the quality of smoked fish produced. The moisture content contained in smoked fish can affect the shelf life of smoked fish because water in food is a medium for microbes to grow (Kaban *et al*., 2019). The smoking process was reported to reduce the moisture content of fresh fish to a range between 9 and 17% (Sikoki & Aminigo, 2002). The moisture content of smoked fish fillets is presented in Fig. 3.

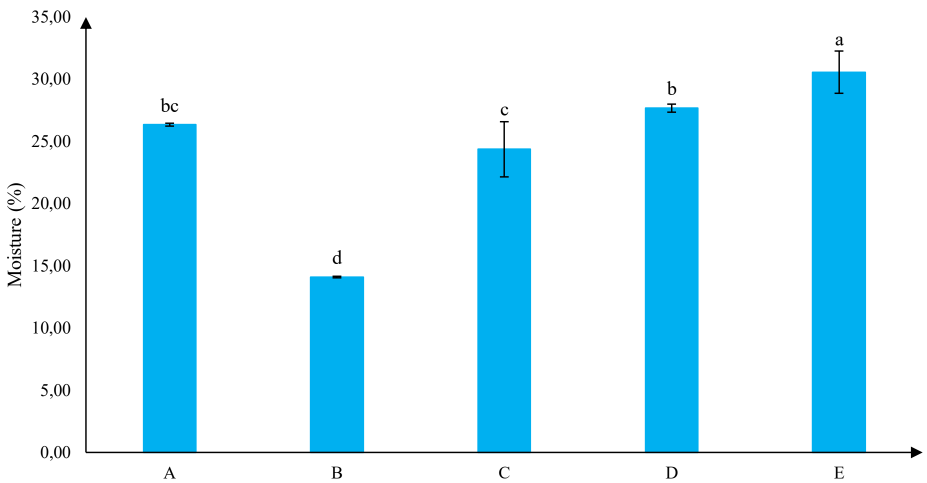

Fig. 3. Moisture content of *julung julung* smoked fillet

Fig. 3 shows the analysis of the variance of smoked *julung julung* fillets with different smoking methods treatment affects moisture content ($p<0.05$). The moisture content of smoked *julung julung* fillets ranged from 14.10 – 30.57%, with the lowest moisture content in treatment B. The moisture content

value in smoked fish products from all treatments still meets the Indonesian National Standard No. 2725:2013 on smoked fish, which is a maximum of 60% (Indonesia Standardization Agency, 2013). Darianto *et al*. (2018) reported that the smoking process can reduce the moisture content of fish to below 40%, which can help preserve it longer. The treatment of the smoking method with liquid smoke with corn cob (Treatment A, B, C) has a lower moisture content when compared to the treatment of the conventional smoking method (Treatment D and E). This result is because the smoking chamber is not fully enclosed in the conventional smoking method, so the heat generated could be more optimal. Suboptimal heat can increase moisture content and cause the moisture content of smoked fish to decrease only slightly (Amos & Paulina, 2017). Whereas in the liquid smoke smoking method, temperature and humidity can be controlled better so that the moisture content of the product can be reduced efficiently (Salindeho & Lumoindong, 2017).

The statistical analysis showed a difference between treatments B, A – C, and D – E on the TVB value of smoked fish fillets showed a difference between treatment B, treatment A – C, and treatment D – E on the moisture content of smoked fish fillets. The results of the variance study indicate that the drying process of smoked fish fillets before the dip in liquid smoke in treatment B can help reduce the moisture content. This study's results are from previous research, which also reported a significant decrease in moisture content in smoked fish products with two times drying treatment (Messina *et al*., 2021). The statistical analysis also showed that treatments A and C were not different because steaming in treatment C will increase the moisture content of the product (Salmatia *et al*., 2020). The conventional smoking method with corn cob fuel has the highest moisture content; this result is the conventional smoking process with corn cobs, and the heat generated is lower than smoking using wood (Asmara *et al*., 2022).

Water activity (Aw)

Water activity is one of the essential parameters in the quality of smoked fish. Water activity (Aw) is expressed as the ratio of the vapor pressure in the food to the vapor pressure of pure water, and it predicts whether water tends to move from the food product into the cells of microorganisms that may be present. A well-smoked fish has a water activity of <0.50 and a moisture content between 15 and 25% to inhibit the growth of pathogenic microorganisms in smoked fish products (Mondo *et al*., 2020). According to British Columbia Centre for Disease Control (2013), the maximum water activity value for smoked fish is 0.97. Water activity correlates with the moisture content of a smoked fish product and is two important factors affecting food safety and quality (Fitri *et al*., 2022). The water activity values of smoked fish fillets can be seen in Fig. 4.

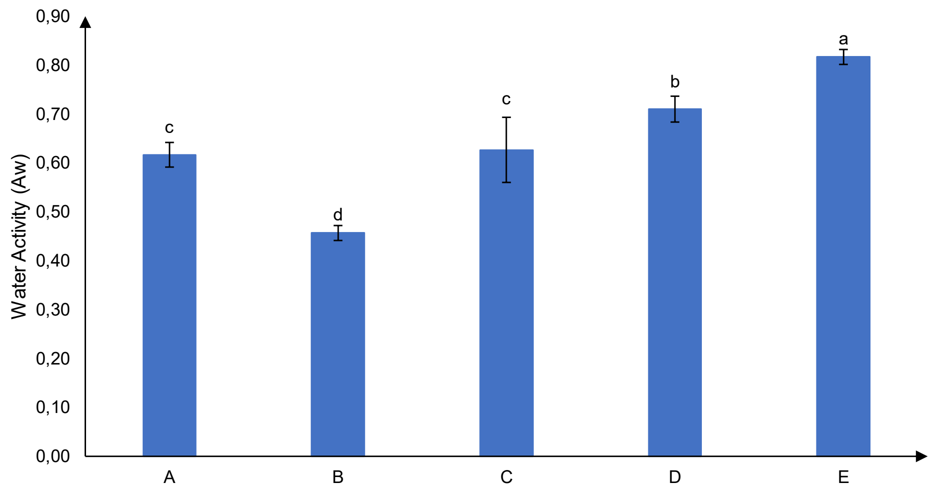

Fig. 4. Water activity of *julung julung* smoked fillet

Fig. 4 shows the analysis of the variance of smoked fish fillet *julung julung* with different smoking method treatments affecting water activity ($p<0.05$). The water activity value of smoked fish fillet ranged from 0.46 – 0.82, with the lowest water activity in treatment B. The treatment of the smoking method with corn cob liquid smoke (Treatment A, B, C) had a lower water content when compared to the treatment of the conventional smoking method (treatment D and E).

The water activity value of fish meat can vary depending on the cooking method, temperature, and duration of heating (Oliveira *et al*., 2017; Zhang *et al*., 2023). Therefore, the factor of fish fillet treatment before immersion in liquid smoke caused the difference in water activity value in treatment B compared to treatments A and C. The water activity value is directly proportional to the water content value of each treatment. The heating process can reduce the moisture content of fish meat (Kiczorowska *et al*., 2019), thus affecting the water activity of fish meat (Gómez *et al*., 2020).

pH

The pH value of smoked fish is an essential factor affecting its quality. The pH value will decrease with increasing smoking time (Baten *et al*., 2020b). The analysis of the variance of smoked fish fillets *julung julung* with different smoking method treatments affects the pH value ($p<0.05$). The pH of smoked fish fillet ranged from 4.60 to 5.81 (Fig. 5). The treatment of the smoking method with corn cob liquid smoke (treatments A, B, and C) had a lower pH value when compared to the conventional smoking method (treatments D and E). Swastawati *et al*. (2022) also reported that the pH value of smoked barracuda fish with the liquid smoke method was significantly lower than that of the conventional

method. Another study also reported that using liquid smoke can reduce pH caused by the condensation of organic acids in the smoking process (Puke & Galoburda, 2020). The pH value of smoked fish correlates with the phenol content contained in the smoke component, and an increase in phenol content causes a decrease in pH (Berhimpon *et al*., 2018).

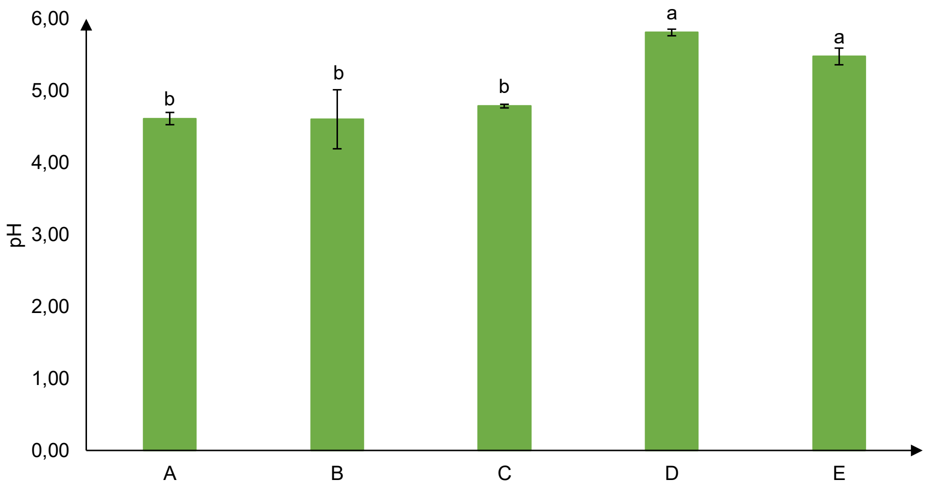

Fig. 5. pH of *julung julung* smoked fillet

Phenol level

Phenol is a compound found in wood smoke. Phenol compounds enter food through diffusion and capillary action, affecting taste, color, and aroma and extending shelf life (Remy *et al*., 2016). In addition, phenol compounds are also reported to act as antioxidants that can prevent rancidity in fish meat (Sérot *et al*., 2004). Leksono *et al*. (2020) also noted that the higher the phenol level in smoke, the stronger the flavor and aroma of smoked fish. The analysis of the variance of smoked fish fillets julung julung with different smoking method treatments affecting phenol level ($p<0.05$). The phenol level of smoked fish fillet ranged from 4.42 to 16.11 mg/g (Fig. 6). The treatment of the smoking method with corn cob liquid smoke (treatments A, B, and C) had higher phenol level when compared to the conventional smoking method (treatments D and E).

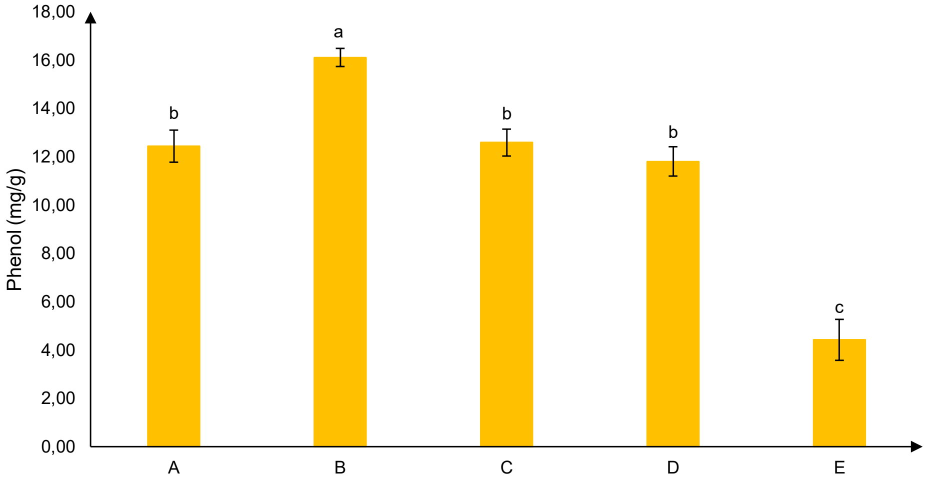

Fig. 6. Phenol level of *julung julung* smoked fillet

The statistical analysis showed a difference between treatments B, A – C, and D – E on the TVB value of smoked fish fillets showed phenol levels in treatments A, C, and D were not different. Previous research reported that the drying treatment of fish meat can increase phenolic compounds in smoked fish products (Sérot *et al*., 2004). The high phenol content in treatment B is thought to be because the pre-heated process causes the fish meat to lose water so that when dipped in liquid smoke, the fish fillets absorb a large amount of liquid smoke. Previous studies have reported that when the fish surface is dried, there is less smoke condensation than products smoked at lower temperatures. The results of this study indicate that a dry fish surface allows for better absorption of smoke components (Belichovska *et al*., 2019).

The study reported phenol levels in liquid smoke from corn cob waste of 0.335 mg/g (Swastawati *et al*., 2007) and 2.55% (Leviyani *et al*., 2019). The phenol level of smoked fish with the conventional method of smoking with corn cobs is lower due to the high acid content, which can potentially reduce the phenol content of smoked fish (Swastawati *et al*., 2012). Anggraini & Nurhazisa (2017) reported that the phenol content of liquid smoke from coconut shells was 3.04%, while liquid smoke from corn cob was 1.38%.

Polycyclic aromatic hydrocarbon (PAH) levels

Polycyclic aromatic hydrocarbons are a group of organic compounds formed by incomplete combustion, such as wood. Therefore, the type of fuel used in the fish smoking process affects the PAH content of smoked fish (Jinadasa *et al*., 2020). One of the carcinogenic PAHs, benzo(a)pyrene, is a carcinogenic

marker in smoked fish products (Stołyhwo & Sikorski, 2005). Polycyclic aromatic hydrocarbon (PAH) levels in smoked fish fillets can be seen in Table 2.

Table 2. Polycyclic aromatic hydrocarbon levels in *julung julung* smoked fillet

Commented [WT8]: PAH testing is not replicated because because it uses the HPLC method

Polycyclic Aromatic Hydrocarbon	Smoked *Julung Julung* fish (µg/kg)					Corn Cob Liquid Smoke 0.8%(µg/kg)
	A	B	C	D	E	
Benzo(a)pyrene	nd	nd	nd	1.5	0.2	0.5
Benzo(b)fluoranthene	1.2	1.0	1.2	2	1.8	2.5
Benzo(a)anthracene	nd	nd	nd	0.5	nd	0.2
Benzo(g)perylene	0.4	0.2	0.4	1.0	0.8	1.0

nd = not detected

Table 2 shows that the PAH levels in smoked fish are still below the standard required in the Indonesian National Standard No. 2725 of 2013 on smoked fish, which is benzo(a)pyrene max 5 µg/kg (Indonesia Standardization Agency, 2013). The polycyclic aromatic hydrocarbon compound regulated in the Indonesian National Standard is only benzo(a)pyrene. Stołyhwo & Sikorski (2005) reported benzo(a)pyrene levels in smoked fish with hot and cold smoking processes ranging from 0.05 to about 60 µg/kg. Other studies have reported benzo(b,k)fluoranthene levels in salmon and rainbow trout ranging from 1.83 to 9.55 µg/kg, while benzo(g,h)perylene levels in salmon were 0.44 µg/kg (Basak *et al*., 2010). Berhimpon *et al*. (2018) also reported benzo(a)pyrene levels in smoked skipjack fish of 0.25 µg/kg. The level of benzo(a)pyrene in smoked barracuda was reported to be 0.32 µg/kg, benzo(b)fluoranthene 0.35 µg/kg, benzo(k)fluoranthene 0.21 µg/kg, benzo(a)anthracene 0.44 µg/kg, and benzo(g,h)perylene 2.56 µg/kg (Asamoah *et al*., 2021).

High PAH levels in smoked fish are due to the lignin content of the material used as fuel in the smoking process (Asamoah *et al*., 2021). The lignin content in corn cobs is reported to be about 17 – 21% (Thangavelu *et al*., 2018; Olajuyigbe *et al*., 2019; Gandam *et al*., 2022). The lignin content of coconut shell was approximately 59.5% (Wang & Sarkar, 2018), while that of coconut wood was about 50% on a dry weight basis (Anuchi *et al*., 2022). Lignin is reported to absorb PAH compounds because PAH compounds can bind well with lignin (Oliveira *et al*., 2019). During pyrolysis and gasification processes, lignin components react to produce aromatic tar and coke, which can form PAH compounds (Zhou *et al*., 2014; Kawamoto, 2017). Therefore, PAH compounds in smoked fish smoked using corncobs are lower than those smoked with shells and coconut wood.

Sensory assessment

A sensory assessment is carried out to evaluate the panelist's preference level, including appearance, aroma, taste, and texture. A sensory assessment must determine the quality of the smoked fish product and ensure it meets the applicable standards (Hadanu & Lomo, 2019). The sensory characteristics of

smoked fish products affect consumer acceptance and preference. Sensory assessment by panelists helps to identify the sensory attributes that are most appealing to consumers, allowing manufacturers to customize products according to consumer assessment (Ekelemu *et al*., 2021). Indonesian National Standard No. 2725:2013 regarding smoked fish requires a minimum sensory assessment of 7 (Score 1-9) (Indonesia Standardization Agency, 2013). The results of the panelists' assessment of *julung julung* smoked fillet from each treatment can be seen in Fig. 7.

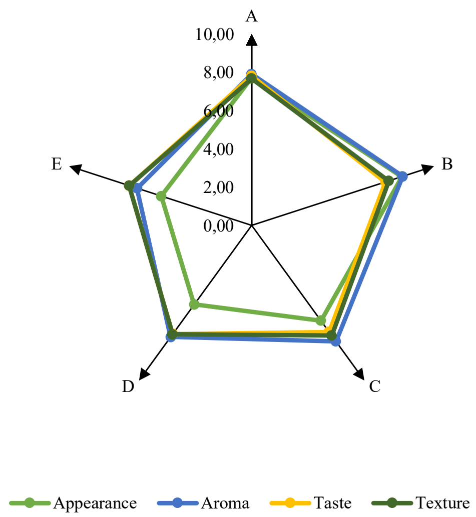

Fig. 7. Sensory assesment of *julung julung* smoked fillet

Fig. 7 shows the analysis of the variance of the sensory assessment of the appearance of smoked *julung julung* fillets with different smoking method treatments affecting the appearance of smoked fish ($p<0.05$). The panelists' assessment of the appearance of smoked fish fillets ranged from 4.95 to 8.24, with the highest panelists' assessment in treatment B. Based on the requirements of the Indonesian National Standard, only treatments A and B met the minimum panelist assessment requirement of 7. The moisture content factor is thought to have influenced the panelists' assessment of the appearance of smoked fish, so panelists less favored treatment C with steaming. Moisture content can affect the physical properties of fish, such as the appearance and texture of smoked fish (Baten *et al*., 2020a). Smoked fish with high moisture content will make the color of smoked fish look paler (Flick, 2010). Treatments D and E were less favored by panelists, presumably because conventional smoking methods produce darker products (dark brown), less bright and less shiny. Smoking fish with liquid smoke has smoked fish products with a golden color and clean and shiny surface (Berhimpon *et al*., 2018). Another

factor that affects the appearance of smoked fish is the length of the smoking process. The longer the smoking process, the darker the color will be. The time of the smoking process for smoked fish using the liquid smoke method is shorter than the conventional method (Puke & Galoburda, 2020; Baten *et al*., 2020b). The smoking process can change the color of fish, giving it a distinctive golden color due to the interaction of carbonyls with amino components on the surface of the meat. The color and appearance of smoked fish also positively correlate with phenol content. Phenol compounds in smoke interact with amino acid components in fish meat, producing a distinctive golden color in smoked fish (Montazeri *et al*., 2013).

Fig. 7 shows the analysis of the variance of the sensory assessment of the aroma of smoked fish fillets *julung julung* with different smoking method treatments that did not affect the appearance of smoked fish ($p>0.05$). However, the aroma sensory assessment of treatments A, B, C, and D still meets the minimum assessment of the Indonesian National Standard on smoked fish. The sensory assessment of smoked fish aroma was lowest in treatment E. Previous studies have reported that smoking fish with corn cobs as a fuel source resulted in the lowest aroma scores, indicating less aromatic smoked fish (Asmara *et al*., 2022). The aroma and taste of smoked fish are strongly influenced by the time of smoking (Tahir *et al*., 2020). This assessment indicates that the time of the smoking process of 4 and 6 hours in all treatments has not influenced the taste and aroma of smoked fish.

Fig. 7 shows the analysis of the variance of the sensory assessment of the taste of smoked fish fillets *julung julung* with different smoking method treatments that did not affect the appearance of smoked fish ($p>0.05$). The taste of smoked fish is influenced by the time of the smoking process. Previous studies have reported that longer smoking time can improve the sensory attributes of taste (Baten *et al*., 2020b). This result indicates that the 2-hour time difference between the liquid smoke smoking treatment and the conventional smoking method has not affected the taste of smoked fish. A distinctive smoked taste without bitterness is a criterion for assessing the taste of good quality smoked fish (Sukowati *et al*., 2021).

Fig. 7 shows the analysis of variance of the sensory assessment of the texture of smoked fish fillet *julung julung* with different smoking method treatments giving effect to the texture of smoked fish ($p<0.05$). Panelists' assessment of the texture of smoked fish *julung julung*, ranged from 6.71 – 7.67, with the highest panelist assessment in treatments A and B. The statistical analysis showed that treatments A and B differed from treatments C, D, and E. This result was thought to be because the fish fillets were dipped in liquid smoke, giving a dry, compact, and tender texture. Whereas in treatment C (fish fillets subjected to steaming), the texture of the smoked fillets was rather sticky and not solid. Treatments D and E produced the texture of smoked fish fillets which were less dense and not compact. The texture of smoked fish is negatively correlated with its moisture content. The higher the moisture content in

smoked fish, the softer and less firm the texture (Chan *et al*., 2022). High moisture content in smoked fish products results in less dense fish muscle fibers (Chang *et al*., 2021).
A good texture of smoked fish is compact, moderately elastic, and not sticky (Sulistijowati *et al*., 2021). The Indonesian National Standard requires that the texture of smoked fish is dense and compact and the tissue is very tight (Indonesia Standardization Agency, 2013). Based on consumer surveys, the preferred texture of smoked fish is dense, slightly wetter, and easy to chew, and there is a sensation of liquid in the mouth when chewed (Ticoalu *et al*., 2019).

Conclusion

Based on the evaluation of the parameters of total volatile bases, moisture content, water activity, pH, phenol content, polycyclic aromatic hydrocarbon content, and sensory assessment of smoked *julung julung* fillets, it can be concluded that treatment B is the best treatment, i.e. the fillets were preheated for 4 hours at 60 – 80 °C, then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated for 4 hours at 90 °C. In general, the characteristics of smoked fillets using liquid smoke were better when compared to the conventional smoking treatment. It is necessary to evaluate different smoking times on smoked *julung julung* fillets with corn cob liquid smoke method.

Acknowledgment

Author contributions

Conflicts of interest

The authors declare that there is no conflict of interest.

Highlights

Effectiveness of using liquid smoke derived from corn cob waste in the smoking process of *julung julung* fillets

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Characteristics of *Julung Julung* Smoked Fillets (*Hemiramphus* sp.) using Liquid Smoke from Corn Cobs Waste

Abstract

This study aimed to obtain the best smoking method for *julung julung* fillets (*Hemiramphus* sp.) using liquid smoke from corn cob waste. This study used three different fillet treatment methods (dried and steamed) before liquid smoke application. This study also compared fish smoking between using liquid smoke with conventional methods (using shell, coconut wood, and corn cob). The parameters to determine the quality of smoked fish were Total Volatile Bases (TVB), moisture, water activity, pH, phenol levels, polycyclic aromatic hydrocarbon (PAH), and sensory assessment. The results showed that the TVB of smoked *julung julung* fillets ranged from 19.83 – 32.27 mg N/100g. The moisture ranged from 14.10 – 30.57%. Water activity ranged from 0.46 – 0.82. pH ranged from 4.60 – 5.81. Phenol levels ranged from 4.42 – 16.11 mg/g. PAH levels are still below the standard required in the Indonesian National Standard. Panelists rated neutral to really like the appearance, aroma, taste, and texture of smoked fish. From these research, it can be concluded that treatment B is the best treatment, namely fresh fillets are first heated for 4 hours at 60 – 80 ^{o}C and then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated for 4 hours at 90 ^{o}C.

Keywords: Corn cobs, Indonesia, Liquid smoke, North Sulawesi, Wood

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Reviewer comment for author

1-The responses to reviewers comments should be attached (Reviewers 1, 2 and 3), (Necessary)

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Introduction

Corn is the second most widely grown crop in Indonesia after rice. Indonesia's maize production in 2023 is 14.46 million tons, ranking 9th in the world. (Indonesia Center of Statistic Agency, 2023). North Sulawesi is one of the contributing regions that produce corn in Indonesia. Considerable corn production will also produce waste, especially corn cobs, which are generally thrown away and burned by most Indonesians (Cahyadi *et al*., 2021).

One of the corn cobs was used to make liquid smoke. Previous research reported that corn cobs can produce liquid smoke, a by-product of the pyrolysis of corn cob waste. The yield of liquid smoke from corn cobs is about 28.37%, with a pH value of 3.5 (Sriharti *et al*., 2020). The particle size of corn cobs affects the yield of liquid smoke, with smaller particle sizes resulting in higher yields (Aladin *et al*., 2018). Swastawati et al. (2007) reported the phenolic content in liquid smoke produced from 335 mg/L corn cobs.

Liquid smoke is a natural product made from the condensation of smoke from burning wood (Andy *et al*., 2021). Liquid smoke is commonly used as a flavoring in food to provide a smoked flavor without the food undergoing the actual smoking process (Sari *et al*., 2006). Using liquid smoke in food will save time, energy, and labor, so that can reduce production costs (Krah *et al*., 2019). In addition, using liquid smoke in food can speed up and standardize the smoking process, adding flavor and microbiological safety while reducing production time (Indiarto *et al*., 2020). Generally, various types of hardwood were used to manufacture liquid smoke (Diatmika *et al*., 2019). The quality of the liquid smoke produced

may vary depending on the type of wood used (Budaraga *et al*., 2016). In addition to imparting specific flavors and aromas to foods (Maulina *et al*., 2020), liquid smoke is reported to be antimicrobial (Septana *et al*., 2020) and antioxidant (Budaraga & Putra, 2021).

The conventional smoking processes generally use hardwood as fuel, such as melon wood (Umar *et al*., 2018), mangrove wood (Cissoko *et al*., 2020), teak wood (Daramola *et al*., 2020), coconut shells, and coconut husks (Nugroho *et al*., 2018). Corn cobs have also been reported to be used in the fish smoking process (Sukowati, 2023). According to literature, traditional fish smoking in North Sulawesi, Indonesia, uses smoke from burning coconut shells or wood charcoal (Landangkasiang *et al*., 2017; Primalasari *et al*., 2019; Saediman *et al*., 2021). Smoked fish processing has been known for a long time because of its traditional processing techniques, simplicity, ease of implementation, and low cost (Islamiyah, 2021). In North Sulawesi, Indonesia, smoked fish commonly known as *fufu* is traditionally made from skipjack (*Katsuwonus pelamis*); *julung julung* (*Hemiramphus far*) (Berhimpon *et al*., 2018); and scad fish (*Decapterus* spp.) known as *pinekuhe* (Ansar & Ijong, 2021). The smoked fish product of *julung julung* is a typical product from North Sulawesi and Gorontalo, commonly called *roa* or *galavea* (Azis & Akolo, 2020).

The study's reported that the conventional smoking process has disadvantages such as smoking time, concentration of carcinogenic substances, temperature, and inconsistent product quality (Racovita *et al*., 2020). Conventional smoking of food products has been shown to produce carcinogenic components such as polycyclic aromatic hydrocarbons (Jinadasa *et al*., 2020). Benzo(a)pyrene ($C_{20}H_{12}$) is one of the carcinogenic compounds produced from traditional smoking products (Jinadasa *et al*., 2020). One method to reduce carcinogenic compounds in smoked fish products is to use liquid smoke in the smoking process (Nithin *et al*., 2020; Xin *et al*., 2021).

The utilization of liquid smoke in the fish smoking industry in North Sulawesi is very likely to be developed. North Sulawesi was reported to have both large-scale and domestic fish-smoking industries (Dotulong *et al*., 2018; Primalasari *et al*., 2019). Making liquid smoke is relatively simple, so it will be readily accepted by fish-smoking industry players (Ali & Al Fiqri, 2020). Previous research reported that using liquid smoke in smoked fish products resulted in good quality in terms of appearance, flavor, and aroma (Rizal *et al*., 2020). In addition, using liquid smoke in smoked fish products can add nutritional value and durability, making it possible to reach a wider market area (Ali *et al*., 2021).

Previous research reported that smoked fish dipped in liquid smoke for 15 minutes had a total plate count value of 4.7×10^4 CFU/g on day six and moisture content below 60% during storage (Suroso *et al*., 2018). Dipping fish in liquid smoke with a concentration of 15% and a dipping time of 60 minutes showed a bacterial colony of 2.12×10^2 CFU/g (Ali *et al*., 2021). The Indonesian National Standard on smoked fish requires a maximum total plate count of 5×10^4 CFU/g and a maximum moisture content of 60% (Indonesia Standardization Agency, 2013). Another study reported that smoked fish processed

with liquid smoke had a moisture content of 47.63%, phenol content of 12.62%, and pH of 4.8 (Berhimpon *et al*., 2018). The results of these studies indicate that liquid smoke is good to apply to smoked fish products. This study characterized smoked fish fillets of *julung julung* (*Hemiramphus* sp.) produced with liquid smoke from corn cobs waste. The study was conducted by dipping the fish fillets of *julung julung* with liquid smoke, with drying and steaming treatments on the fish fillets. This study aims to obtain the best smoking method for fish fillets using liquid smoke from corn cobs waste based on the assessment of total volatile bases, moisture content, water activity, pH value, phenol content, polycyclic aromatic hydrocarbon content, and sensory assessment.

Materials and methods

The main materials of the study were *julung julung* fish (*Hemiramphus* sp.), corn cob (*Zea mays* L.) from Gangga Island in Likupang, North Sulawesi, Indonesia. This study includes the preparation of liquid smoke from corn cobs waste, preparing fish (weeding and filleting), and treating fish fillets by dip in liquid smoke. Fresh *julung julung* fish was obtained from traditional fishermen in Manado, North Sulawesi, Indonesia (Fig. 1). The fish was placed in a cool box and given ice in a ratio of 1:1 (ice:fish) (Mishra *et al*., 2021). Then, the fish was washed, cleaned, and filleted. The cleaned fillets were dipped in a 5% salt solution for 30 minutes. The concentration of liquid smoke from corn cob waste was 0.8% (Berhimpon *et al*., 2018).

Fig. 1. *Julung julung* (*Hemiramphus* sp.)

The treatment in this study was applying liquid smoke from corn cobs waste to the smoking process of *julung julung* fillets, which was compared with the conventional method (using coconut wood, coconut shells, and corn cobs).

(A) : Fresh fillets were dipped in liquid smoke for 20 minutes and then dried in a cabinet dryer for 4 hours at 90 °C.

(B) : Fresh fillets were preheated in a cabinet dryer for 4 hours at 60 – 80 °C, then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated in a cabinet dryer for 4 hours at 90 °C.

(C) : Fresh fillets were steamed for 30 minutes, then dipped in liquid smoke for 20 minutes. After that, the fillets were dried in a cabinet dryer for 4 hours at 90 °C.

(D) : Fresh fillets were smoked with the conventional method using wood and coconut shell for 6 hours at 90 ^{o}C.

(E) : Fresh fillets were smoked with the conventional method using corncob for 6 hours at 90 ^{o}C.

Liquid smoke manufacturing process

Making liquid smoke refers to modified research (Aladin *et al*., 2018). Modifications were made to the equipment used in the pyrolysis process. Dry corn cobs were weighed to determine fuel efficiency. Corn cobs are gradually burned into the combustion furnace, at 400 ^{o}C and have a pyrolysis time of about 120 minutes. The furnace was closed to prevent smoke from escaping from the tank. The smoke generated from the combustion flows through a pipe connected to a storage tank covered with ice cubes. The smoke that passes through the pipe will become cold, so condensation occurs, turning the smoke into liquid. The smoke that has been formed is collected into a container attached to the end of the pipe. The liquid smoke obtained is then allowed to settle the tar formed for three weeks and filtered (using Whatman filter paper No. 10) to obtain clear liquid smoke.

Total volatile bases assay

Total Volatile Base (TVB) assay refers to research by Tambunan & Chamidah (2021). TVB assay aims to determine the amount of volatile base compounds formed from protein breakdown—the principle of TVB analysis, namely by evaporating volatile base compounds at room temperature for 24 hours. The compound is then bound with boric acid and titrated with an HCl solution. After the boric acid solution was stored in the inner chamber of the Conway cell containing the blank (V_0) and the filtrate (V_1), the boric acid solution was titrated with 0.02 N HCl so that the boric acid solution changed color to pink. TVB levels in the smoked fish meat were expressed as mg N/100g.

$$TVB\left(\mathrm{mg}\frac{\mathrm{N}}{100\mathrm{g}}\right) = \frac{(V_1 - V_0) \times N.HCl \times dilution \times 100}{sample\ weight\ (g)}$$

Moisture content assay

The moisture content is determined by the Indonesia National Standard (Indonesia Standardization Agency, 2006a). An empty porcelain cup is weighed (A). Then, the sample was weighed to ± 2 g in a porcelain cup of known mass (B). The samples were dried in an oven at 105°C for 20 hours. The samples were then cooled in a desiccator, after which the samples in the cup were weighed (C). Moisture content was expressed as %.

$$Moisture\ (\%) = \frac{B\ (g) - C\ (g)}{B(g) - A\ (g)} \times 100\%$$

Water activity (Aw) assay

Water activity assay used an Aw meter (Rotronic, HygroPalm 23-AW-A). The Aw meter was set up at room temperature for 2 hours. The water activity of smoked fish was measured by placing the sample into a sample container and conditioning it for 30-60 minutes. The Aw meter sensor is contacted with the sample in the container. Then the water activity (Aw) value can be read on the Aw meter panel (Saputra *et al*., 2014).

pH assay

The sample weighed as much as 30 g, then 400 mL of distilled water was added at 28 ºC. The sample was homogenized with a magnetic stirrer to be evenly stirred. The pH meter electrode was inserted into the sample solution. The data taken in this test is the pH value read by the pH meter (Hanna Instruments HI99192) (Lekahena & Jamin, 2018).

Phenol level assay

Samples were extracted by adding 1 mL ethanol p.a., 5 mL distilled water, and 0.5 mL Folin Ciocalteu 50%, homogenized, and allowed to stand for 5 minutes. Then, 1 mL of 5% Na_2CO_3 and left in the dark for ± 60 minutes with gallic acid as a standard. The absorbance value was read on a spectrophotometer (Thermo Scientific Genesys 50) with a wavelength of 725 nm. Total phenolics were expressed as mg/g (Indonesia Standardization Agency, 2004; Muliadin *et al*., 2022).

$$Total\ Phenol\ (\frac{mg}{g}) = \frac{Gallic\ acid\ equivalence\ \left(\frac{mg}{L}\right) \times Volume\ (L)}{Sample\ weight\ (g)}$$

Polycyclic aromatic hydrocarbon (PAH) assay

PAH content testing was carried out based on the instructions Basak *et al*. (2010). A 5 g sample was weighed and collected in a 100 ml flask. Then 5 mL of 50% KOH solution, 75 mL of methanol, 1 mL of internal standard solution (9,10 dimethylantracene), and some boiling stones were added. After that,

the mixture was boiled for 4 hours in a soxhelet. The liquid phase was transferred to a separatory funnel and extracted with 100 mL of n-hexane with shaking for 3 min. The MeOH:KOH phase was drained and discarded. The combined n-hexane phase was rinsed twice, each with 50 mL of H_2O-MeOH (8:1) and 50 mL of water. The methanol-water and water phases were dried and discarded. The organic phase was concentrated in a rotary evaporator (40°C) to a volume of about 10 mL. The concentrated extract was dried under nitrogen gas.

After that, the silica gel column was cleaned by dissolving 15 g of silica gel in n-hexane and transferred to a column (200 mm long; diameter: 12 mm). Then, 1 g of anhydrous sodium sulfate was added. The concentrated sample extract was transferred to the column and eluted with 1 mL of n-hexane. A stock solution containing 0.1 mg mL^{-1} of 9,10 dimethylantracene was dissolved in n-hexane and stored at 4°C in a volumetric flask (with a glass stopper) wrapped in aluminum foil to avoid possible light degradation. Working standard solutions were prepared from the stock solutions. Analysis of PAH compounds used a Hewlett-Packard 1100 HPLC equipped with an Agilent-110 fluorescence detector. The injection volume was 10 µL. Mobile phase gradient (acetonitrile-water): 80% acetonitrile + 20% water with a flow rate of 1 ml min^{-1} and a wavelength of 270 nm. The external standard mixture was used for PAH concentration calculation.

Sensory assessment assay

Sensory assessment assay refers to the Indonesia National Standard (Indonesia Standardization Agency, 2006b). The sensory assessment was based on a hedonic scale (Table 1). Smoked fish samples were placed on a plastic plate with a glass of water, coded, and presented to 30 randomly selected semi-trained panelists (university students who had studied sensory assessment techniques), and the assessment process was carried out under light. The parameters observed were appearance, aroma, taste, and texture. Panelists rated acceptance of the samples on a scale of 1 – 9.

Table 1. Score sheet of hedonic assessment

Specification	Score
Really like it	9
Really like	8
Like	7
Rather like	6
Neutral	5
Rather dislike	4
do not like	3
Very dislike	2
Really don't like it	1

Statistical analysis

Statistical analysis was performed using Statistical Product and Service Solutions (SPSS) version 20.0. Statistical analysis of data was performed based on one-way analysis of variance (ANOVA) using a significance level of ($P<0.05$). Specific group differences were determined using Duncan's test. The TVB, total phenol, pH, moisture, water activity, and sensory assessment parameters were evaluated with three replicates.

Results and discussion

Liquid smoke production was carried out using a simple liquid smoke distillation device. This process consumed 17 kg of corn cobs and 12 kg of crushed ice. Liquid smoke from corn cobs is more accessible than coconut shells or wood (Maulina & Karo, 2021). Using coconut shells or wood as fuel will form a flame, thus reducing the volume of smoke in the furnace (Kabir Ahmad *et al*., 2022). The smoked *julung julung* were analyzed for TVB, moisture content, water activity, pH value, phenol content, PAH content, and sensory assessment.

Total volatile bases

The total volatile base (TVB) value is measured to determine the quality of the smoked fish. TVB value is one of the parameters used to determine the decline in fish quality and measures the amount of volatile base compounds formed due to protein degradation (Castro *et al*., 2006). Volatile bases formed in fish muscle tissue mainly consist of ammonia, trimethylamine (TMA), and dimethylamine (DMA) (VELP Scientifica, 2013). TVB value is an essential characteristic for the quality assessment of seafood products and is emerging as the most commonly used chemical parameter to assess the palatability of seafood (Rasulu *et al*., 2020). The results of *total volatile bases* on smoked fish fillets of *julung julung* are presented in Fig. 2.

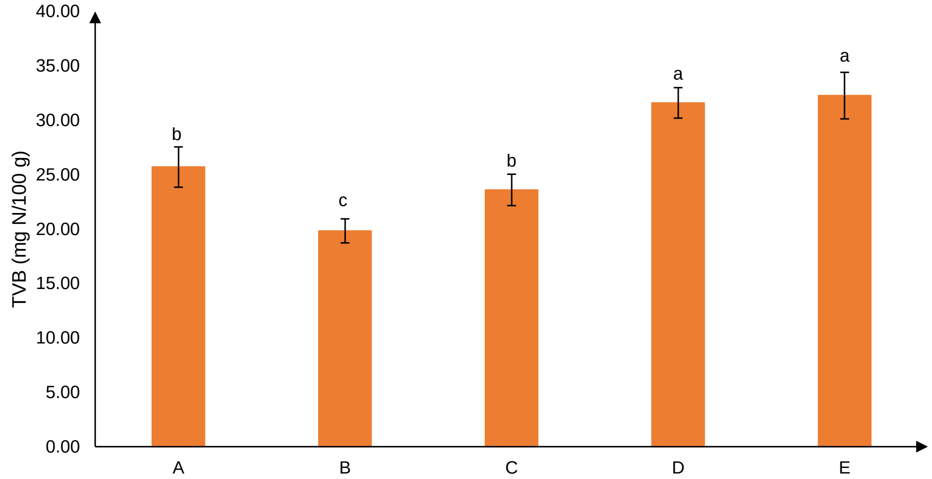

Fig. 2. Total volatile bases of *julung julung* smoked fillet

Fig. 2 shows the analysis of the variance of smoked fish fillets with different smoking method treatments affecting changes in TVB content ($p<0.05$). The TVB content of smoked fish fillets ranged from 19.83 – 32.27 mg N/100g, with the lowest TVB value in treatment B. The treatment of the smoking method with liquid smoke from corn cob waste (treatments A, B, and C) had lower TVB levels when compared to the conventional smoking method (treatments D and E). A literature search has not found why liquid smoking has a lower TVB value than conventional smoking methods. However, one possibility is that the liquid smoke smoking method allows the smoke components to be absorbed into the fish meat better than the conventional smoking method. Better absorption of liquid smoke phenol components in fish meat can inhibit and control microbial growth that causes a decrease in the quality of smoked fish (Santoso *et al*., 2015). A literature study showed that the average TVB value of smoked mackerel (*Euthynnus affinis*) treated with liquid smoke addition ranged from 24.63 – 28.38 mg N/100g (Hardianto & Yunianta, 2015). Another study reported smoked mackerel with a smoking process using rubber wood liquid smoke, producing smoked fish products with lower TVB values (Suroso *et al*., 2018).
The statistical analysis showed a difference between treatments B, A – C, and D – E on the TVB value of smoked fish fillets. The analysis of variance indicates that the drying process of smoked fish fillets before immersion into liquid smoke can maintain the quality of smoked fish (Kaparang *et al*., 2013). The statistical analysis also showed that treatments A – C and D – E had no difference in TVB value, indicating that the moisture content between the two treatments was not different. TVB value is closely related to moisture content (Rasulu *et al*., 2020).

The European standard (European Market Observatory for Fisheries and Aquaculture, 2020) states that the limit range of TVB value for smoked fish is 25 – 35 mg N/100 g. All treatments' TVB value of the smoked fish fillets still met the European standard. Previous studies reported TVB values of fresh fish fillets of 8.70 ± 0.86 mgN/100g (Moosavi-Nasab *et al*., 2021) and 12.94 ± 0.92 mg N/100g (Bouzgarrou *et al*., 2020). Messina *et al*. (2021) reported that smoked fish fillets that underwent two drying processes had a TVB value of <20 mg N/100g. Another study also reported the TVB value of smoked fish fillet samples with hot smoking and cold smoking, which amounted to 17.80 ± 0.17 and 18.95 ± 0.20 mg N/100g, respectively (El-Lahamy *et al*., 2019). Aydin *et al*. (2020) reported a TVB value of smoked fish with hot smoking of 11.82 ± 0.05 mg N/100g.

Moisture content

Moisture content is an important parameter to determine the quality of smoked fish produced. The moisture content contained in smoked fish can affect the shelf life of smoked fish because water in food is a medium for microbes to grow (Kaban *et al*., 2019). The smoking process was reported to reduce the moisture content of fresh fish to a range between 9 and 17% (Sikoki & Aminigo, 2002). The moisture content of smoked fish fillets is presented in Fig. 3.

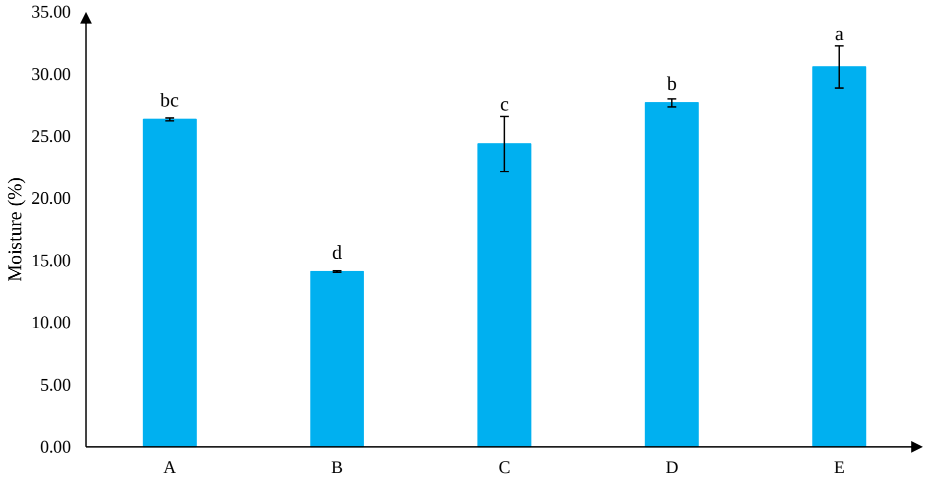

Fig. 3. Moisture content of *julung julung* smoked fillet

Fig. 3 shows the analysis of the variance of smoked *julung julung* fillets with different smoking methods treatment affects moisture content ($p<0.05$). The moisture content of smoked *julung julung* fillets ranged from 14.10 – 30.57%, with the lowest moisture content in treatment B. The moisture content

value in smoked fish products from all treatments still meets the Indonesian National Standard No. 2725:2013 on smoked fish, which is a maximum of 60% (Indonesia Standardization Agency, 2013). Darianto *et al*. (2018) reported that the smoking process can reduce the moisture content of fish to below 40%, which can help preserve it longer. The treatment of the smoking method with liquid smoke with corn cob (Treatment A, B, C) has a lower moisture content when compared to the treatment of the conventional smoking method (Treatment D and E). This result is because the smoking chamber is not fully enclosed in the conventional smoking method, so the heat generated could be more optimal. Suboptimal heat can increase moisture content and cause the moisture content of smoked fish to decrease only slightly (Amos & Paulina, 2017). Whereas in the liquid smoke smoking method, temperature and humidity can be controlled better so that the moisture content of the product can be reduced efficiently (Salindeho & Lumoindong, 2017).

The statistical analysis showed a difference between treatments B, A – C, and D – E on the TVB value of smoked fish fillets showed a difference between treatment B, treatment A – C, and treatment D – E on the moisture content of smoked fish fillets. The results of the variance study indicate that the drying process of smoked fish fillets before the dip in liquid smoke in treatment B can help reduce the moisture content. This study's results are from previous research, which also reported a significant decrease in moisture content in smoked fish products with two times drying treatment (Messina *et al*., 2021). The statistical analysis also showed that treatments A and C were not different because steaming in treatment C will increase the moisture content of the product (Salmatia *et al*., 2020). The conventional smoking method with corn cob fuel has the highest moisture content; this result is the conventional smoking process with corn cobs, and the heat generated is lower than smoking using wood (Asmara *et al*., 2022).

Water activity (Aw)

Water activity is one of the essential parameters in the quality of smoked fish. Water activity (Aw) is expressed as the ratio of the vapor pressure in the food to the vapor pressure of pure water, and it predicts whether water tends to move from the food product into the cells of microorganisms that may be present. A well-smoked fish has a water activity of <0.50 and a moisture content between 15 and 25% to inhibit the growth of pathogenic microorganisms in smoked fish products (Mondo *et al*., 2020). According to British Columbia Centre for Disease Control (2013), the maximum water activity value for smoked fish is 0.97. Water activity correlates with the moisture content of a smoked fish product and is two important factors affecting food safety and quality (Fitri *et al*., 2022). The water activity values of smoked fish fillets can be seen in Fig. 4.

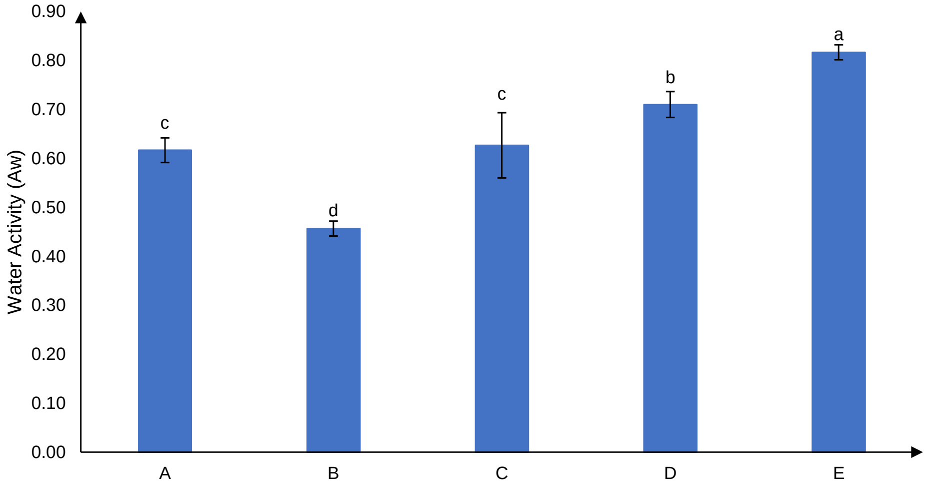

Fig. 4. Water activity of *julung julung* smoked fillet

Fig. 4 shows the analysis of the variance of smoked fish fillet *julung julung* with different smoking method treatments affecting water activity ($p<0.05$). The water activity value of smoked fish fillet ranged from 0.46 – 0.82, with the lowest water activity in treatment B. The treatment of the smoking method with corn cob liquid smoke (Treatment A, B, C) had a lower water content when compared to the treatment of the conventional smoking method (treatment D and E).

The water activity value of fish meat can vary depending on the cooking method, temperature, and duration of heating (Oliveira *et al*., 2017; Zhang *et al*., 2023). Therefore, the factor of fish fillet treatment before immersion in liquid smoke caused the difference in water activity value in treatment B compared to treatments A and C. The water activity value is directly proportional to the water content value of each treatment. The heating process can reduce the moisture content of fish meat (Kiczorowska *et al*., 2019), thus affecting the water activity of fish meat (Gómez *et al*., 2020).

pH

The pH value of smoked fish is an essential factor affecting its quality. The pH value will decrease with increasing smoking time (Baten *et al*., 2020b). The analysis of the variance of smoked fish fillets *julung julung* with different smoking method treatments affects the pH value ($p<0.05$). The pH of smoked fish fillet ranged from 4.60 to 5.81 (Fig. 5). The treatment of the smoking method with corn cob liquid smoke (treatments A, B, and C) had a lower pH value when compared to the conventional smoking method (treatments D and E). Swastawati *et al*. (2022) also reported that the pH value of smoked barracuda fish with the liquid smoke method was significantly lower than that of the conventional

method. Another study also reported that using liquid smoke can reduce pH caused by the condensation of organic acids in the smoking process (Puke & Galoburda, 2020). The pH value of smoked fish correlates with the phenol content contained in the smoke component, and an increase in phenol content causes a decrease in pH (Berhimpon *et al*., 2018).

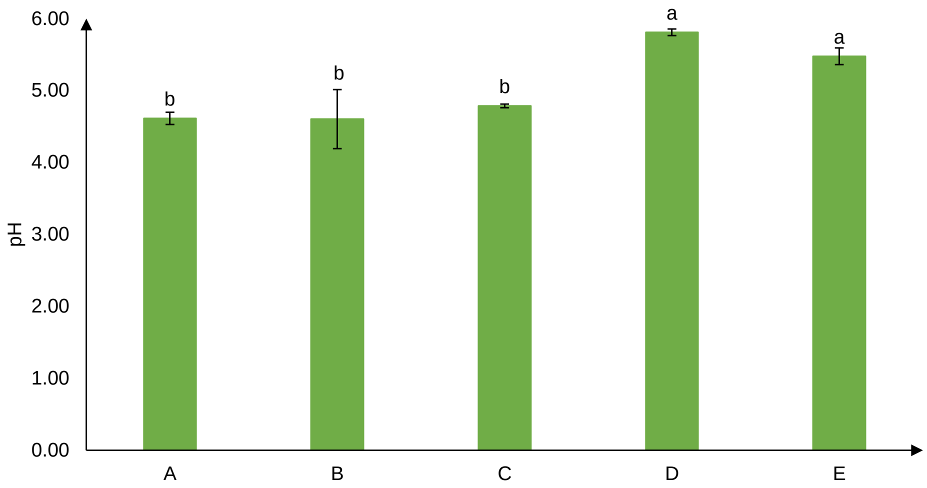

Fig. 5. pH of *julung julung* smoked fillet

Phenol level

Phenol is a compound found in wood smoke. Phenol compounds enter food through diffusion and capillary action, affecting taste, color, and aroma and extending shelf life (Remy *et al*., 2016). In addition, phenol compounds are also reported to act as antioxidants that can prevent rancidity in fish meat (Sérot *et al*., 2004). Leksono *et al*. (2020) also noted that the higher the phenol level in smoke, the stronger the flavor and aroma of smoked fish. The analysis of the variance of smoked fish fillets julung julung with different smoking method treatments affecting phenol level ($p<0.05$). The phenol level of smoked fish fillet ranged from 4.42 to 16.11 mg/g (Fig. 6). The treatment of the smoking method with corn cob liquid smoke (treatments A, B, and C) had higher phenol level when compared to the conventional smoking method (treatments D and E).

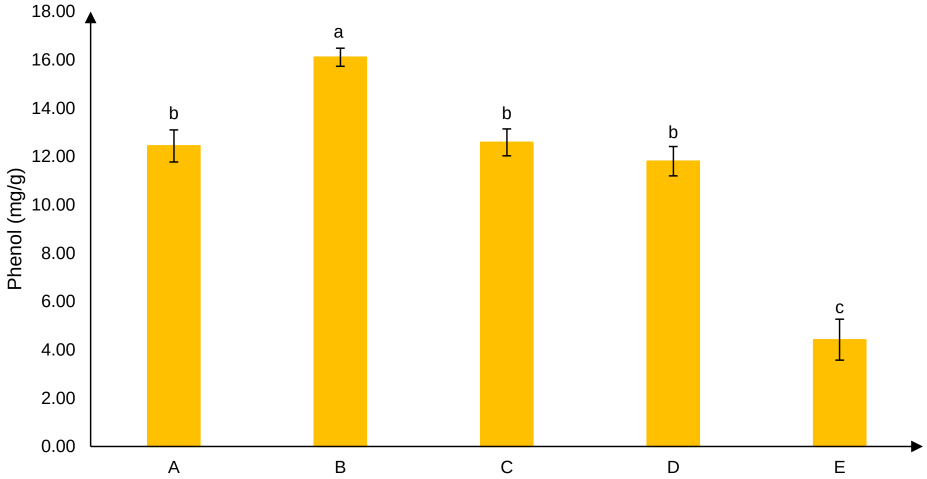

Fig. 6. Phenol level of *julung julung* smoked fillet

The statistical analysis showed a difference between treatments B, A – C, and D – E on the TVB value of smoked fish fillets showed phenol levels in treatments A, C, and D were not different. Previous research reported that the drying treatment of fish meat can increase phenolic compounds in smoked fish products (Sérot *et al*., 2004). The high phenol content in treatment B is thought to be because the pre-heated process causes the fish meat to lose water so that when dipped in liquid smoke, the fish fillets absorb a large amount of liquid smoke. Previous studies have reported that when the fish surface is dried, there is less smoke condensation than products smoked at lower temperatures. The results of this study indicate that a dry fish surface allows for better absorption of smoke components (Belichovska *et al*., 2019).

The study reported phenol levels in liquid smoke from corn cob waste of 0.335 mg/g (Swastawati *et al*., 2007) and 2.55% (Leviyani *et al*., 2019). The phenol level of smoked fish with the conventional method of smoking with corn cobs is lower due to the high acid content, which can potentially reduce the phenol content of smoked fish (Swastawati *et al*., 2012). Anggraini & Nurhazisa (2017) reported that the phenol content of liquid smoke from coconut shells was 3.04%, while liquid smoke from corn cob was 1.38%.

Polycyclic aromatic hydrocarbon (PAH) levels

Polycyclic aromatic hydrocarbons are a group of organic compounds formed by incomplete combustion, such as wood. Therefore, the type of fuel used in the fish smoking process affects the PAH content of smoked fish (Jinadasa *et al*., 2020). One of the carcinogenic PAHs, benzo(a)pyrene, is a carcinogenic

marker in smoked fish products (Stołyhwo & Sikorski, 2005). Polycyclic aromatic hydrocarbon (PAH) levels in smoked fish fillets can be seen in Table 2.

Table 2. Polycyclic aromatic hydrocarbon levels in *julung julung* smoked fillet

Polycyclic Aromatic Hydrocarbon	Smoked *Julung Julung* fish (µg/kg)					Corn Cob Liquid Smoke 0.8%(µg/kg)
	A	B	C	D	E	
Benzo(a)pyrene	nd	nd	nd	1.5	0.2	0.5
Benzo(b)fluoranthene	1.2	1.0	1.2	2	1.8	2.5
Benzo(a)anthracene	nd	nd	nd	0.5	nd	0.2
Benzo(g)perylene	0.4	0.2	0.4	1.0	0.8	1.0

nd = not detected

Table 2 shows that the PAH levels in smoked fish are still below the standard required in the Indonesian National Standard No. 2725 of 2013 on smoked fish, which is benzo(a)pyrene max 5 µg/kg (Indonesia Standardization Agency, 2013). The polycyclic aromatic hydrocarbon compound regulated in the Indonesian National Standard is only benzo(a)pyrene. Stołyhwo & Sikorski (2005) reported benzo(a)pyrene levels in smoked fish with hot and cold smoking processes ranging from 0.05 to about 60 µg/kg. Other studies have reported benzo(b,k)fluoranthene levels in salmon and rainbow trout ranging from 1.83 to 9.55 µg/kg, while benzo(g,h)perylene levels in salmon were 0.44 µg/kg (Basak *et al*., 2010). Berhimpon *et al*. (2018) also reported benzo(a)pyrene levels in smoked skipjack fish of 0.25 µg/kg. The level of benzo(a)pyrene in smoked barracuda was reported to be 0.32 µg/kg, benzo(b)fluoranthene 0.35 µg/kg, benzo(k)fluoranthene 0.21 µg/kg, benzo(a)anthracene 0.44 µg/kg, and benzo(g,h)perylene 2.56 µg/kg (Asamoah *et al*., 2021).

High PAH levels in smoked fish are due to the lignin content of the material used as fuel in the smoking process (Asamoah *et al*., 2021). The lignin content in corn cobs is reported to be about 17 – 21% (Thangavelu *et al*., 2018; Olajuyigbe *et al*., 2019; Gandam *et al*., 2022). The lignin content of coconut shell was approximately 59.5% (Wang & Sarkar, 2018), while that of coconut wood was about 50% on a dry weight basis (Anuchi *et al*., 2022). Lignin is reported to absorb PAH compounds because PAH compounds can bind well with lignin (Oliveira *et al*., 2019). During pyrolysis and gasification processes, lignin components react to produce aromatic tar and coke, which can form PAH compounds (Zhou *et al*., 2014; Kawamoto, 2017). Therefore, PAH compounds in smoked fish smoked using corncobs are lower than those smoked with shells and coconut wood.

Sensory assessment

A sensory assessment is carried out to evaluate the panelist's preference level, including appearance, aroma, taste, and texture. A sensory assessment must determine the quality of the smoked fish product and ensure it meets the applicable standards (Hadanu & Lomo, 2019). The sensory characteristics of

smoked fish products affect consumer acceptance and preference. Sensory assessment by panelists helps to identify the sensory attributes that are most appealing to consumers, allowing manufacturers to customize products according to consumer assessment (Ekelemu *et al*., 2021). Indonesian National Standard No. 2725:2013 regarding smoked fish requires a minimum sensory assessment of 7 (Score 1-9) (Indonesia Standardization Agency, 2013). The results of the panelists' assessment of *julung julung* smoked fillet from each treatment can be seen in Fig. 7.

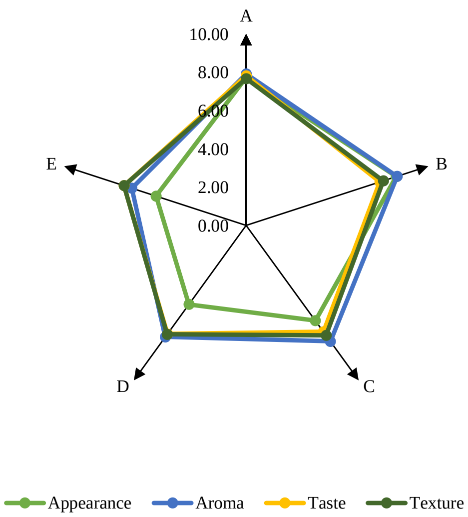

Fig. 7. Sensory assesment of *julung julung* smoked fillet

Fig. 7 shows the analysis of the variance of the sensory assessment of the appearance of smoked *julung julung* fillets with different smoking method treatments affecting the appearance of smoked fish ($p<0.05$). The panelists' assessment of the appearance of smoked fish fillets ranged from 4.95 to 8.24, with the highest panelists' assessment in treatment B. Based on the requirements of the Indonesian National Standard, only treatments A and B met the minimum panelist assessment requirement of 7. The moisture content factor is thought to have influenced the panelists' assessment of the appearance of smoked fish, so panelists less favored treatment C with steaming. Moisture content can affect the physical properties of fish, such as the appearance and texture of smoked fish (Baten *et al*., 2020a). Smoked fish with high moisture content will make the color of smoked fish look paler (Flick, 2010). Treatments D and E were less favored by panelists, presumably because conventional smoking methods produce darker products (dark brown), less bright and less shiny. Smoking fish with liquid smoke has smoked fish products with a golden color and clean and shiny surface (Berhimpon *et al*., 2018). Another

factor that affects the appearance of smoked fish is the length of the smoking process. The longer the smoking process, the darker the color will be. The time of the smoking process for smoked fish using the liquid smoke method is shorter than the conventional method (Puke & Galoburda, 2020; Baten *et al*., 2020b). The smoking process can change the color of fish, giving it a distinctive golden color due to the interaction of carbonyls with amino components on the surface of the meat. The color and appearance of smoked fish also positively correlate with phenol content. Phenol compounds in smoke interact with amino acid components in fish meat, producing a distinctive golden color in smoked fish (Montazeri *et al*., 2013).

Fig. 7 shows the analysis of the variance of the sensory assessment of the aroma of smoked fish fillets *julung julung* with different smoking method treatments that did not affect the appearance of smoked fish ($p>0.05$). However, the aroma sensory assessment of treatments A, B, C, and D still meets the minimum assessment of the Indonesian National Standard on smoked fish. The sensory assessment of smoked fish aroma was lowest in treatment E. Previous studies have reported that smoking fish with corn cobs as a fuel source resulted in the lowest aroma scores, indicating less aromatic smoked fish (Asmara *et al*., 2022). The aroma and taste of smoked fish are strongly influenced by the time of smoking (Tahir *et al*., 2020). This assessment indicates that the time of the smoking process of 4 and 6 hours in all treatments has not influenced the taste and aroma of smoked fish.

Fig. 7 shows the analysis of the variance of the sensory assessment of the taste of smoked fish fillets *julung julung* with different smoking method treatments that did not affect the appearance of smoked fish ($p>0.05$). The taste of smoked fish is influenced by the time of the smoking process. Previous studies have reported that longer smoking time can improve the sensory attributes of taste (Baten *et al*., 2020b). This result indicates that the 2-hour time difference between the liquid smoke smoking treatment and the conventional smoking method has not affected the taste of smoked fish. A distinctive smoked taste without bitterness is a criterion for assessing the taste of good quality smoked fish (Sukowati *et al*., 2021).

Fig. 7 shows the analysis of variance of the sensory assessment of the texture of smoked fish fillet *julung julung* with different smoking method treatments giving effect to the texture of smoked fish ($p<0.05$). Panelists' assessment of the texture of smoked fish *julung julung*, ranged from 6.71 – 7.67, with the highest panelist assessment in treatments A and B. The statistical analysis showed that treatments A and B differed from treatments C, D, and E. This result was thought to be because the fish fillets were dipped in liquid smoke, giving a dry, compact, and tender texture. Whereas in treatment C (fish fillets subjected to steaming), the texture of the smoked fillets was rather sticky and not solid. Treatments D and E produced the texture of smoked fish fillets which were less dense and not compact. The texture of smoked fish is negatively correlated with its moisture content. The higher the moisture content in

smoked fish, the softer and less firm the texture (Chan *et al*., 2022). High moisture content in smoked fish products results in less dense fish muscle fibers (Chang *et al*., 2021).

A good texture of smoked fish is compact, moderately elastic, and not sticky (Sulistijowati *et al*., 2021). The Indonesian National Standard requires that the texture of smoked fish is dense and compact and the tissue is very tight (Indonesia Standardization Agency, 2013). Based on consumer surveys, the preferred texture of smoked fish is dense, slightly wetter, and easy to chew, and there is a sensation of liquid in the mouth when chewed (Ticoalu *et al*., 2019).

Conclusion

Based on the evaluation of the parameters of total volatile bases, moisture content, water activity, pH, phenol content, polycyclic aromatic hydrocarbon content, and sensory assessment of smoked *julung julung* fillets, it can be concluded that treatment B is the best treatment, i.e. the fillets were preheated for 4 hours at 60 – 80 °C, then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated for 4 hours at 90 °C. In general, the characteristics of smoked fillets using liquid smoke were better when compared to the conventional smoking treatment. It is necessary to evaluate different smoking times on smoked *julung julung* fillets with corn cob liquid smoke method.

Acknowledgment

Author contributions

Conflicts of interest

The authors declare that there is no conflict of interest.

Highlights

Effectiveness of using liquid smoke derived from corn cob waste in the smoking process of *julung julung* fillets

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6. Bukti respons kepada reviewer terkait revisi manuskrip

Characteristics of *Julung Julung* Smoked Fillets (*Hemiramphus* sp.) using Liquid Smoke from Corn Cobs Waste

Abstract

This study aimed to obtain the best smoking method for *julung julung* fillets (*Hemiramphus* sp.) using liquid smoke from corn cob waste. This study used three different fillet treatment methods (dried and steamed) before liquid smoke application. This study also compared fish smoking between using liquid smoke with conventional methods (using shell, coconut wood, and corn cob). The parameters to determine the quality of smoked fish were Total Volatile Bases (TVB), moisture, water activity, pH, phenol levels, polycyclic aromatic hydrocarbon (PAH), and sensory assessment. The results showed that the TVB of smoked *julung julung* fillets ranged from 19.83 – 32.27 mg N/100g. The moisture ranged from 14.10 – 30.57%. Water activity ranged from 0.46 – 0.82. pH ranged from 4.60 – 5.81. Phenol levels ranged from 4.42 – 16.11 mg/g. PAH levels are still below the standard required in the Indonesian National Standard. Panelists rated neutral to really like the appearance, aroma, taste, and texture of smoked fish. From these research, it can be concluded that treatment B is the best treatment, namely fresh fillets are first heated for 4 hours at 60 – 80 ºC and then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated for 4 hours at 90 ºC.

Keywords: Corn cobs, Indonesia, Liquid smoke, North Sulawesi, Wood

Introduction

Corn is the second most widely grown crop in Indonesia after rice. Indonesia's maize production in 2023 is 14.46 million tons, ranking 9th in the world. (Indonesia Center of Statistic Agency, 2023). North Sulawesi is one of the contributing regions that produce corn in Indonesia. Considerable corn production will also produce waste, especially corn cobs, which are generally thrown away and burned by most Indonesians (Cahyadi *et al*., 2021).

One of the corn cobs was used to make liquid smoke. Previous research reported that corn cobs can produce liquid smoke, a by-product of the pyrolysis of corn cob waste. The yield of liquid smoke from corn cobs is about 28.37%, with a pH value of 3.5 (Sriharti *et al*., 2020). The particle size of corn cobs affects the yield of liquid smoke, with smaller particle sizes resulting in higher yields (Aladin *et al*., 2018). Swastawati et al. (2007) reported the phenolic content in liquid smoke produced from 335 mg/L corn cobs.

Liquid smoke is a natural product made from the condensation of smoke from burning wood (Andy *et al*., 2021). Liquid smoke is commonly used as a flavoring in food to provide a smoked flavor without the food undergoing the actual smoking process (Sari *et al*., 2006). Using liquid smoke in food will save time, energy, and labor, so that can reduce production costs (Krah *et al*., 2019). In addition, using liquid smoke in food can speed up and standardize the smoking process, adding flavor and microbiological safety while reducing production time (Indiarto *et al*., 2020). Generally, various types of hardwood were used to manufacture liquid smoke (Diatmika *et al*., 2019). The quality of the liquid smoke produced

may vary depending on the type of wood used (Budaraga *et al*., 2016). In addition to imparting specific flavors and aromas to foods (Maulina *et al*., 2020), liquid smoke is reported to be antimicrobial (Septana *et al*., 2020) and antioxidant (Budaraga & Putra, 2021).

The conventional smoking processes generally use hardwood as fuel, such as melon wood (Umar *et al*., 2018), mangrove wood (Cissoko *et al*., 2020), teak wood (Daramola *et al*., 2020), coconut shells, and coconut husks (Nugroho *et al*., 2018). Corn cobs have also been reported to be used in the fish smoking process (Sukowati, 2023). According to literature, traditional fish smoking in North Sulawesi, Indonesia, uses smoke from burning coconut shells or wood charcoal (Landangkasiang *et al*., 2017; Primalasari *et al*., 2019; Saediman *et al*., 2021). Smoked fish processing has been known for a long time because of its traditional processing techniques, simplicity, ease of implementation, and low cost (Islamiyah, 2021). In North Sulawesi, Indonesia, smoked fish commonly known as *fufu* is traditionally made from skipjack (*Katsuwonus pelamis*); *julung julung* (*Hemiramphus far*) (Berhimpon *et al*., 2018); and scad fish (*Decapterus* spp.) known as *pinekuhe* (Ansar & Ijong, 2021). The smoked fish product of *julung julung* is a typical product from North Sulawesi and Gorontalo, commonly called *roa* or *galavea* (Azis & Akolo, 2020).

The study's reported that the conventional smoking process has disadvantages such as smoking time, concentration of carcinogenic substances, temperature, and inconsistent product quality (Racovita *et al*., 2020). Conventional smoking of food products has been shown to produce carcinogenic components such as polycyclic aromatic hydrocarbons (Jinadasa *et al*., 2020). Benzo(a)pyrene ($C_{20}H_{12}$) is one of the carcinogenic compounds produced from traditional smoking products (Jinadasa *et al*., 2020). One method to reduce carcinogenic compounds in smoked fish products is to use liquid smoke in the smoking process (Nithin *et al*., 2020; Xin *et al*., 2021).

The utilization of liquid smoke in the fish smoking industry in North Sulawesi is very likely to be developed. North Sulawesi was reported to have both large-scale and domestic fish-smoking industries (Dotulong *et al*., 2018; Primalasari *et al*., 2019). Making liquid smoke is relatively simple, so it will be readily accepted by fish-smoking industry players (Ali & Al Fiqri, 2020). Previous research reported that using liquid smoke in smoked fish products resulted in good quality in terms of appearance, flavor, and aroma (Rizal *et al*., 2020). In addition, using liquid smoke in smoked fish products can add nutritional value and durability, making it possible to reach a wider market area (Ali *et al*., 2021).

Previous research reported that smoked fish dipped in liquid smoke for 15 minutes had a total plate count value of 4.7×10^4 CFU/g on day six and moisture content below 60% during storage (Suroso *et al*., 2018). Dipping fish in liquid smoke with a concentration of 15% and a dipping time of 60 minutes showed a bacterial colony of 2.12×10^2 CFU/g (Ali *et al*., 2021). The Indonesian National Standard on smoked fish requires a maximum total plate count of 5×10^4 CFU/g and a maximum moisture content of 60% (Indonesia Standardization Agency, 2013). Another study reported that smoked fish processed

with liquid smoke had a moisture content of 47.63%, phenol content of 12.62%, and pH of 4.8 (Berhimpon *et al*., 2018). The results of these studies indicate that liquid smoke is good to apply to smoked fish products. This study characterized smoked fish fillets of *julung julung* (*Hemiramphus* sp.) produced with liquid smoke from corn cobs waste. The study was conducted by dipping the fish fillets of *julung julung* with liquid smoke, with drying and steaming treatments on the fish fillets. This study aims to obtain the best smoking method for fish fillets using liquid smoke from corn cobs waste based on the assessment of total volatile bases, moisture content, water activity, pH value, phenol content, polycyclic aromatic hydrocarbon content, and sensory assessment.

Materials and methods

The main materials of the study were *julung julung* fish (*Hemiramphus* sp.), corn cob (*Zea mays* L.) from Gangga Island in Likupang, North Sulawesi, Indonesia. This study includes the preparation of liquid smoke from corn cobs waste, preparing fish (weeding and filleting), and treating fish fillets by dip in liquid smoke. Fresh *julung julung* fish was obtained from traditional fishermen in Manado, North Sulawesi, Indonesia (Fig. 1). The fish was placed in a cool box and given ice in a ratio of 1:1 (ice:fish) (Mishra *et al*., 2021). Then, the fish was washed, cleaned, and filleted. The cleaned fillets were dipped in a 5% salt solution for 30 minutes. The concentration of liquid smoke from corn cob waste was 0.8% (Berhimpon *et al*., 2018).

Commented [WT1]: Because based on the results of Berhimpon *et al*., 2018, the best concentration of liquid acid from corn cobs for fish smoking is at a concentration of 0.8%.

Fig. 1. *Julung julung* (*Hemiramphus* sp.)

The treatment in this study was applying liquid smoke from corn cobs waste to the smoking process of *julung julung* fillets, which was compared with the conventional method (using coconut wood, coconut shells, and corn cobs).

(A) : Fresh fillets were dipped in liquid smoke for 20 minutes and then dried in a cabinet dryer for 4 hours at 90 °C.

(B) : Fresh fillets were preheated in a cabinet dryer for 4 hours at 60 – 80 °C, then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated in a cabinet dryer for 4 hours at 90 °C.

(C) : Fresh fillets were steamed for 30 minutes, then dipped in liquid smoke for 20 minutes. After that, the fillets were dried in a cabinet dryer for 4 hours at 90 °C.

(D) : Fresh fillets were smoked with the conventional method using wood and coconut shell for 6 hours at 90 ºC.

(E) : Fresh fillets were smoked with the conventional method using corncob for 6 hours at 90 ºC.

Liquid smoke manufacturing process

Making liquid smoke refers to modified research (Aladin *et al*., 2018). Modifications were made to the equipment used in the pyrolysis process. Dry corn cobs were weighed to determine fuel efficiency. Corn cobs are gradually burned into the combustion furnace, at 400 ºC and have a pyrolysis time of about 120 minutes. The furnace was closed to prevent smoke from escaping from the tank. The smoke generated from the combustion flows through a pipe connected to a storage tank covered with ice cubes. The smoke that passes through the pipe will become cold, so condensation occurs, turning the smoke into liquid. The smoke that has been formed is collected into a container attached to the end of the pipe. The liquid smoke obtained is then allowed to settle the tar formed for three weeks and filtered (using Whatman filter paper No. 10) to obtain clear liquid smoke.

Total volatile bases assay

Total Volatile Base (TVB) assay refers to research by Tambunan & Chamidah (2021). TVB assay aims to determine the amount of volatile base compounds formed from protein breakdown—the principle of TVB analysis, namely by evaporating volatile base compounds at room temperature for 24 hours. The compound is then bound with boric acid and titrated with an HCl solution. After the boric acid solution was stored in the inner chamber of the Conway cell containing the blank (V_0) and the filtrate (V_1), the boric acid solution was titrated with 0.02 N HCl so that the boric acid solution changed color to pink. TVB levels in the smoked fish meat were expressed as mg N/100g.

$$TVB\left(\mathrm{mg}\frac{\mathrm{N}}{100\mathrm{g}}\right) = \frac{(V_1 - V_0) \times N.HCl \times dilution \times 100}{sample\ weight\ (g)}$$

Commented [WT2]: The treatment in this study was based on the results of Berhimpon et al. (2018) with some modifications, namely smoking fish without liquid smoke (using wood, head, coconut shell, and corn cobs).

Commented [WT3]: Based Aladin *et al*., 2018

Commented [WT4]: charcoal formed from the pyrolysis process

Commented [WT5]: Based Aladin *et al*., 2018

Commented [WT6]: The level of benzopyrene from liquid smoke was evaluated by HPLC. And the benzopyrene in liquid smoke was about 0.5 µg/kg.

Commented [WT7]: The TVB testing method is based on the instructions of Tambunan and Chamidah (2021). We still include the complete work procedure, so that readers will get complete information.

Moisture content assay

The moisture content is determined by the Indonesia National Standard (Indonesia Standardization Agency, 2006a). An empty porcelain cup is weighed (A). Then, the sample was weighed to ± 2 g in a porcelain cup of known mass (B). The samples were dried in an oven at 105°C for 20 hours. The samples were then cooled in a desiccator, after which the samples in the cup were weighed (C). Moisture content was expressed as %.

$$Moisture\ (\%) = \frac{B\ (g) - C\ (g)}{B(g) - A\ (g)} \times 100\%$$

Water activity (Aw) assay

Water activity assay used an Aw meter (Rotronic, HygroPalm 23-AW-A). The Aw meter was set up at room temperature for 2 hours. The water activity of smoked fish was measured by placing the sample into a sample container and conditioning it for 30-60 minutes. The Aw meter sensor is contacted with the sample in the container. Then the water activity (Aw) value can be read on the Aw meter panel (Saputra *et al*., 2014).

pH assay

The sample weighed as much as 30 g, then 400 mL of distilled water was added at 28 ºC. The sample was homogenized with a magnetic stirrer to be evenly stirred. The pH meter electrode was inserted into the sample solution. The data taken in this test is the pH value read by the pH meter (Hanna Instruments HI99192) (Lekahena & Jamin, 2018).

Phenol level assay

Samples were extracted by adding 1 mL ethanol p.a., 5 mL distilled water, and 0.5 mL Folin Ciocalteu 50%, homogenized, and allowed to stand for 5 minutes. Then, 1 mL of 5% Na_2CO_3 and left in the dark for ± 60 minutes with gallic acid as a standard. The absorbance value was read on a spectrophotometer (Thermo Scientific Genesys 50) with a wavelength of 725 nm. Total phenolics were expressed as mg/g (Indonesia Standardization Agency, 2004; Muliadin *et al*., 2022).

Commented [WT8]: Yes,using gallic acid as standar curve

$$Total\ Phenol\ (\frac{mg}{g}) = \frac{Gallic\ acid\ equivalence\ \left(\frac{mg}{L}\right) \times Volume\ (L)}{Sample\ weight\ (g)}$$

Polycyclic aromatic hydrocarbon (PAH) assay

PAH content testing was carried out based on the instructions Basak *et al*. (2010). A 5 g sample was weighed and collected in a 100 ml flask. Then 5 mL of 50% KOH solution, 75 mL of methanol, 1 mL of internal standard solution (9,10 dimethylantracene), and some boiling stones were added. After that,

the mixture was boiled for 4 hours in a soxhelet. The liquid phase was transferred to a separatory funnel and extracted with 100 mL of n-hexane with shaking for 3 min. The MeOH:KOH phase was drained and discarded. The combined n-hexane phase was rinsed twice, each with 50 mL of H_2O-MeOH (8:1) and 50 mL of water. The methanol-water and water phases were dried and discarded. The organic phase was concentrated in a rotary evaporator (40°C) to a volume of about 10 mL. The concentrated extract was dried under nitrogen gas.

After that, the silica gel column was cleaned by dissolving 15 g of silica gel in n-hexane and transferred to a column (200 mm long; diameter: 12 mm). Then, 1 g of anhydrous sodium sulfate was added. The concentrated sample extract was transferred to the column and eluted with 1 mL of n-hexane. A stock solution containing 0.1 mg mL^{-1} of 9,10 dimethylantracene was dissolved in n-hexane and stored at 4°C in a volumetric flask (with a glass stopper) wrapped in aluminum foil to avoid possible light degradation. Working standard solutions were prepared from the stock solutions. Analysis of PAH compounds used a Hewlett-Packard 1100 HPLC equipped with an Agilent-110 fluorescence detector. The injection volume was 10 µL. Mobile phase gradient (acetonitrile-water): 80% acetonitrile + 20% water with a flow rate of 1 ml min^{-1} and a wavelength of 270 nm. The external standard mixture was used for PAH concentration calculation.

Sensory assessment assay

Sensory assessment assay refers to the Indonesia National Standard (Indonesia Standardization Agency, 2006b). The sensory assessment was based on a hedonic scale (Table 1). Smoked fish samples were placed on a plastic plate with a glass of water, coded, and presented to 30 randomly selected semi-trained panelists (university students who had studied sensory assessment techniques), and the assessment process was carried out under light. The parameters observed were appearance, aroma, taste, and texture. Panelists rated acceptance of the samples on a scale of 1 – 9.

Commented [WT9]: Without preparation,because the smoked fish samples used are still suitable for consumption (under 24 hours since smoking).

Table 1. Score sheet of hedonic assessment

Specification	Score
Really like it	9
Really like	8
Like	7
Rather like	6
Neutral	5
Rather dislike	4
do not like	3
Very dislike	2
Really don't like it	1

Statistical analysis

Statistical analysis was performed using Statistical Product and Service Solutions (SPSS) version 20.0. Statistical analysis of data was performed based on one-way analysis of variance (ANOVA) using a significance level of ($P<0.05$). Specific group differences were determined using Duncan's test. The TVB, total phenol, pH, moisture, water activity, and sensory assessment parameters were evaluated with three replicates.

Results and discussion

Liquid smoke production was carried out using a simple liquid smoke distillation device. This process consumed 17 kg of corn cobs and 12 kg of crushed ice. Liquid smoke from corn cobs is more accessible than coconut shells or wood (Maulina & Karo, 2021). Using coconut shells or wood as fuel will form a flame, thus reducing the volume of smoke in the furnace (Kabir Ahmad *et al*., 2022). The smoked *julung julung* were analyzed for TVB, moisture content, water activity, pH value, phenol content, PAH content, and sensory assessment.

Total volatile bases

The total volatile base (TVB) value is measured to determine the quality of the smoked fish. TVB value is one of the parameters used to determine the decline in fish quality and measures the amount of volatile base compounds formed due to protein degradation (Castro *et al*., 2006). Volatile bases formed in fish muscle tissue mainly consist of ammonia, trimethylamine (TMA), and dimethylamine (DMA) (VELP Scientifica, 2013). TVB value is an essential characteristic for the quality assessment of seafood products and is emerging as the most commonly used chemical parameter to assess the palatability of seafood (Rasulu *et al*., 2020). The results of *total volatile bases* on smoked fish fillets of *julung julung* are presented in Fig. 2.

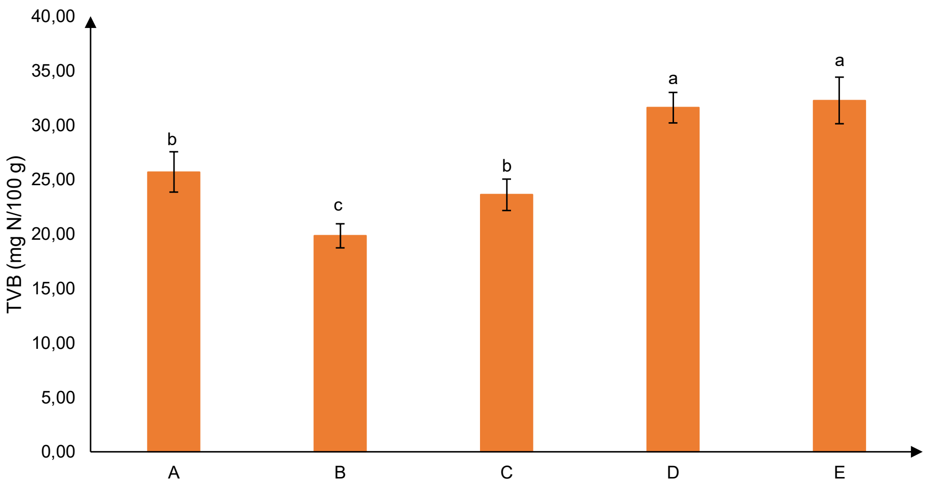

Fig. 2. Total volatile bases of *julung julung* smoked fillet

Fig. 2 shows the analysis of the variance of smoked fish fillets with different smoking method treatments affecting changes in TVB content ($p<0.05$). The TVB content of smoked fish fillets ranged from 19.83 – 32.27 mg N/100g, with the lowest TVB value in treatment B. The treatment of the smoking method with liquid smoke from corn cob waste (treatments A, B, and C) had lower TVB levels when compared to the conventional smoking method (treatments D and E). A literature search has not found why liquid smoking has a lower TVB value than conventional smoking methods. However, one possibility is that the liquid smoke smoking method allows the smoke components to be absorbed into the fish meat better than the conventional smoking method. Better absorption of liquid smoke phenol components in fish meat can inhibit and control microbial growth that causes a decrease in the quality of smoked fish (Santoso *et al*., 2015). A literature study showed that the average TVB value of smoked mackerel (*Euthynnus affinis*) treated with liquid smoke addition ranged from 24.63 – 28.38 mg N/100g (Hardianto & Yunianta, 2015). Another study reported smoked mackerel with a smoking process using rubber wood liquid smoke, producing smoked fish products with lower TVB values (Suroso *et al*., 2018). The statistical analysis showed a difference between treatments B, A – C, and D – E on the TVB value of smoked fish fillets. The analysis of variance indicates that the drying process of smoked fish fillets before immersion into liquid smoke can maintain the quality of smoked fish (Kaparang *et al*., 2013). The statistical analysis also showed that treatments A – C and D – E had no difference in TVB value, indicating that the moisture content between the two treatments was not different. TVB value is closely related to moisture content (Rasulu *et al*., 2020).

The European standard (European Market Observatory for Fisheries and Aquaculture, 2020) states that the limit range of TVB value for smoked fish is 25 – 35 mg N/100 g. All treatments' TVB value of the smoked fish fillets still met the European standard. Previous studies reported TVB values of fresh fish fillets of 8.70 ± 0.86 mgN/100g (Moosavi-Nasab *et al*., 2021) and 12.94 ± 0.92 mg N/100g (Bouzgarrou *et al*., 2020). Messina *et al*. (2021) reported that smoked fish fillets that underwent two drying processes had a TVB value of <20 mg N/100g. Another study also reported the TVB value of smoked fish fillet samples with hot smoking and cold smoking, which amounted to 17.80 ± 0.17 and 18.95 ± 0.20 mg N/100g, respectively (El-Lahamy *et al*., 2019). Aydin *et al*. (2020) reported a TVB value of smoked fish with hot smoking of 11.82 ± 0.05 mg N/100g.

Moisture content

Moisture content is an important parameter to determine the quality of smoked fish produced. The moisture content contained in smoked fish can affect the shelf life of smoked fish because water in food is a medium for microbes to grow (Kaban *et al*., 2019). The smoking process was reported to reduce the moisture content of fresh fish to a range between 9 and 17% (Sikoki & Aminigo, 2002). The moisture content of smoked fish fillets is presented in Fig. 3.

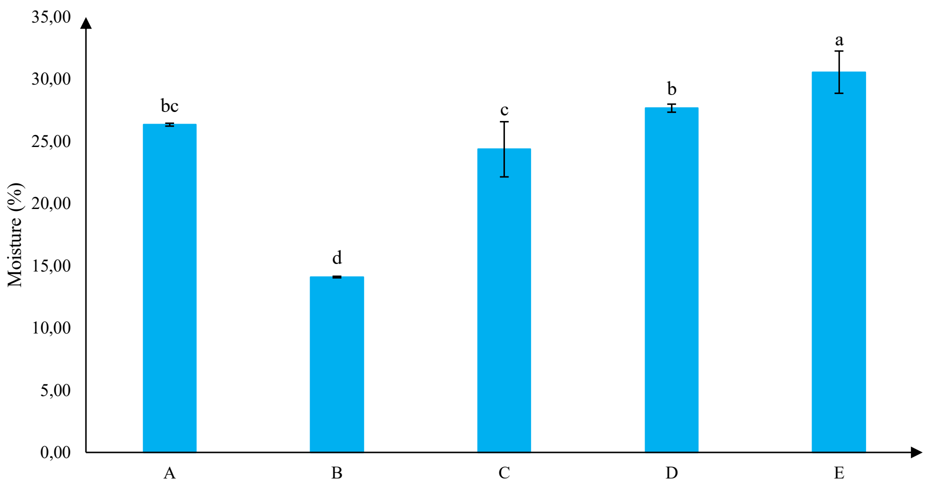

Fig. 3. Moisture content of *julung julung* smoked fillet

Fig. 3 shows the analysis of the variance of smoked *julung julung* fillets with different smoking methods treatment affects moisture content (p<0.05). The moisture content of smoked *julung julung* fillets ranged from 14.10 – 30.57%, with the lowest moisture content in treatment B. The moisture content

value in smoked fish products from all treatments still meets the Indonesian National Standard No. 2725:2013 on smoked fish, which is a maximum of 60% (Indonesia Standardization Agency, 2013). Darianto *et al*. (2018) reported that the smoking process can reduce the moisture content of fish to below 40%, which can help preserve it longer. The treatment of the smoking method with liquid smoke with corn cob (Treatment A, B, C) has a lower moisture content when compared to the treatment of the conventional smoking method (Treatment D and E). This result is because the smoking chamber is not fully enclosed in the conventional smoking method, so the heat generated could be more optimal. Suboptimal heat can increase moisture content and cause the moisture content of smoked fish to decrease only slightly (Amos & Paulina, 2017). Whereas in the liquid smoke smoking method, temperature and humidity can be controlled better so that the moisture content of the product can be reduced efficiently (Salindeho & Lumoindong, 2017).

The statistical analysis showed a difference between treatments B, A – C, and D – E on the TVB value of smoked fish fillets showed a difference between treatment B, treatment A – C, and treatment D – E on the moisture content of smoked fish fillets. The results of the variance study indicate that the drying process of smoked fish fillets before the dip in liquid smoke in treatment B can help reduce the moisture content. This study's results are from previous research, which also reported a significant decrease in moisture content in smoked fish products with two times drying treatment (Messina *et al*., 2021). The statistical analysis also showed that treatments A and C were not different because steaming in treatment C will increase the moisture content of the product (Salmatia *et al*., 2020). The conventional smoking method with corn cob fuel has the highest moisture content; this result is the conventional smoking process with corn cobs, and the heat generated is lower than smoking using wood (Asmara *et al*., 2022).

Water activity (Aw)

Water activity is one of the essential parameters in the quality of smoked fish. Water activity (Aw) is expressed as the ratio of the vapor pressure in the food to the vapor pressure of pure water, and it predicts whether water tends to move from the food product into the cells of microorganisms that may be present. A well-smoked fish has a water activity of <0.50 and a moisture content between 15 and 25% to inhibit the growth of pathogenic microorganisms in smoked fish products (Mondo *et al*., 2020). According to British Columbia Centre for Disease Control (2013), the maximum water activity value for smoked fish is 0.97. Water activity correlates with the moisture content of a smoked fish product and is two important factors affecting food safety and quality (Fitri *et al*., 2022). The water activity values of smoked fish fillets can be seen in Fig. 4.

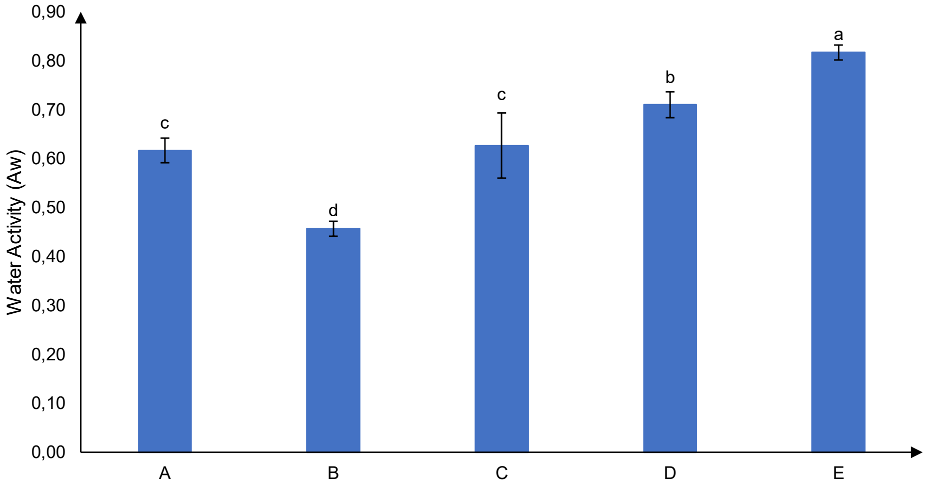

Fig. 4. Water activity of *julung julung* smoked fillet

Fig. 4 shows the analysis of the variance of smoked fish fillet *julung julung* with different smoking method treatments affecting water activity (p<0.05). The water activity value of smoked fish fillet ranged from 0.46 – 0.82, with the lowest water activity in treatment B. The treatment of the smoking method with corn cob liquid smoke (Treatment A, B, C) had a lower water content when compared to the treatment of the conventional smoking method (treatment D and E).

The water activity value of fish meat can vary depending on the cooking method, temperature, and duration of heating (Oliveira *et al*., 2017; Zhang *et al*., 2023). Therefore, the factor of fish fillet treatment before immersion in liquid smoke caused the difference in water activity value in treatment B compared to treatments A and C. The water activity value is directly proportional to the water content value of each treatment. The heating process can reduce the moisture content of fish meat (Kiczorowska *et al*., 2019), thus affecting the water activity of fish meat (Gómez *et al*., 2020).

pH

The pH value of smoked fish is an essential factor affecting its quality. The pH value will decrease with increasing smoking time (Baten *et al*., 2020b). The analysis of the variance of smoked fish fillets *julung julung* with different smoking method treatments affects the pH value (p<0.05). The pH of smoked fish fillet ranged from 4.60 to 5.81 (Fig. 5). The treatment of the smoking method with corn cob liquid smoke (treatments A, B, and C) had a lower pH value when compared to the conventional smoking method (treatments D and E). Swastawati *et al*. (2022) also reported that the pH value of smoked barracuda fish with the liquid smoke method was significantly lower than that of the conventional

method. Another study also reported that using liquid smoke can reduce pH caused by the condensation of organic acids in the smoking process (Puke & Galoburda, 2020). The pH value of smoked fish correlates with the phenol content contained in the smoke component, and an increase in phenol content causes a decrease in pH (Berhimpon *et al*., 2018).

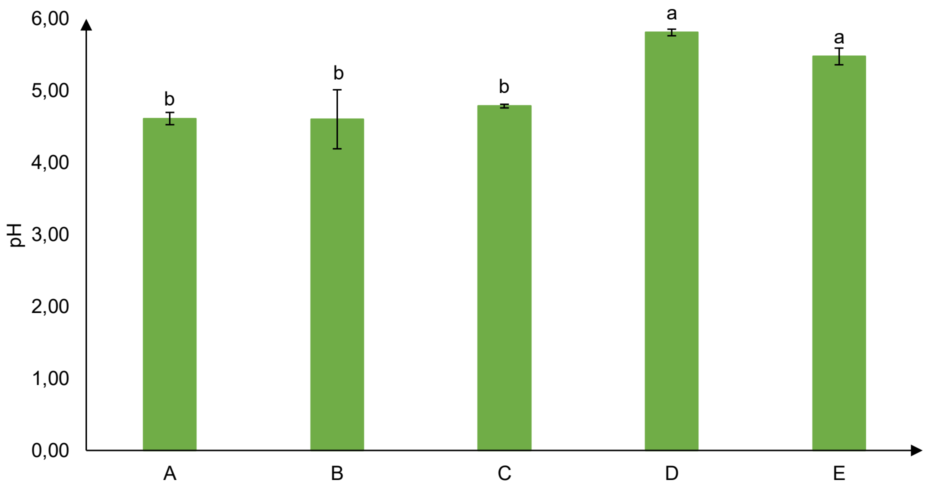

Fig. 5. pH of *julung julung* smoked fillet

Phenol level

Phenol is a compound found in wood smoke. Phenol compounds enter food through diffusion and capillary action, affecting taste, color, and aroma and extending shelf life (Remy *et al*., 2016). In addition, phenol compounds are also reported to act as antioxidants that can prevent rancidity in fish meat (Sérot *et al*., 2004). Leksono *et al*. (2020) also noted that the higher the phenol level in smoke, the stronger the flavor and aroma of smoked fish. The analysis of the variance of smoked fish fillets julung julung with different smoking method treatments affecting phenol level ($p<0.05$). The phenol level of smoked fish fillet ranged from 4.42 to 16.11 mg/g (Fig. 6). The treatment of the smoking method with corn cob liquid smoke (treatments A, B, and C) had higher phenol level when compared to the conventional smoking method (treatments D and E).

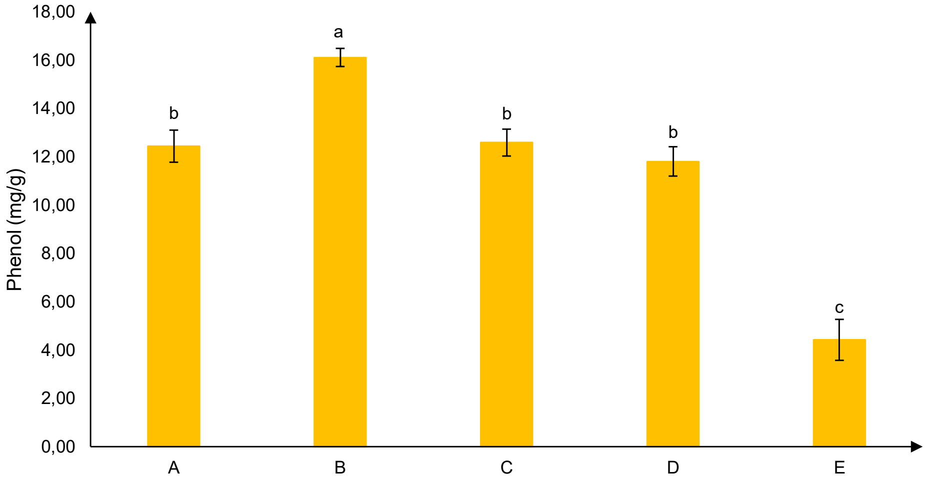

Fig. 6. Phenol level of *julung julung* smoked fillet

The statistical analysis showed a difference between treatments B, A – C, and D – E on the TVB value of smoked fish fillets showed phenol levels in treatments A, C, and D were not different. Previous research reported that the drying treatment of fish meat can increase phenolic compounds in smoked fish products (Sérot *et al*., 2004). The high phenol content in treatment B is thought to be because the pre-heated process causes the fish meat to lose water so that when dipped in liquid smoke, the fish fillets absorb a large amount of liquid smoke. Previous studies have reported that when the fish surface is dried, there is less smoke condensation than products smoked at lower temperatures. The results of this study indicate that a dry fish surface allows for better absorption of smoke components (Belichovska *et al*., 2019).

The study reported phenol levels in liquid smoke from corn cob waste of 0.335 mg/g (Swastawati *et al*., 2007) and 2.55% (Leviyani *et al*., 2019). The phenol level of smoked fish with the conventional method of smoking with corn cobs is lower due to the high acid content, which can potentially reduce the phenol content of smoked fish (Swastawati *et al*., 2012). Anggraini & Nurhazisa (2017) reported that the phenol content of liquid smoke from coconut shells was 3.04%, while liquid smoke from corn cob was 1.38%.

Polycyclic aromatic hydrocarbon (PAH) levels

Polycyclic aromatic hydrocarbons are a group of organic compounds formed by incomplete combustion, such as wood. Therefore, the type of fuel used in the fish smoking process affects the PAH content of smoked fish (Jinadasa *et al*., 2020). One of the carcinogenic PAHs, benzo(a)pyrene, is a carcinogenic

marker in smoked fish products (Stołyhwo & Sikorski, 2005). Polycyclic aromatic hydrocarbon (PAH) levels in smoked fish fillets can be seen in Table 2.

Table 2. Polycyclic aromatic hydrocarbon levels in *julung julung* smoked fillet

Commented [WT10]: PAH testing is not replicated because because it uses the HPLC method

Polycyclic Aromatic Hydrocarbon	Smoked *Julung Julung* fish (µg/kg)					Corn Cob Liquid Smoke 0.8%(µg/kg)
	A	B	C	D	E	
Benzo(a)pyrene	nd	nd	nd	1.5	0.2	0.5
Benzo(b)fluoranthene	1.2	1.0	1.2	2	1.8	2.5
Benzo(a)anthracene	nd	nd	nd	0.5	nd	0.2
Benzo(g)perylene	0.4	0.2	0.4	1.0	0.8	1.0

nd = not detected

Table 2 shows that the PAH levels in smoked fish are still below the standard required in the Indonesian National Standard No. 2725 of 2013 on smoked fish, which is benzo(a)pyrene max 5 µg/kg (Indonesia Standardization Agency, 2013). The polycyclic aromatic hydrocarbon compound regulated in the Indonesian National Standard is only benzo(a)pyrene. Stołyhwo & Sikorski (2005) reported benzo(a)pyrene levels in smoked fish with hot and cold smoking processes ranging from 0.05 to about 60 µg/kg. Other studies have reported benzo(b,k)fluoranthene levels in salmon and rainbow trout ranging from 1.83 to 9.55 µg/kg, while benzo(g,h)perylene levels in salmon were 0.44 µg/kg (Basak *et al*., 2010). Berhimpon *et al*. (2018) also reported benzo(a)pyrene levels in smoked skipjack fish of 0.25 µg/kg. The level of benzo(a)pyrene in smoked barracuda was reported to be 0.32 µg/kg, benzo(b)fluoranthene 0.35 µg/kg, benzo(k)fluoranthene 0.21 µg/kg, benzo(a)anthracene 0.44 µg/kg, and benzo(g,h)perylene 2.56 µg/kg (Asamoah *et al*., 2021).

High PAH levels in smoked fish are due to the lignin content of the material used as fuel in the smoking process (Asamoah *et al*., 2021). The lignin content in corn cobs is reported to be about 17 – 21% (Thangavelu *et al*., 2018; Olajuyigbe *et al*., 2019; Gandam *et al*., 2022). The lignin content of coconut shell was approximately 59.5% (Wang & Sarkar, 2018), while that of coconut wood was about 50% on a dry weight basis (Anuchi *et al*., 2022). Lignin is reported to absorb PAH compounds because PAH compounds can bind well with lignin (Oliveira *et al*., 2019). During pyrolysis and gasification processes, lignin components react to produce aromatic tar and coke, which can form PAH compounds (Zhou *et al*., 2014; Kawamoto, 2017). Therefore, PAH compounds in smoked fish smoked using corncobs are lower than those smoked with shells and coconut wood.

Sensory assessment

A sensory assessment is carried out to evaluate the panelist's preference level, including appearance, aroma, taste, and texture. A sensory assessment must determine the quality of the smoked fish product and ensure it meets the applicable standards (Hadanu & Lomo, 2019). The sensory characteristics of

smoked fish products affect consumer acceptance and preference. Sensory assessment by panelists helps to identify the sensory attributes that are most appealing to consumers, allowing manufacturers to customize products according to consumer assessment (Ekelemu *et al*., 2021). Indonesian National Standard No. 2725:2013 regarding smoked fish requires a minimum sensory assessment of 7 (Score 1-9) (Indonesia Standardization Agency, 2013). The results of the panelists' assessment of *julung julung* smoked fillet from each treatment can be seen in Fig. 7.

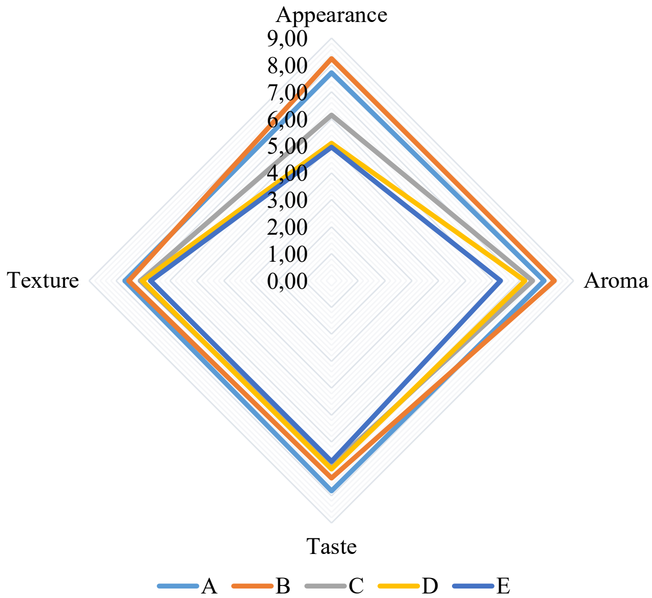

Fig. 7. Sensory assesment of *julung julung* smoked fillet

Fig. 7 shows the analysis of the variance of the sensory assessment of the appearance of smoked *julung julung* fillets with different smoking method treatments affecting the appearance of smoked fish ($p<0.05$). The panelists' assessment of the appearance of smoked fish fillets ranged from 4.95 to 8.24, with the highest panelists' assessment in treatment B. Based on the requirements of the Indonesian National Standard, only treatments A and B met the minimum panelist assessment requirement of 7. The moisture content factor is thought to have influenced the panelists' assessment of the appearance of smoked fish, so panelists less favored treatment C with steaming. Moisture content can affect the physical properties of fish, such as the appearance and texture of smoked fish (Baten *et al*., 2020a). Smoked fish with high moisture content will make the color of smoked fish look paler (Flick, 2010).
Treatments D and E were less favored by panelists, presumably because conventional smoking methods produce darker products (dark brown), less bright and less shiny. Smoking fish with liquid smoke has

smoked fish products with a golden color and clean and shiny surface (Berhimpon *et al*., 2018). Another factor that affects the appearance of smoked fish is the length of the smoking process. The longer the smoking process, the darker the color will be. The time of the smoking process for smoked fish using the liquid smoke method is shorter than the conventional method (Puke & Galoburda, 2020; Baten *et al*., 2020b). The smoking process can change the color of fish, giving it a distinctive golden color due to the interaction of carbonyls with amino components on the surface of the meat. The color and appearance of smoked fish also positively correlate with phenol content. Phenol compounds in smoke interact with amino acid components in fish meat, producing a distinctive golden color in smoked fish (Montazeri *et al*., 2013).

Fig. 7 shows the analysis of the variance of the sensory assessment of the aroma of smoked fish fillets *julung julung* with different smoking method treatments that did not affect the appearance of smoked fish ($p>0.05$). However, the aroma sensory assessment of treatments A, B, C, and D still meets the minimum assessment of the Indonesian National Standard on smoked fish. The sensory assessment of smoked fish aroma was lowest in treatment E. Previous studies have reported that smoking fish with corn cobs as a fuel source resulted in the lowest aroma scores, indicating less aromatic smoked fish (Asmara *et al*., 2022). The aroma and taste of smoked fish are strongly influenced by the time of smoking (Tahir *et al*., 2020). This assessment indicates that the time of the smoking process of 4 and 6 hours in all treatments has not influenced the taste and aroma of smoked fish.

Fig. 7 shows the analysis of the variance of the sensory assessment of the taste of smoked fish fillets *julung julung* with different smoking method treatments that did not affect the appearance of smoked fish ($p>0.05$). The taste of smoked fish is influenced by the time of the smoking process. Previous studies have reported that longer smoking time can improve the sensory attributes of taste (Baten *et al*., 2020b). This result indicates that the 2-hour time difference between the liquid smoke smoking treatment and the conventional smoking method has not affected the taste of smoked fish. A distinctive smoked taste without bitterness is a criterion for assessing the taste of good quality smoked fish (Sukowati *et al*., 2021).

Fig. 7 shows the analysis of variance of the sensory assessment of the texture of smoked fish fillet *julung julung* with different smoking method treatments giving effect to the texture of smoked fish ($p<0.05$). Panelists' assessment of the texture of smoked fish *julung julung*, ranged from 6.71 – 7.67, with the highest panelist assessment in treatments A and B. The statistical analysis showed that treatments A and B differed from treatments C, D, and E. This result was thought to be because the fish fillets were dipped in liquid smoke, giving a dry, compact, and tender texture. Whereas in treatment C (fish fillets subjected to steaming), the texture of the smoked fillets was rather sticky and not solid. Treatments D and E produced the texture of smoked fish fillets which were less dense and not compact. The texture of smoked fish is negatively correlated with its moisture content. The higher the moisture content in

smoked fish, the softer and less firm the texture (Chan *et al*., 2022). High moisture content in smoked fish products results in less dense fish muscle fibers (Chang *et al*., 2021).

A good texture of smoked fish is compact, moderately elastic, and not sticky (Sulistijowati *et al*., 2021). The Indonesian National Standard requires that the texture of smoked fish is dense and compact and the tissue is very tight (Indonesia Standardization Agency, 2013). Based on consumer surveys, the preferred texture of smoked fish is dense, slightly wetter, and easy to chew, and there is a sensation of liquid in the mouth when chewed (Ticoalu *et al*., 2019).

Conclusion

Based on the evaluation of the parameters of total volatile bases, moisture content, water activity, pH, phenol content, polycyclic aromatic hydrocarbon content, and sensory assessment of smoked *julung julung* fillets using liquid smoke from corn cobs, it can be concluded that treatment B is the best treatment, i.e. the fillets were preheated for 4 hours at 60 – 80 °C, then dipped in liquid smoke for 20 minutes, and the fillets were reheated for 4 hours at 90 °C. In general, the characteristics of smoked fillets using liquid smoke from corn cobs were better when compared to the conventional smoking treatment. It is necessary to evaluate different smoking times on smoked *julung julung* fillets with the corn cob liquid smoke method.

Acknowledgment

Author contributions

Conflicts of interest

The authors declare that there is no conflict of interest.

Highlights

Effectiveness of using liquid smoke derived from corn cob waste in the smoking process of *julung julung* fillets

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Commented [WT11]: Thanks for the suggestion, but We apologize, we used a lot of literature in compiling this article. This is to support our research results. If it is reduced, we think it will reduce this article's comprehensive discussion.

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Reviewer 1

Comment 1

Line 179. Change Data analysis to Statistical analysis.

State replicates for all tests.

Response

Statistical analysis

Statistical analysis was performed using Statistical Product and Service Solutions (SPSS) version 20.0. Statistical analysis of data was performed based on one-way analysis of variance (ANOVA) using a significance level of ($P<0.05$). Specific group differences were determined using Duncan's test. The TVB, total phenol, pH, moisture, water activity, and sensory assessment parameters were evaluated with three replicates.

Comment 2

In figures just put Duncan's letters on the bars. There is no need to mention the numbers on the bars.

Provide the proper abbreviations for the treatments in figures, tables as well as the manuscript text.

Duncan's letters are misattributed, the first letters must show the biggest numbers.

Response

It has been added and corrected

Comment 3

Also, Duncan's letters should be added in Table 2.

Response

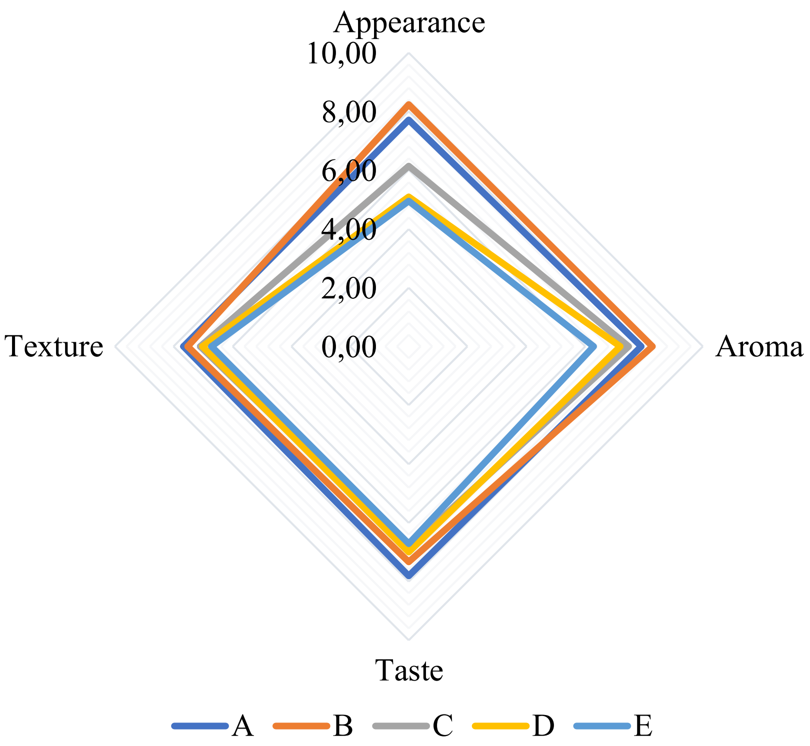
PAH testing is not replicated because because Data in table 2 using the HPLC method

Comment 4

Sensory evaluation data should be present as a radar plot.

Response

It has been corrected



Comment 5

Delete methods from the conclusion section.

It has been corrected

Conclusion

Based on the evaluation of the parameters of total volatile bases, moisture content, water activity, pH, phenol content, polycyclic aromatic hydrocarbon content, and sensory assessment of smoked *julung julung* fillets using liquid smoke from corn cobs, it can be concluded that treatment B is the best treatment, i.e. the fillets were preheated for 4 hours at 60 – 80 °C, then dipped in liquid smoke for 20 minutes, and the fillets were reheated for 4 hours at 90 °C. In general, the characteristics of smoked fillets using liquid smoke from corn cobs were better when compared to the conventional smoking treatment. It is necessary to evaluate different smoking times on smoked *julung julung* fillets with the corn cob liquid smoke method.

Comment 6

The last line of the abstract and conclusion should be revised and the best method mentioned.

Response

It has been corrected

Abstract

This study aimed to obtain the best smoking method for *julung julung* fillets (*Hemiramphus* sp.) using liquid smoke from corn cob waste. This study used three different fillet treatment methods (dried and steamed) before liquid smoke application. This study also compared fish smoking between using liquid smoke with conventional methods (using shell, coconut wood, and corn cob). The parameters to determine the quality of smoked fish were Total Volatile Bases (TVB), moisture, water activity, pH, phenol levels, polycyclic aromatic hydrocarbon (PAH), and sensory assessment. The results showed that the TVB of smoked *julung julung* fillets ranged from 19.83 – 32.27 mg N/100g. The moisture ranged from 14.10 – 30.57%. Water activity ranged from 0.46 – 0.82. pH ranged from 4.60 – 5.81. Phenol levels ranged from 4.42 – 16.11 mg/g. PAH levels are still below the standard required in the Indonesian National Standard. Panelists rated neutral to really like the appearance, aroma, taste, and texture of smoked fish. From these research, it can be concluded that treatment B is the best treatment, namely fresh fillets are first heated for 4 hours at 60 – 80 ºC and then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated for 4 hours at 90 ºC.

Reviewer 2

Comment 1

Keywords : Corn cobs, Liquid smoke

Response

Keywords: Corn cobs, Indonesia, Liquid smoke, North Sulawesi, Wood

Comment 2

Materials and Methods (Line 90) : Why used this concentration??

Response

Because based on the results of Berhimpon et al., 2018, the best concentration of liquid acid from corn cobs for fish smoking is at a concentration of 0.8%.

Commnet 3

Line 98 Treatments : References

Response

The treatments in this study were based on the results of Berhimpon et al. (2018) with added treatments, namely smoking fish without liquid smoke (using wood, head, coconut shell, and corn cobs).

Comment 4

Line 103 : Temperature of combustion furnace.

Response

The combustion furnace, at 400 ºC and have a pyrolysis time of about 120 minutes (Based Aladin *et al*., 2018).

Commnet 5

Line 108 : Reference and How?? To separate what?? how about benzopyrene?

Response

The pyrolysis based on Based Aladin *et al*., 2018

HPLC evaluated the level of benzopyrene from liquid smoke. And the benzopyrene in liquid smoke was about 0.5 µg/kg (Table 2).

Comment 6

Line 111 : Volatile Base

Response

It has been corrected

Comment 7

Line 172 : Without any prepation??

Response

Without preparation,because the smoked fish samples used are still suitable for consumption (under 24 hours since smoking).

Comment 8

Line 458 : A lot! Decrease that

Response

Thanks for the suggestion, but I apologize, we used a lot of literature in compiling this article. This is to support our research results. If it is reduced, we think that it will reduce the comprehensive discussion in this article.

Reviewer 3

Comment 1

Title : ~~Fish~~

Response

It has been corrected

Characteristics of Julung Julung Smoked Fillets (*Hemiramphus* sp.) using Liquid Smoke from Corn Cobs Waste

Comment 2

Line 4-6 : Delete please

Response

It has been deleted

Comment 3

Line 7 : Edit the sentence

Response

It has been edited

This study used three different fillet treatment methods (dried and steamed) before liquid smoke application. This study also compared fish smoking between using liquid smoke with conventional methods (using shell, coconut wood, and corn cob).

Comment 4

Line 11 : Delete

Response

It has been deleted

Comment 5

Line 22 : update the data for 2024

Response

There is no data yet for 2024. We only have data for 2023.

Indonesia's maize production in 2023 is 14.46 million tons, ranking 9th in the world. (Indonesia Center of Statistic Agency, 2023).

Comment 6

Line 30 : edit please

Response

Swastawati et al. (2007) reported the phenolic content in liquid smoke produced from 335 mg/L corn cobs.

Comment 7

Line 35 : Double "and"

Response

Using liquid smoke in food will save time, energy, and labor, so that can reduce production costs.

Comment 8

Line 89 : Weeded ???

Response

It has been corrected to be cleaned

Comment 9

Line 107 : Tar???

Response

charcoal formed from the pyrolysis process

Comment 10

Line 110-116 : It doesn't need to write the details of routin methods

Response

The TVB testing method is based on the instructions of Tambunan and Chamidah (2021). We still include the complete work procedure, so that readers will get complete information.

Comment 11

Line 146 : Did you used standard curve?

Response

Yes, using gallic acid as standar curve

Comment 12

Line 173 : Trained?

Response

Smoked fish samples were placed on a plastic plate with a glass of water, coded, and presented to 30 randomly selected semi-trained panelists (university students who had studied sensory assessment techniques), and the assessment process was carried out under light.

Comment 13

Line 203 : Please use a code for each treatment the figure is difficult to understand

Response

It has been corrected

Comment 14

Line 218 : The statistical analysis showed

Response

It has been corrected

Comment 15

Line 221 : Please define code below the figure and then mention in the text

Response

It has been corrected

Comment 16

Line 412 : delete

Response

It has been deleted

Comment 17

Line 438 : delete in conclusion

Response

It has been deleted

7. Bukti manuskrip telah diterima setelah revisi II

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Jefri Mand <jefri.mandeno76@gmail.com>

Acknowledgement of Revision (#JRIFST-2406-1571 (R2))

1 pesan

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Manuscript ID: JRIFST-2406-1571 (R2)

Manuscript Title: **Characteristics of Julung Julung Smoked Fillets (Hemiramphus sp.) using Liquid Smoke from Corn Cobs W**

Authors: Jefri Anthonius Mandeno, Wendy Alexander Tanod, Eko Cahyono, Yana Sammbeka, Frets Jonas Rieuwpassa, Novalina Ma
Putra Palawe, Putut Har Riyadi

Date: 2024-06-03

Dear Mr. Jefri Anthonius Mandeno

Thank you for submitting the revised file of your manuscript to the **Research and Innovation in Food Science and Technology**

The Editorial Office will proceed on your manuscript and inform you in the earliest time.

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8. Bukti manuskrip telah diterima dan akan dipublikasikan

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Acceptance of Manuscript (#JRIFST-2406-1571 (R2))

1 pesan

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Certificate of Acceptance

Number: JRIFST-2406-1571 (R2)/137

Accepted: February 16, 2025

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Authors: Jefri Anthonius Mandeno, Wendy Alexander Tanod, Eko Cahyono, Yana Sammbeka, Frets Jonas Rieuwpassa, Novalina
Putra Palawe, Putut Har Riyadi

Dear **Mr. Jefri Anthonius Mandeno**

Thank you for submitting your manuscript to Research and Innovation in Food Science and Technology.

I am pleased to inform you that the submitted manuscript entitled " **Characteristics of *Julung Julung* Smoked Fillets (*Hemira*
from Corn Cobs Waste " has been accepted as " Original Paper " for publication and it will now be transferred to our production d

We appreciate you selecting our journal to publish the scientific work of yours and hope you will consider us again for future submi

Yours sincerely,

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9. Bukti permohonan revisi final sebelum dipublikasikan

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Manuscript ID: JRIFST-2406-1571 (R2)

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Dear **Mr. Jefri Anthonius Mandeno**

I, on behalf of JRIFST, appreciate choosing our journal for publishing the invaluable work of yours.

Please revise the manuscript based on the comments provided by the English editor and then send it to the journal at jrifst@gmail.com within **2 days.**

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Best wishes,

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10. Artikel published

Characteristics of *Julung Julung* Smoked Fillets (*Hemiramphus* sp.) using Liquid Smoke from Corn Cobs Waste

Commented [t1]:

Reviewer comment for author

1-The responses to reviewers comments should be attached (Reviewers 1, 2 and 3), (Necessary)

2-The responses to final reviewer comments should be attached (Necessary).

3-The radar plot is drawn incorrectly and needs to be corrected. Attributes must be at the vertices of the polygon.

Abstract

This study aimed to obtain the best smoking method for *julung julung* fillets (*Hemiramphus* sp.) using liquid smoke from corn cob waste. This study used three different fillet treatment methods (dried and steamed) before liquid smoke application. This study also compared fish smoking between using liquid smoke with conventional methods (using shell, coconut wood, and corn cob). The parameters to determine the quality of smoked fish were Total Volatile Bases (TVB), moisture, water activity, pH, phenol levels, polycyclic aromatic hydrocarbon (PAH), and sensory assessment. The results showed that the TVB of smoked *julung julung* fillets ranged from 19.83 – 32.27 mg N/100g. The moisture ranged from 14.10 – 30.57%. Water activity ranged from 0.46 – 0.82. pH ranged from 4.60 – 5.81. Phenol levels ranged from 4.42 – 16.11 mg/g. PAH levels are still below the standard required in the Indonesian National Standard. Panelists rated neutral to really like the appearance, aroma, taste, and texture of smoked fish. From these research, it can be concluded that treatment B is the best treatment, namely fresh fillets are first heated for 4 hours at 60 – 80 ^{o}C and then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated for 4 hours at 90 ^{o}C.

Keywords: Corn cobs, Indonesia, Liquid smoke, North Sulawesi, Wood

Introduction

Corn is the second most widely grown crop in Indonesia after rice. Indonesia's maize production in 2023 is 14.46 million tons, ranking 9th in the world. (Indonesia Center of Statistic Agency, 2023). North Sulawesi is one of the contributing regions that produce corn in Indonesia. Considerable corn production will also produce waste, especially corn cobs, which are generally thrown away and burned by most Indonesians (Cahyadi *et al*., 2021).

One of the corn cobs was used to make liquid smoke. Previous research reported that corn cobs can produce liquid smoke, a by-product of the pyrolysis of corn cob waste. The yield of liquid smoke from corn cobs is about 28.37%, with a pH value of 3.5 (Sriharti *et al*., 2020). The particle size of corn cobs affects the yield of liquid smoke, with smaller particle sizes resulting in higher yields (Aladin *et al*., 2018). Swastawati et al. (2007) reported the phenolic content in liquid smoke produced from 335 mg/L corn cobs.

Liquid smoke is a natural product made from the condensation of smoke from burning wood (Andy *et al*., 2021). Liquid smoke is commonly used as a flavoring in food to provide a smoked flavor without the food undergoing the actual smoking process (Sari *et al*., 2006). Using liquid smoke in food will save time, energy, and labor, so that can reduce production costs (Krah *et al*., 2019). In addition, using liquid smoke in food can speed up and standardize the smoking process, adding flavor and microbiological safety while reducing production time (Indiarto *et al*., 2020). Generally, various types of hardwood were used to manufacture liquid smoke (Diatmika *et al*., 2019). The quality of the liquid smoke produced

may vary depending on the type of wood used (Budaraga *et al*., 2016). In addition to imparting specific flavors and aromas to foods (Maulina *et al*., 2020), liquid smoke is reported to be antimicrobial (Septana *et al*., 2020) and antioxidant (Budaraga & Putra, 2021).

The conventional smoking processes generally use hardwood as fuel, such as melon wood (Umar *et al*., 2018), mangrove wood (Cissoko *et al*., 2020), teak wood (Daramola *et al*., 2020), coconut shells, and coconut husks (Nugroho *et al*., 2018). Corn cobs have also been reported to be used in the fish smoking process (Sukowati, 2023). According to literature, traditional fish smoking in North Sulawesi, Indonesia, uses smoke from burning coconut shells or wood charcoal (Landangkasiang *et al*., 2017; Primalasari *et al*., 2019; Saediman *et al*., 2021). Smoked fish processing has been known for a long time because of its traditional processing techniques, simplicity, ease of implementation, and low cost (Islamiyah, 2021). In North Sulawesi, Indonesia, smoked fish commonly known as *fufu* is traditionally made from skipjack (*Katsuwonus pelamis*); *julung julung* (*Hemiramphus far*) (Berhimpon *et al*., 2018); and scad fish (*Decapterus* spp.) known as *pinekuhe* (Ansar & Ijong, 2021). The smoked fish product of *julung julung* is a typical product from North Sulawesi and Gorontalo, commonly called *roa* or *galavea* (Azis & Akolo, 2020).

The study's reported that the conventional smoking process has disadvantages such as smoking time, concentration of carcinogenic substances, temperature, and inconsistent product quality (Racovita *et al*., 2020). Conventional smoking of food products has been shown to produce carcinogenic components such as polycyclic aromatic hydrocarbons (Jinadasa *et al*., 2020). Benzo(a)pyrene ($C_{20}H_{12}$) is one of the carcinogenic compounds produced from traditional smoking products (Jinadasa *et al*., 2020). One method to reduce carcinogenic compounds in smoked fish products is to use liquid smoke in the smoking process (Nithin *et al*., 2020; Xin *et al*., 2021).

The utilization of liquid smoke in the fish smoking industry in North Sulawesi is very likely to be developed. North Sulawesi was reported to have both large-scale and domestic fish-smoking industries (Dotulong *et al*., 2018; Primalasari *et al*., 2019). Making liquid smoke is relatively simple, so it will be readily accepted by fish-smoking industry players (Ali & Al Fiqri, 2020). Previous research reported that using liquid smoke in smoked fish products resulted in good quality in terms of appearance, flavor, and aroma (Rizal *et al*., 2020). In addition, using liquid smoke in smoked fish products can add nutritional value and durability, making it possible to reach a wider market area (Ali *et al*., 2021).

Previous research reported that smoked fish dipped in liquid smoke for 15 minutes had a total plate count value of 4.7×10^4 CFU/g on day six and moisture content below 60% during storage (Suroso *et al*., 2018). Dipping fish in liquid smoke with a concentration of 15% and a dipping time of 60 minutes showed a bacterial colony of 2.12×10^2 CFU/g (Ali *et al*., 2021). The Indonesian National Standard on smoked fish requires a maximum total plate count of 5×10^4 CFU/g and a maximum moisture content of 60% (Indonesia Standardization Agency, 2013). Another study reported that smoked fish processed

with liquid smoke had a moisture content of 47.63%, phenol content of 12.62%, and pH of 4.8 (Berhimpon *et al*., 2018). The results of these studies indicate that liquid smoke is good to apply to smoked fish products. This study characterized smoked fish fillets of *julung julung* (*Hemiramphus* sp.) produced with liquid smoke from corn cobs waste. The study was conducted by dipping the fish fillets of *julung julung* with liquid smoke, with drying and steaming treatments on the fish fillets. This study aims to obtain the best smoking method for fish fillets using liquid smoke from corn cobs waste based on the assessment of total volatile bases, moisture content, water activity, pH value, phenol content, polycyclic aromatic hydrocarbon content, and sensory assessment.

Materials and methods

The main materials of the study were *julung julung* fish (*Hemiramphus* sp.), corn cob (*Zea mays* L.) from Gangga Island in Likupang, North Sulawesi, Indonesia. This study includes the preparation of liquid smoke from corn cobs waste, preparing fish (weeding and filleting), and treating fish fillets by dip in liquid smoke. Fresh *julung julung* fish was obtained from traditional fishermen in Manado, North Sulawesi, Indonesia (Fig. 1). The fish was placed in a cool box and given ice in a ratio of 1:1 (ice:fish) (Mishra *et al*., 2021). Then, the fish was washed, cleaned, and filleted. The cleaned fillets were dipped in a 5% salt solution for 30 minutes. The concentration of liquid smoke from corn cob waste was 0.8% (Berhimpon *et al*., 2018).

Fig. 1. *Julung julung* (*Hemiramphus* sp.)

The treatment in this study was applying liquid smoke from corn cobs waste to the smoking process of *julung julung* fillets, which was compared with the conventional method (using coconut wood, coconut shells, and corn cobs).

(A) : Fresh fillets were dipped in liquid smoke for 20 minutes and then dried in a cabinet dryer for 4 hours at 90 °C.

(B) : Fresh fillets were preheated in a cabinet dryer for 4 hours at 60 – 80 °C, then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated in a cabinet dryer for 4 hours at 90 °C.

(C) : Fresh fillets were steamed for 30 minutes, then dipped in liquid smoke for 20 minutes. After that, the fillets were dried in a cabinet dryer for 4 hours at 90 °C.

(D) : Fresh fillets were smoked with the conventional method using wood and coconut shell for 6 hours at 90 ^{o}C.

(E) : Fresh fillets were smoked with the conventional method using corncob for 6 hours at 90 ^{o}C.

Liquid smoke manufacturing process

Making liquid smoke refers to modified research (Aladin *et al*., 2018). Modifications were made to the equipment used in the pyrolysis process. Dry corn cobs were weighed to determine fuel efficiency. Corn cobs are gradually burned into the combustion furnace, at 400 ^{o}C and have a pyrolysis time of about 120 minutes. The furnace was closed to prevent smoke from escaping from the tank. The smoke generated from the combustion flows through a pipe connected to a storage tank covered with ice cubes. The smoke that passes through the pipe will become cold, so condensation occurs, turning the smoke into liquid. The smoke that has been formed is collected into a container attached to the end of the pipe. The liquid smoke obtained is then allowed to settle the tar formed for three weeks and filtered (using Whatman filter paper No. 10) to obtain clear liquid smoke.

Total volatile bases assay

Total Volatile Base (TVB) assay refers to research by Tambunan & Chamidah (2021). TVB assay aims to determine the amount of volatile base compounds formed from protein breakdown—the principle of TVB analysis, namely by evaporating volatile base compounds at room temperature for 24 hours. The compound is then bound with boric acid and titrated with an HCl solution. After the boric acid solution was stored in the inner chamber of the Conway cell containing the blank (V_0) and the filtrate (V_1), the boric acid solution was titrated with 0.02 N HCl so that the boric acid solution changed color to pink. TVB levels in the smoked fish meat were expressed as mg N/100g.

$$TVB\left(\mathrm{mg}\frac{\mathrm{N}}{100\mathrm{g}}\right) = \frac{(V_1 - V_0) \times N.HCl \times dilution \times 100}{sample\ weight\ (g)}$$

Moisture content assay

The moisture content is determined by the Indonesia National Standard (Indonesia Standardization Agency, 2006a). An empty porcelain cup is weighed (A). Then, the sample was weighed to ± 2 g in a porcelain cup of known mass (B). The samples were dried in an oven at 105°C for 20 hours. The samples were then cooled in a desiccator, after which the samples in the cup were weighed (C). Moisture content was expressed as %.

$$Moisture\ (\%) = \frac{B\ (g) - C\ (g)}{B(g) - A\ (g)} \times 100\%$$

Water activity (Aw) assay

Water activity assay used an Aw meter (Rotronic, HygroPalm 23-AW-A). The Aw meter was set up at room temperature for 2 hours. The water activity of smoked fish was measured by placing the sample into a sample container and conditioning it for 30-60 minutes. The Aw meter sensor is contacted with the sample in the container. Then the water activity (Aw) value can be read on the Aw meter panel (Saputra *et al*., 2014).

pH assay

The sample weighed as much as 30 g, then 400 mL of distilled water was added at 28 ºC. The sample was homogenized with a magnetic stirrer to be evenly stirred. The pH meter electrode was inserted into the sample solution. The data taken in this test is the pH value read by the pH meter (Hanna Instruments HI99192) (Lekahena & Jamin, 2018).

Phenol level assay

Samples were extracted by adding 1 mL ethanol p.a., 5 mL distilled water, and 0.5 mL Folin Ciocalteu 50%, homogenized, and allowed to stand for 5 minutes. Then, 1 mL of 5% Na_2CO_3 and left in the dark for ± 60 minutes with gallic acid as a standard. The absorbance value was read on a spectrophotometer (Thermo Scientific Genesys 50) with a wavelength of 725 nm. Total phenolics were expressed as mg/g (Indonesia Standardization Agency, 2004; Muliadin *et al*., 2022).

$$Total\ Phenol\ (\frac{mg}{g}) = \frac{Gallic\ acid\ equivalence\ \left(\frac{mg}{L}\right) \times Volume\ (L)}{Sample\ weight\ (g)}$$

Polycyclic aromatic hydrocarbon (PAH) assay

PAH content testing was carried out based on the instructions Basak *et al*. (2010). A 5 g sample was weighed and collected in a 100 ml flask. Then 5 mL of 50% KOH solution, 75 mL of methanol, 1 mL of internal standard solution (9,10 dimethylantracene), and some boiling stones were added. After that,

the mixture was boiled for 4 hours in a soxhelet. The liquid phase was transferred to a separatory funnel and extracted with 100 mL of n-hexane with shaking for 3 min. The MeOH:KOH phase was drained and discarded. The combined n-hexane phase was rinsed twice, each with 50 mL of H_2O-MeOH (8:1) and 50 mL of water. The methanol-water and water phases were dried and discarded. The organic phase was concentrated in a rotary evaporator (40°C) to a volume of about 10 mL. The concentrated extract was dried under nitrogen gas.

After that, the silica gel column was cleaned by dissolving 15 g of silica gel in n-hexane and transferred to a column (200 mm long; diameter: 12 mm). Then, 1 g of anhydrous sodium sulfate was added. The concentrated sample extract was transferred to the column and eluted with 1 mL of n-hexane. A stock solution containing 0.1 mg mL^{-1} of 9,10 dimethylantracene was dissolved in n-hexane and stored at 4°C in a volumetric flask (with a glass stopper) wrapped in aluminum foil to avoid possible light degradation. Working standard solutions were prepared from the stock solutions. Analysis of PAH compounds used a Hewlett-Packard 1100 HPLC equipped with an Agilent-110 fluorescence detector. The injection volume was 10 μL. Mobile phase gradient (acetonitrile-water): 80% acetonitrile + 20% water with a flow rate of 1 ml min^{-1} and a wavelength of 270 nm. The external standard mixture was used for PAH concentration calculation.

Sensory assessment assay

Sensory assessment assay refers to the Indonesia National Standard (Indonesia Standardization Agency, 2006b). The sensory assessment was based on a hedonic scale (Table 1). Smoked fish samples were placed on a plastic plate with a glass of water, coded, and presented to 30 randomly selected semi-trained panelists (university students who had studied sensory assessment techniques), and the assessment process was carried out under light. The parameters observed were appearance, aroma, taste, and texture. Panelists rated acceptance of the samples on a scale of 1 – 9.

Table 1. Score sheet of hedonic assessment

Specification	Score
Really like it	9
Really like	8
Like	7
Rather like	6
Neutral	5
Rather dislike	4
do not like	3
Very dislike	2
Really don't like it	1

Statistical analysis

Statistical analysis was performed using Statistical Product and Service Solutions (SPSS) version 20.0. Statistical analysis of data was performed based on one-way analysis of variance (ANOVA) using a significance level of ($P<0.05$). Specific group differences were determined using Duncan's test. The TVB, total phenol, pH, moisture, water activity, and sensory assessment parameters were evaluated with three replicates.

Results and discussion

Liquid smoke production was carried out using a simple liquid smoke distillation device. This process consumed 17 kg of corn cobs and 12 kg of crushed ice. Liquid smoke from corn cobs is more accessible than coconut shells or wood (Maulina & Karo, 2021). Using coconut shells or wood as fuel will form a flame, thus reducing the volume of smoke in the furnace (Kabir Ahmad *et al*., 2022). The smoked *julung julung* were analyzed for TVB, moisture content, water activity, pH value, phenol content, PAH content, and sensory assessment.

Total volatile bases

The total volatile base (TVB) value is measured to determine the quality of the smoked fish. TVB value is one of the parameters used to determine the decline in fish quality and measures the amount of volatile base compounds formed due to protein degradation (Castro *et al*., 2006). Volatile bases formed in fish muscle tissue mainly consist of ammonia, trimethylamine (TMA), and dimethylamine (DMA) (VELP Scientifica, 2013). TVB value is an essential characteristic for the quality assessment of seafood products and is emerging as the most commonly used chemical parameter to assess the palatability of seafood (Rasulu *et al*., 2020). The results of *total volatile bases* on smoked fish fillets of *julung julung* are presented in Fig. 2.

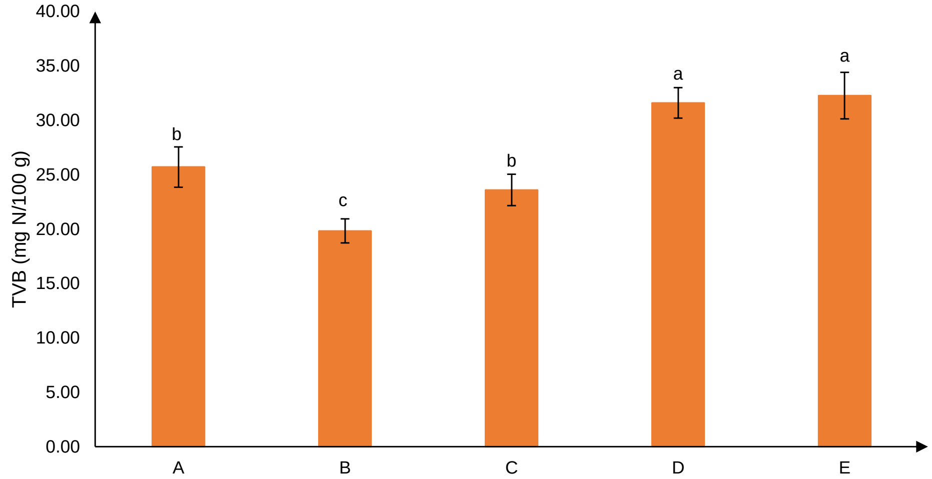

Fig. 2. Total volatile bases of *julung julung* smoked fillet

Fig. 2 shows the analysis of the variance of smoked fish fillets with different smoking method treatments affecting changes in TVB content ($p<0.05$). The TVB content of smoked fish fillets ranged from 19.83 – 32.27 mg N/100g, with the lowest TVB value in treatment B. The treatment of the smoking method with liquid smoke from corn cob waste (treatments A, B, and C) had lower TVB levels when compared to the conventional smoking method (treatments D and E). A literature search has not found why liquid smoking has a lower TVB value than conventional smoking methods. However, one possibility is that the liquid smoke smoking method allows the smoke components to be absorbed into the fish meat better than the conventional smoking method. Better absorption of liquid smoke phenol components in fish meat can inhibit and control microbial growth that causes a decrease in the quality of smoked fish (Santoso *et al*., 2015). A literature study showed that the average TVB value of smoked mackerel (*Euthynnus affinis*) treated with liquid smoke addition ranged from 24.63 – 28.38 mg N/100g (Hardianto & Yunianta, 2015). Another study reported smoked mackerel with a smoking process using rubber wood liquid smoke, producing smoked fish products with lower TVB values (Suroso *et al*., 2018).
The statistical analysis showed a difference between treatments B, A – C, and D – E on the TVB value of smoked fish fillets. The analysis of variance indicates that the drying process of smoked fish fillets before immersion into liquid smoke can maintain the quality of smoked fish (Kaparang *et al*., 2013). The statistical analysis also showed that treatments A – C and D – E had no difference in TVB value, indicating that the moisture content between the two treatments was not different. TVB value is closely related to moisture content (Rasulu *et al*., 2020).

The European standard (European Market Observatory for Fisheries and Aquaculture, 2020) states that the limit range of TVB value for smoked fish is 25 – 35 mg N/100 g. All treatments' TVB value of the smoked fish fillets still met the European standard. Previous studies reported TVB values of fresh fish fillets of 8.70 ± 0.86 mgN/100g (Moosavi-Nasab *et al*., 2021) and 12.94 ± 0.92 mg N/100g (Bouzgarrou *et al*., 2020). Messina *et al*. (2021) reported that smoked fish fillets that underwent two drying processes had a TVB value of <20 mg N/100g. Another study also reported the TVB value of smoked fish fillet samples with hot smoking and cold smoking, which amounted to 17.80 ± 0.17 and 18.95 ± 0.20 mg N/100g, respectively (El-Lahamy *et al*., 2019). Aydin *et al*. (2020) reported a TVB value of smoked fish with hot smoking of 11.82 ± 0.05 mg N/100g.

Moisture content

Moisture content is an important parameter to determine the quality of smoked fish produced. The moisture content contained in smoked fish can affect the shelf life of smoked fish because water in food is a medium for microbes to grow (Kaban *et al*., 2019). The smoking process was reported to reduce the moisture content of fresh fish to a range between 9 and 17% (Sikoki & Aminigo, 2002). The moisture content of smoked fish fillets is presented in Fig. 3.

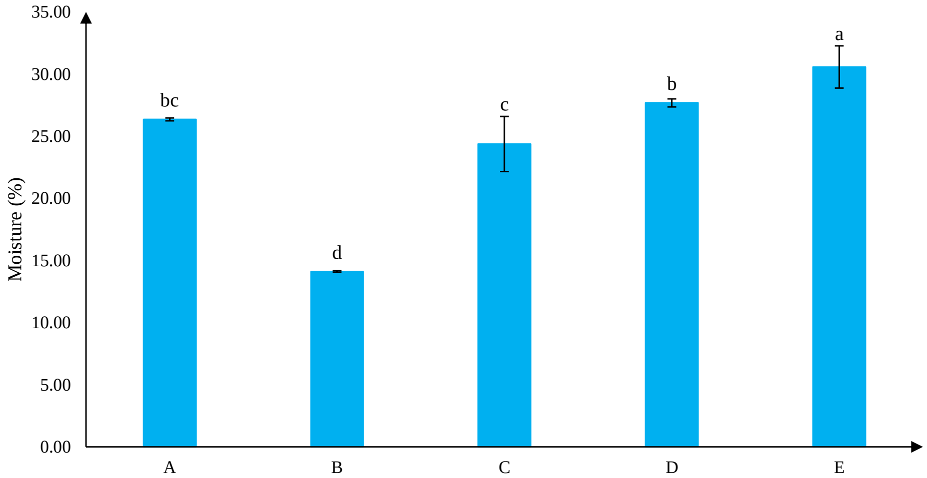

Fig. 3. Moisture content of *julung julung* smoked fillet

Fig. 3 shows the analysis of the variance of smoked *julung julung* fillets with different smoking methods treatment affects moisture content ($p<0.05$). The moisture content of smoked *julung julung* fillets ranged from 14.10 – 30.57%, with the lowest moisture content in treatment B. The moisture content

value in smoked fish products from all treatments still meets the Indonesian National Standard No. 2725:2013 on smoked fish, which is a maximum of 60% (Indonesia Standardization Agency, 2013). Darianto *et al*. (2018) reported that the smoking process can reduce the moisture content of fish to below 40%, which can help preserve it longer. The treatment of the smoking method with liquid smoke with corn cob (Treatment A, B, C) has a lower moisture content when compared to the treatment of the conventional smoking method (Treatment D and E). This result is because the smoking chamber is not fully enclosed in the conventional smoking method, so the heat generated could be more optimal. Suboptimal heat can increase moisture content and cause the moisture content of smoked fish to decrease only slightly (Amos & Paulina, 2017). Whereas in the liquid smoke smoking method, temperature and humidity can be controlled better so that the moisture content of the product can be reduced efficiently (Salindeho & Lumoindong, 2017).

The statistical analysis showed a difference between treatments B, A – C, and D – E on the TVB value of smoked fish fillets showed a difference between treatment B, treatment A – C, and treatment D – E on the moisture content of smoked fish fillets. The results of the variance study indicate that the drying process of smoked fish fillets before the dip in liquid smoke in treatment B can help reduce the moisture content. This study's results are from previous research, which also reported a significant decrease in moisture content in smoked fish products with two times drying treatment (Messina *et al*., 2021). The statistical analysis also showed that treatments A and C were not different because steaming in treatment C will increase the moisture content of the product (Salmatia *et al*., 2020). The conventional smoking method with corn cob fuel has the highest moisture content; this result is the conventional smoking process with corn cobs, and the heat generated is lower than smoking using wood (Asmara *et al*., 2022).

Water activity (Aw)

Water activity is one of the essential parameters in the quality of smoked fish. Water activity (Aw) is expressed as the ratio of the vapor pressure in the food to the vapor pressure of pure water, and it predicts whether water tends to move from the food product into the cells of microorganisms that may be present. A well-smoked fish has a water activity of <0.50 and a moisture content between 15 and 25% to inhibit the growth of pathogenic microorganisms in smoked fish products (Mondo *et al*., 2020). According to British Columbia Centre for Disease Control (2013), the maximum water activity value for smoked fish is 0.97. Water activity correlates with the moisture content of a smoked fish product and is two important factors affecting food safety and quality (Fitri *et al*., 2022). The water activity values of smoked fish fillets can be seen in Fig. 4.

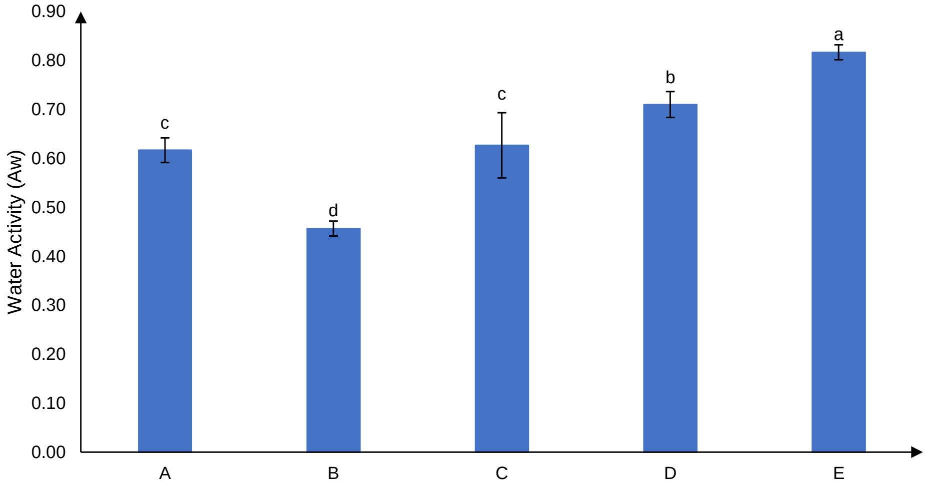

Fig. 4. Water activity of *julung julung* smoked fillet

Fig. 4 shows the analysis of the variance of smoked fish fillet *julung julung* with different smoking method treatments affecting water activity ($p<0.05$). The water activity value of smoked fish fillet ranged from 0.46 – 0.82, with the lowest water activity in treatment B. The treatment of the smoking method with corn cob liquid smoke (Treatment A, B, C) had a lower water content when compared to the treatment of the conventional smoking method (treatment D and E).

The water activity value of fish meat can vary depending on the cooking method, temperature, and duration of heating (Oliveira *et al*., 2017; Zhang *et al*., 2023). Therefore, the factor of fish fillet treatment before immersion in liquid smoke caused the difference in water activity value in treatment B compared to treatments A and C. The water activity value is directly proportional to the water content value of each treatment. The heating process can reduce the moisture content of fish meat (Kiczorowska *et al*., 2019), thus affecting the water activity of fish meat (Gómez *et al*., 2020).

pH

The pH value of smoked fish is an essential factor affecting its quality. The pH value will decrease with increasing smoking time (Baten *et al*., 2020b). The analysis of the variance of smoked fish fillets *julung julung* with different smoking method treatments affects the pH value ($p<0.05$). The pH of smoked fish fillet ranged from 4.60 to 5.81 (Fig. 5). The treatment of the smoking method with corn cob liquid smoke (treatments A, B, and C) had a lower pH value when compared to the conventional smoking method (treatments D and E). Swastawati *et al*. (2022) also reported that the pH value of smoked barracuda fish with the liquid smoke method was significantly lower than that of the conventional

method. Another study also reported that using liquid smoke can reduce pH caused by the condensation of organic acids in the smoking process (Puke & Galoburda, 2020). The pH value of smoked fish correlates with the phenol content contained in the smoke component, and an increase in phenol content causes a decrease in pH (Berhimpon *et al*., 2018).

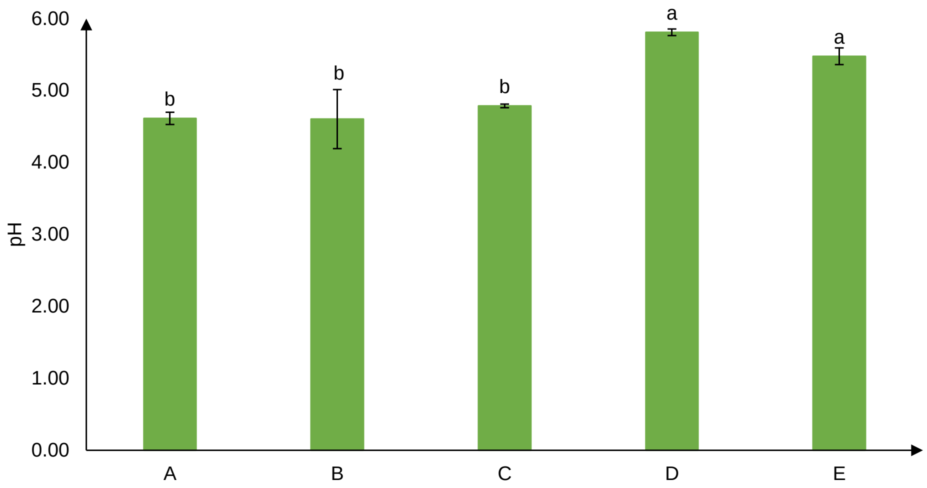

Fig. 5. pH of *julung julung* smoked fillet

Phenol level

Phenol is a compound found in wood smoke. Phenol compounds enter food through diffusion and capillary action, affecting taste, color, and aroma and extending shelf life (Remy *et al*., 2016). In addition, phenol compounds are also reported to act as antioxidants that can prevent rancidity in fish meat (Sérot *et al*., 2004). Leksono *et al*. (2020) also noted that the higher the phenol level in smoke, the stronger the flavor and aroma of smoked fish. The analysis of the variance of smoked fish fillets julung julung with different smoking method treatments affecting phenol level ($p<0.05$). The phenol level of smoked fish fillet ranged from 4.42 to 16.11 mg/g (Fig. 6). The treatment of the smoking method with corn cob liquid smoke (treatments A, B, and C) had higher phenol level when compared to the conventional smoking method (treatments D and E).

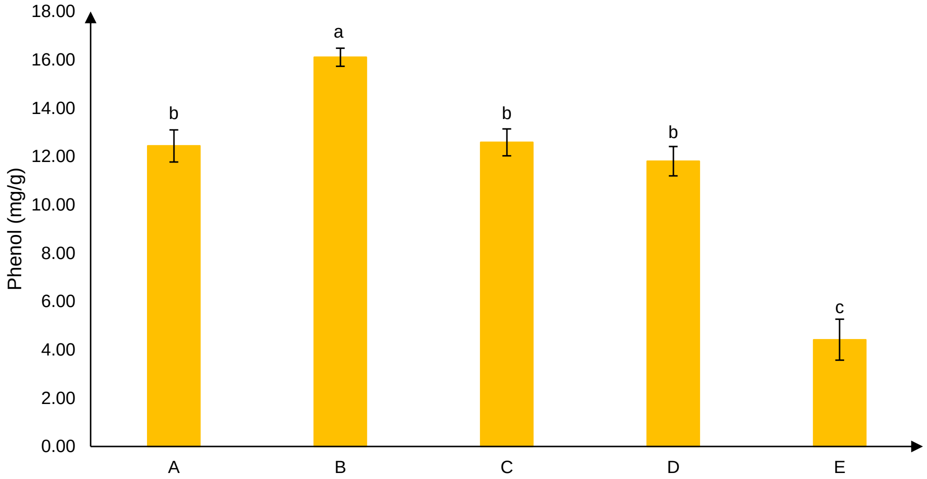

Fig. 6. Phenol level of *julung julung* smoked fillet

The statistical analysis showed a difference between treatments B, A – C, and D – E on the TVB value of smoked fish fillets showed phenol levels in treatments A, C, and D were not different. Previous research reported that the drying treatment of fish meat can increase phenolic compounds in smoked fish products (Sérot *et al*., 2004). The high phenol content in treatment B is thought to be because the pre-heated process causes the fish meat to lose water so that when dipped in liquid smoke, the fish fillets absorb a large amount of liquid smoke. Previous studies have reported that when the fish surface is dried, there is less smoke condensation than products smoked at lower temperatures. The results of this study indicate that a dry fish surface allows for better absorption of smoke components (Belichovska *et al*., 2019).

The study reported phenol levels in liquid smoke from corn cob waste of 0.335 mg/g (Swastawati *et al.*, 2007) and 2.55% (Leviyani *et al*., 2019). The phenol level of smoked fish with the conventional method of smoking with corn cobs is lower due to the high acid content, which can potentially reduce the phenol content of smoked fish (Swastawati *et al*., 2012). Anggraini & Nurhazisa (2017) reported that the phenol content of liquid smoke from coconut shells was 3.04%, while liquid smoke from corn cob was 1.38%.

Polycyclic aromatic hydrocarbon (PAH) levels

Polycyclic aromatic hydrocarbons are a group of organic compounds formed by incomplete combustion, such as wood. Therefore, the type of fuel used in the fish smoking process affects the PAH content of smoked fish (Jinadasa *et al.*, 2020). One of the carcinogenic PAHs, benzo(a)pyrene, is a carcinogenic

marker in smoked fish products (Stołyhwo & Sikorski, 2005). Polycyclic aromatic hydrocarbon (PAH) levels in smoked fish fillets can be seen in Table 2.

Table 2. Polycyclic aromatic hydrocarbon levels in *julung julung* smoked fillet

Polycyclic Aromatic Hydrocarbon	Smoked *Julung Julung* fish (µg/kg)					Corn Cob Liquid Smoke 0.8%(µg/kg)
	A	B	C	D	E	
Benzo(a)pyrene	nd	nd	nd	1.5	0.2	0.5
Benzo(b)fluoranthene	1.2	1.0	1.2	2	1.8	2.5
Benzo(a)anthracene	nd	nd	nd	0.5	nd	0.2
Benzo(g)perylene	0.4	0.2	0.4	1.0	0.8	1.0

nd = not detected

Table 2 shows that the PAH levels in smoked fish are still below the standard required in the Indonesian National Standard No. 2725 of 2013 on smoked fish, which is benzo(a)pyrene max 5 µg/kg (Indonesia Standardization Agency, 2013). The polycyclic aromatic hydrocarbon compound regulated in the Indonesian National Standard is only benzo(a)pyrene. Stołyhwo & Sikorski (2005) reported benzo(a)pyrene levels in smoked fish with hot and cold smoking processes ranging from 0.05 to about 60 µg/kg. Other studies have reported benzo(b,k)fluoranthene levels in salmon and rainbow trout ranging from 1.83 to 9.55 µg/kg, while benzo(g,h)perylene levels in salmon were 0.44 µg/kg (Basak *et al*., 2010). Berhimpon *et al*. (2018) also reported benzo(a)pyrene levels in smoked skipjack fish of 0.25 µg/kg. The level of benzo(a)pyrene in smoked barracuda was reported to be 0.32 µg/kg, benzo(b)fluoranthene 0.35 µg/kg, benzo(k)fluoranthene 0.21 µg/kg, benzo(a)anthracene 0.44 µg/kg, and benzo(g,h)perylene 2.56 µg/kg (Asamoah *et al*., 2021).

High PAH levels in smoked fish are due to the lignin content of the material used as fuel in the smoking process (Asamoah *et al*., 2021). The lignin content in corn cobs is reported to be about 17 – 21% (Thangavelu *et al*., 2018; Olajuyigbe *et al*., 2019; Gandam *et al*., 2022). The lignin content of coconut shell was approximately 59.5% (Wang & Sarkar, 2018), while that of coconut wood was about 50% on a dry weight basis (Anuchi *et al*., 2022). Lignin is reported to absorb PAH compounds because PAH compounds can bind well with lignin (Oliveira *et al*., 2019). During pyrolysis and gasification processes, lignin components react to produce aromatic tar and coke, which can form PAH compounds (Zhou *et al*., 2014; Kawamoto, 2017). Therefore, PAH compounds in smoked fish smoked using corncobs are lower than those smoked with shells and coconut wood.

Sensory assessment

A sensory assessment is carried out to evaluate the panelist's preference level, including appearance, aroma, taste, and texture. A sensory assessment must determine the quality of the smoked fish product and ensure it meets the applicable standards (Hadanu & Lomo, 2019). The sensory characteristics of

smoked fish products affect consumer acceptance and preference. Sensory assessment by panelists helps to identify the sensory attributes that are most appealing to consumers, allowing manufacturers to customize products according to consumer assessment (Ekelemu *et al*., 2021). Indonesian National Standard No. 2725:2013 regarding smoked fish requires a minimum sensory assessment of 7 (Score 1-9) (Indonesia Standardization Agency, 2013). The results of the panelists' assessment of *julung julung* smoked fillet from each treatment can be seen in Fig. 7.

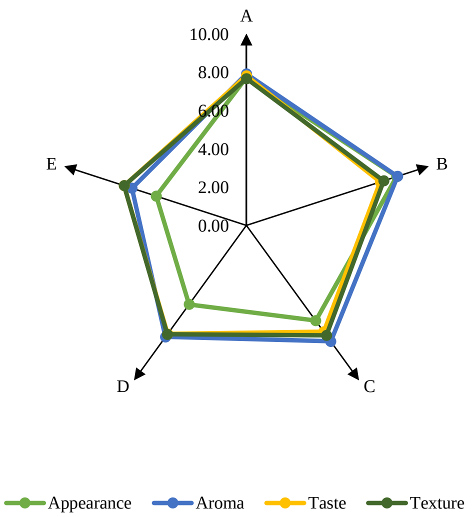

Fig. 7. Sensory assesment of *julung julung* smoked fillet

Fig. 7 shows the analysis of the variance of the sensory assessment of the appearance of smoked *julung julung* fillets with different smoking method treatments affecting the appearance of smoked fish ($p<0.05$). The panelists' assessment of the appearance of smoked fish fillets ranged from 4.95 to 8.24, with the highest panelists' assessment in treatment B. Based on the requirements of the Indonesian National Standard, only treatments A and B met the minimum panelist assessment requirement of 7. The moisture content factor is thought to have influenced the panelists' assessment of the appearance of smoked fish, so panelists less favored treatment C with steaming. Moisture content can affect the physical properties of fish, such as the appearance and texture of smoked fish (Baten *et al*., 2020a). Smoked fish with high moisture content will make the color of smoked fish look paler (Flick, 2010). Treatments D and E were less favored by panelists, presumably because conventional smoking methods produce darker products (dark brown), less bright and less shiny. Smoking fish with liquid smoke has smoked fish products with a golden color and clean and shiny surface (Berhimpon *et al*., 2018). Another

factor that affects the appearance of smoked fish is the length of the smoking process. The longer the smoking process, the darker the color will be. The time of the smoking process for smoked fish using the liquid smoke method is shorter than the conventional method (Puke & Galoburda, 2020; Baten *et al*., 2020b). The smoking process can change the color of fish, giving it a distinctive golden color due to the interaction of carbonyls with amino components on the surface of the meat. The color and appearance of smoked fish also positively correlate with phenol content. Phenol compounds in smoke interact with amino acid components in fish meat, producing a distinctive golden color in smoked fish (Montazeri *et al*., 2013).

Fig. 7 shows the analysis of the variance of the sensory assessment of the aroma of smoked fish fillets *julung julung* with different smoking method treatments that did not affect the appearance of smoked fish ($p>0.05$). However, the aroma sensory assessment of treatments A, B, C, and D still meets the minimum assessment of the Indonesian National Standard on smoked fish. The sensory assessment of smoked fish aroma was lowest in treatment E. Previous studies have reported that smoking fish with corn cobs as a fuel source resulted in the lowest aroma scores, indicating less aromatic smoked fish (Asmara *et al*., 2022). The aroma and taste of smoked fish are strongly influenced by the time of smoking (Tahir *et al*., 2020). This assessment indicates that the time of the smoking process of 4 and 6 hours in all treatments has not influenced the taste and aroma of smoked fish.

Fig. 7 shows the analysis of the variance of the sensory assessment of the taste of smoked fish fillets *julung julung* with different smoking method treatments that did not affect the appearance of smoked fish ($p>0.05$). The taste of smoked fish is influenced by the time of the smoking process. Previous studies have reported that longer smoking time can improve the sensory attributes of taste (Baten *et al*., 2020b). This result indicates that the 2-hour time difference between the liquid smoke smoking treatment and the conventional smoking method has not affected the taste of smoked fish. A distinctive smoked taste without bitterness is a criterion for assessing the taste of good quality smoked fish (Sukowati *et al*., 2021).

Fig. 7 shows the analysis of variance of the sensory assessment of the texture of smoked fish fillet *julung julung* with different smoking method treatments giving effect to the texture of smoked fish ($p<0.05$). Panelists' assessment of the texture of smoked fish *julung julung*, ranged from 6.71 – 7.67, with the highest panelist assessment in treatments A and B. The statistical analysis showed that treatments A and B differed from treatments C, D, and E. This result was thought to be because the fish fillets were dipped in liquid smoke, giving a dry, compact, and tender texture. Whereas in treatment C (fish fillets subjected to steaming), the texture of the smoked fillets was rather sticky and not solid. Treatments D and E produced the texture of smoked fish fillets which were less dense and not compact. The texture of smoked fish is negatively correlated with its moisture content. The higher the moisture content in

smoked fish, the softer and less firm the texture (Chan *et al*., 2022). High moisture content in smoked fish products results in less dense fish muscle fibers (Chang *et al*., 2021).

A good texture of smoked fish is compact, moderately elastic, and not sticky (Sulistijowati *et al*., 2021). The Indonesian National Standard requires that the texture of smoked fish is dense and compact and the tissue is very tight (Indonesia Standardization Agency, 2013). Based on consumer surveys, the preferred texture of smoked fish is dense, slightly wetter, and easy to chew, and there is a sensation of liquid in the mouth when chewed (Ticoalu *et al*., 2019).

Conclusion

Based on the evaluation of the parameters of total volatile bases, moisture content, water activity, pH, phenol content, polycyclic aromatic hydrocarbon content, and sensory assessment of smoked *julung julung* fillets, it can be concluded that treatment B is the best treatment, i.e. the fillets were preheated for 4 hours at 60 – 80 °C, then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated for 4 hours at 90 °C. In general, the characteristics of smoked fillets using liquid smoke were better when compared to the conventional smoking treatment. It is necessary to evaluate different smoking times on smoked *julung julung* fillets with corn cob liquid smoke method.

Acknowledgment

Author contributions

Conflicts of interest

The authors declare that there is no conflict of interest.

Highlights

Effectiveness of using liquid smoke derived from corn cob waste in the smoking process of *julung julung* fillets

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Authors: Jefri Anthonius Mandeno, Wendy Alexander Tanod, Eko Cahyono, Yana Sammbeka, Frets Jonas Rieuwpassa, Novalina I
Frianto Putra Palawe, Putut Har Riyadi

Dear Mr. Jefri Anthonius Mandeno

It is of great pleasure and delight to inform that your manuscript entitled **Characteristics of Julung Julung Smoked Fillets (Hem
Liquid Smoke from Corn Cobs Waste**has been published online via **Research and Innovation in Food Science and Technolo**

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Yours sincerely,

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