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

42 The conventional smoking processes generally use hardwood as fuel, such as melon wood (Umar *et al.*,  
43 2018), mangrove wood (Cissoko *et al.*, 2020), teak wood (Daramola *et al.*, 2020), coconut shells, and  
44 coconut husks (Nugroho *et al.*, 2018). Corn cobs have also been reported to be used in the fish smoking  
45 process (Sukowati, 2023). According to literature, traditional fish smoking in North Sulawesi,  
46 Indonesia, uses smoke from burning coconut shells or wood charcoal (Landangkasiang *et al.*, 2017;  
47 Primalasari *et al.*, 2019; Saediman *et al.*, 2021). Smoked fish processing has been known for a long time  
48 because of its traditional processing techniques, simplicity, ease of implementation, and low cost  
49 (Islamiyah, 2021). In North Sulawesi, Indonesia, smoked fish commonly known as *fufu* is traditionally  
50 made from skipjack (*Katsuwonus pelamis*); *julung julung* (*Hemiramphus far*) (Berhimpon *et al.*, 2018);  
51 and scad fish (*Decapterus* spp.) known as *pinekuhe* (Ansar & Ijong, 2021). The smoked fish product of  
52 *julung julung* is a typical product from North Sulawesi and Gorontalo, commonly called *roa* or *galavea*  
53 (Azis & Akolo, 2020).

54 The study's reported that the conventional smoking process has disadvantages such as smoking time,  
55 concentration of carcinogenic substances, temperature, and inconsistent product quality (Racovita *et al.*,  
56 2020). Conventional smoking of food products has been shown to produce carcinogenic components  
57 such as polycyclic aromatic hydrocarbons (Jinadasa *et al.*, 2020). Benzo(a)pyrene (C<sub>20</sub>H<sub>12</sub>) is one of the  
58 carcinogenic compounds produced from traditional smoking products (Jinadasa *et al.*, 2020). One  
59 method to reduce carcinogenic compounds in smoked fish products is to use liquid smoke in the  
60 smoking process (Nithin *et al.*, 2020; Xin *et al.*, 2021).

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

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

72 smoked fish requires a maximum total plate count of  $5 \times 10^4$  CFU/g and a maximum moisture content of  
73 60% (Indonesia Standardization Agency, 2013). Another study reported that smoked fish processed  
74 with liquid smoke had a moisture content of 47.63%, phenol content of 12.62%, and pH of 4.8  
75 (Berhimpon *et al.*, 2018). The results of these studies indicate that liquid smoke is good to apply to  
76 smoked fish products. This study characterized smoked fish fillets of *julung julung* (*Hemiramphus* sp.)  
77 produced with liquid smoke from corn cobs waste. The study was conducted by dipping the fish fillets  
78 of *julung julung* with liquid smoke, with drying and steaming treatments on the fish fillets. This study  
79 aims to obtain the best smoking method for fish fillets using liquid smoke from corn cobs waste based  
80 on the assessment of total volatile bases, moisture content, water activity, pH value, phenol content,  
81 polycyclic aromatic hydrocarbon content, and sensory assessment.

82

### 83 **Materials and methods**

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

92



93

94

95

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

96 The treatment in this study was applying liquid smoke from corn cobs waste to the smoking process  
97 of *julung julung* fish fillets, which was compared with the conventional method (using coconut wood,  
98 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.

99

#### 100 **Liquid smoke manufacturing process**

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

109

#### 110 **Total volatile bases assay**

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

118 
$$TVB \left( \text{mg} \frac{\text{N}}{100\text{g}} \right) = \frac{(V_1 - V_0) \times N.HCl \times dilution \times 100}{sample\ weight\ (g)}$$

119

120 **Moisture content assay**

121 The moisture content by method of Indonesia National Standard (Indonesia Standardization Agency,  
122 2006a). An empty porcelain cup is weighed (A). Then, the sample was weighed to  $\pm 2$  g in a porcelain  
123 cup of known mass (B). The samples were dried in an oven at  $105^{\circ}\text{C}$  for 20 hours. The samples were  
124 then cooled in a desiccator, after which the samples in the cup were weighed (C). Moisture content was  
125 expressed as %.

126 
$$\text{Moisture (\%)} = \frac{B (g) - C (g)}{B(g) - A (g)} \times 100\%$$

127

128 **Water activity (Aw) assay**

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

134

135 **pH assay**

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

140

141 **Phenol level assay**

142 Samples were extracted by adding 1 mL ethanol p.a., 5 mL distilled water, and 0.5 mL Folin Ciocalteu  
143 50%, homogenized, and allowed to stand for 5 minutes. Then added, 1 mL of 5%  $\text{Na}_2\text{CO}_3$  and left in  
144 the dark for  $\pm 60$  minutes with gallic acid as a standard. The absorbance value was read on a  
145 spectrophotometer (Thermo Scientific Genesys 50) with a wavelength of 725 nm. Total phenolics were  
146 expressed as mg/g (Indonesia Standardization Agency, 2004; Muliadin *et al.*, 2022).

147 
$$\text{Total Phenol } \left(\frac{\text{mg}}{\text{g}}\right) = \frac{\text{Gallic acid equivalence } \left(\frac{\text{mg}}{\text{L}}\right) \times \text{Volume (L)}}{\text{Sample weight (g)}}$$

148

149 **Polycyclic aromatic hydrocarbon (PAH) assay**

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

152 of internal standard solution (9,10 dimethylantracene), and some boiling stones were added. After that,  
153 the mixture was boiled for 4 hours in a soxhlet. The liquid phase was transferred to a separatory funnel  
154 and extracted with 100 mL of n-hexane with shaking for 3 min. The MeOH:KOH phase was drained  
155 and discarded. The combined n-hexane phase was rinsed twice, each with 50 mL of H<sub>2</sub>O-MeOH (8:1)  
156 and 50 mL of water. The methanol-water and water phases were dried and discarded. The organic phase  
157 was concentrated in a rotary evaporator (40°C) to a volume of about 10 mL. The concentrated extract  
158 was dried under nitrogen gas.

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

169

### 170 Sensory assessment assay

171 Sensory assessment assay refers to Indonesia National Standard (Indonesia Standardization Agency,  
172 2006b). The sensory assessment was based on a hedonic scale (Table 1). Smoked fish samples were  
173 placed on a plastic plate with a glass of water, coded, and presented to 30 panelists randomly under  
174 light. The parameters observed were appearance, aroma, taste, and texture. Panelists rated acceptance  
175 of the samples on a scale of 1 – 9.

176

177

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

178

179 **Data analysis**

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

184 **Results and discussion**

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

192 **Total volatile bases**

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

201

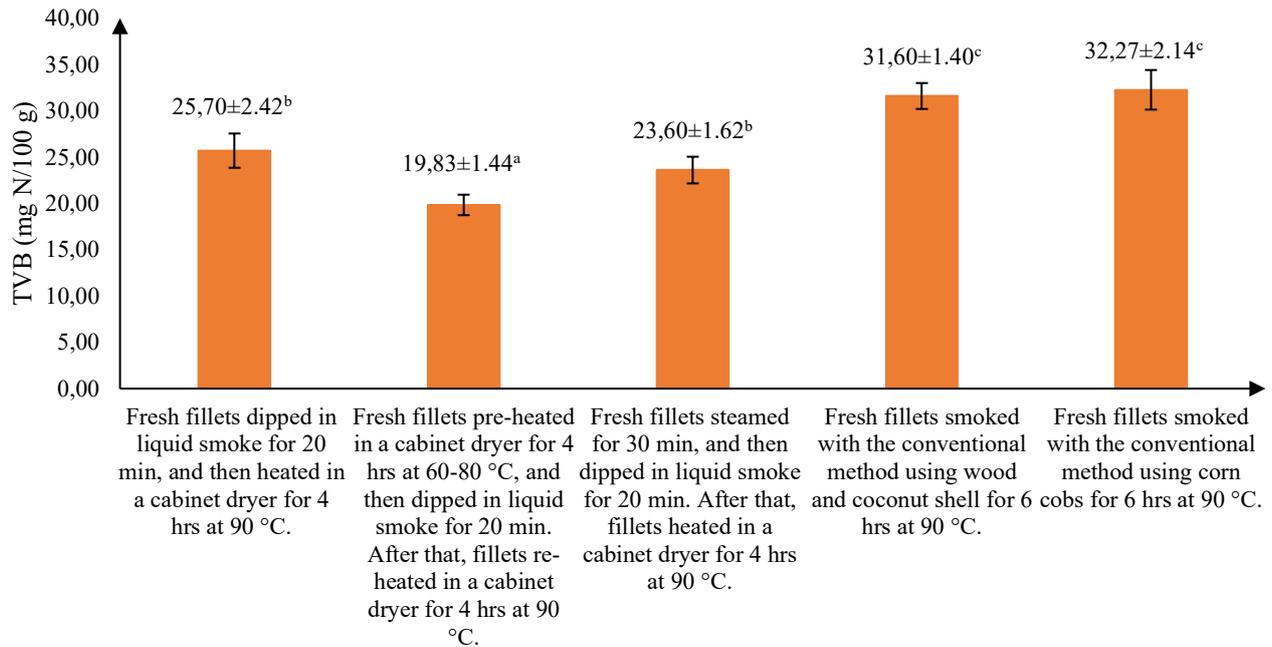


Fig. 2. Total volatile bases of *julung julung* smoked fillet

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).

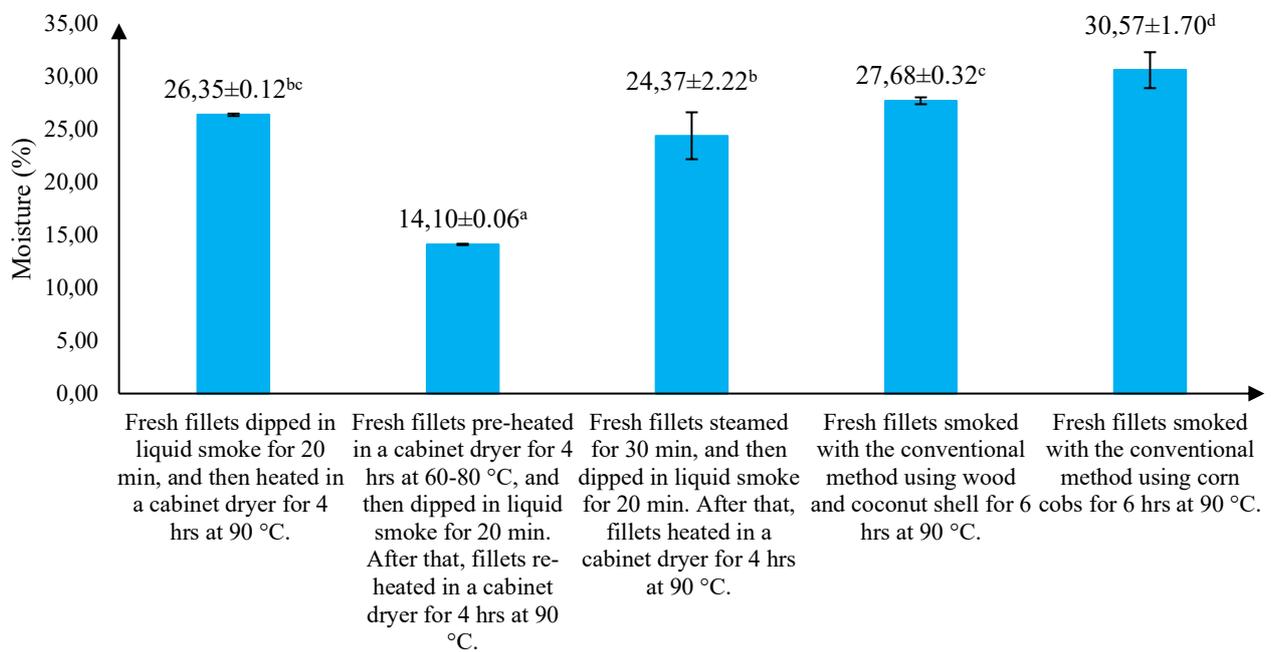
224 The European standard (European Market Observatory for Fisheries and Aquaculture, 2020) states that  
 225 the limit range of TVB value for smoked fish is 25 – 35 mg N/100 g. All treatments' TVB value of the  
 226 smoked fish fillets still met the European standard. Previous studies reported TVB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

233

234 **Moisture content**

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

240



241

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

243

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

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

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

267

#### 268 **Water activity (Aw)**

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

278

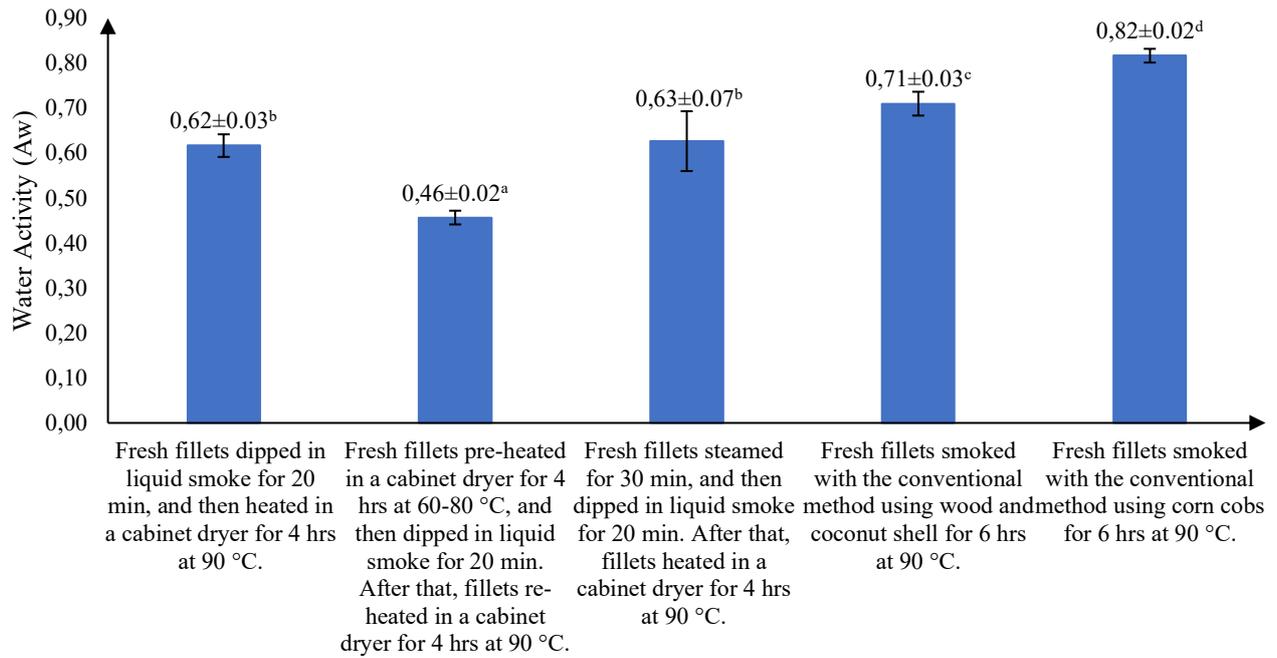


Fig. 4. Water activity of *julung julung* smoked fillet

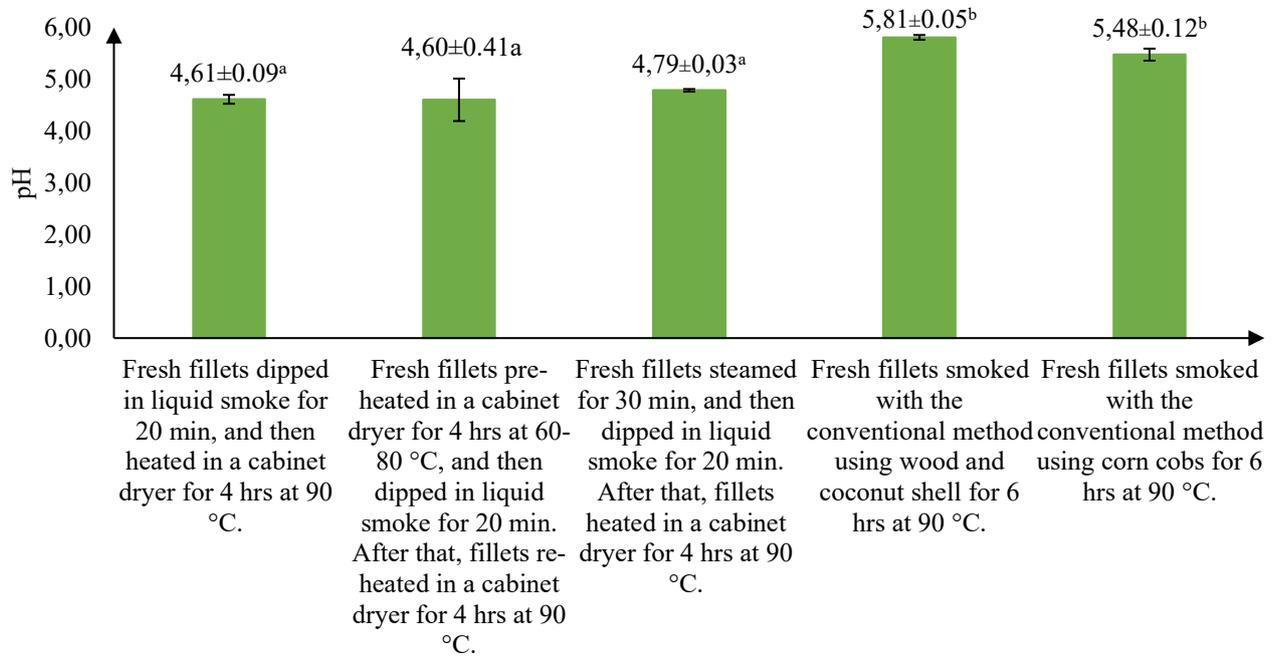
Fig. 4 shows the analysis of the variance of smoked fish fillet *julung julung* with different smoking method treatments affecting water activity ( $p < 0.05$ ). The water activity value of smoked fish fillet ranged from 0.46 – 0.82, with the lowest water activity in treatment B. The treatment of the 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

302 method. Another study also reported that using liquid smoke can reduce pH caused by the condensation  
 303 of organic acids in the smoking process (Puke & Galoburda, 2020). The pH value of smoked fish  
 304 correlates with the phenol content contained in the smoke component, and an increase in phenol content  
 305 causes a decrease in pH (Berhimpon *et al.*, 2018).  
 306



307

308

309

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

310 **Phenol level**

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

320

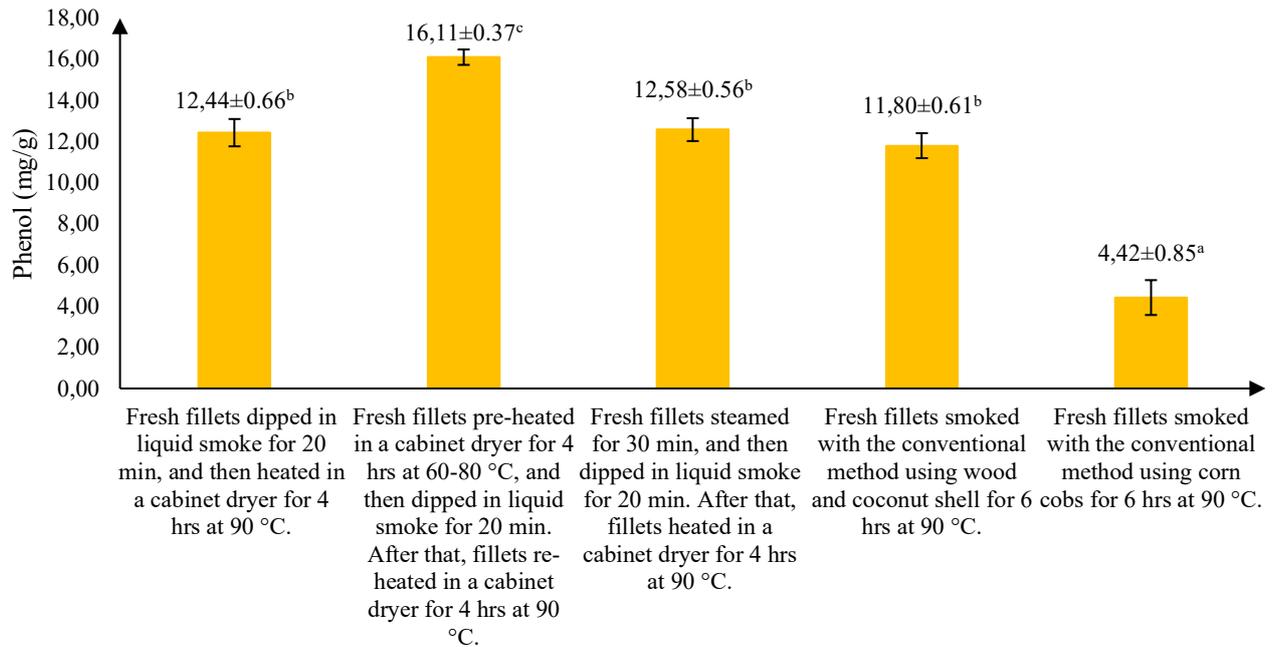


Fig. 6. Phenol level of *julung julung* smoked fillet

321

322

323

324 Duncan's analysis showed phenol levels in treatments A, C, and D were not different. Previous research  
 325 reported that the drying treatment of fish meat can increase phenolic compounds in smoked fish products  
 326 (Sérot *et al.*, 2004). The high phenol content in treatment B is thought to be because the pre-heated  
 327 process causes the fish meat to lose water so that when dipped in liquid smoke, the fish fillets absorb a  
 328 large amount of liquid smoke. Previous studies have reported that when the fish surface is dried, there  
 329 is less smoke condensation than products smoked at lower temperatures. The results of this study  
 330 indicate that a dry fish surface allows for better absorption of smoke components (Belichovska *et al.*,  
 331 2019).

332 The study reported phenol levels in liquid smoke from corn cob waste of 0.335 mg/g (Swastawati *et al.*,  
 333 2007) and 2.55% (Leviyani *et al.*, 2019). The phenol level of smoked fish with the conventional method  
 334 of smoking with corn cobs is lower due to the high acid content, which can potentially reduce the phenol  
 335 content of smoked fish (Swastawati *et al.*, 2012). Anggraini & Nurhazisa (2017) reported that the phenol  
 336 content of liquid smoke from coconut shells was 3.04%, while liquid smoke from corn cob was 1.38%.

337

338 **Polycyclic aromatic hydrocarbon (PAH) levels**

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

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

Polycyclic Aromatic Hydrocarbon	Smoked <i>Julung Julung</i> 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

345 nd = not detected

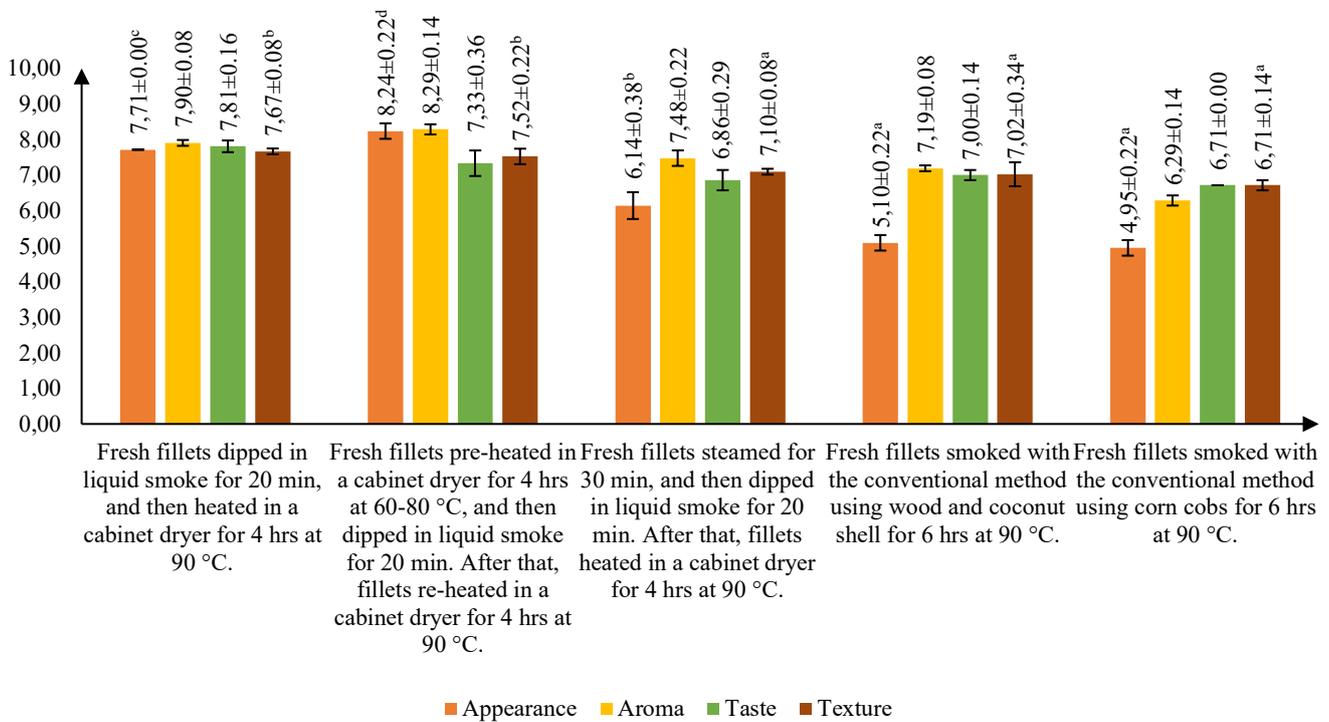
346  
 347 Table 2 shows that the PAH levels in smoked fish are still below the standard required in the Indonesian  
 348 National Standard No. 2725 of 2013 on smoked fish, which is benzo(a)pyrene max 5 µg/kg (Indonesia  
 349 Standardization Agency, 2013). The polycyclic aromatic hydrocarbon compound regulated in the  
 350 Indonesian National Standard is only benzo(a)pyrene. Stołyhwo & Sikorski (2005) reported  
 351 benzo(a)pyrene levels in smoked fish with hot and cold smoking processes ranging from 0.05 to about  
 352 60 µg/kg. Other studies have reported benzo(b,k)fluoranthene levels in salmon and rainbow trout  
 353 ranging from 1.83 to 9.55 µg/kg, while benzo(g,h)perylene levels in salmon were 0.44 µg/kg (Basak *et*  
 354 *al.*, 2010). Berhimon *et al.* (2018) also reported benzo(a)pyrene levels in smoked skipjack fish of 0.25  
 355 µg/kg. The level of benzo(a)pyrene in smoked barracuda was reported to be 0.32 µg/kg,  
 356 benzo(b)fluoranthene 0.35 µg/kg, benzo(k)fluoranthene 0.21 µg/kg, benzo(a)anthracene 0.44 µg/kg,  
 357 and benzo(g,h)perylene 2.56 µg/kg (Asamoah *et al.*, 2021).

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

367  
 368 **Sensory assessment**

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

374 customize products according to consumer assessment (Ekelemu *et al.*, 2021). Indonesian National  
 375 Standard No. 2725:2013 regarding smoked fish requires a minimum sensory assessment of 7 (Score 1-  
 376 9) (Indonesia Standardization Agency, 2013). The results of the panelists' assessment of *julung julung*  
 377 smoked fillet from each treatment can be seen in Fig. 7.  
 378



379 **Fig. 7.** Sensory assesment of *julung julung* smoked fillet  
 380  
 381

382 **Appearance:** Fig. 7 shows the analysis of the variance of the sensory assessment of the appearance of  
 383 smoked *julung julung* fish fillets with different smoking method treatments affecting the appearance of  
 384 smoked fish ( $p < 0.05$ ). The panelists' assessment of the appearance of smoked fish fillets ranged from  
 385 4.95 to 8.24, with the highest panelists' assessment in treatment B. Based on the requirements of the  
 386 Indonesian National Standard, only treatments A and B met the minimum panelist assessment  
 387 requirement of 7. The moisture content factor is thought to have influenced the panelists' assessment of  
 388 the appearance of smoked fish, so panelists less favored treatment C with steaming. Moisture content  
 389 can affect the physical properties of fish, such as the appearance and texture of smoked fish (Baten *et al.*,  
 390 2020a). Smoked fish with high moisture content will make the color of smoked fish look paler  
 391 (Flick, 2010).  
 392 Treatments D and E were less favored by panelists, presumably because conventional smoking methods  
 393 produce darker products (dark brown), less bright and less shiny. Smoking fish with liquid smoke has  
 394 smoked fish products with a golden color and clean and shiny surface (Berhimpon *et al.*, 2018). Another  
 395 factor that affects the appearance of smoked fish is the length of the smoking process. The longer the

396 smoking process, the darker the color will be. The time of the smoking process for smoked fish using  
397 the liquid smoke method is shorter than the conventional method (Puke & Galoburda, 2020; Baten *et al.*,  
398 2020b). The smoking process can change the color of fish, giving it a distinctive golden color due  
399 to the interaction of carbonyls with amino components on the surface of the meat. The color and  
400 appearance of smoked fish also positively correlate with phenol content. Phenol compounds in smoke  
401 interact with amino acid components in fish meat, producing a distinctive golden color in smoked fish  
402 (Montazeri *et al.*, 2013).

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

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

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

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

436

#### 437 **Conclusion**

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

446

#### 447 **Acknowledgement (bold)**

#### 448 **Author contributions**

#### 449 **Conflicts of interest**

450 The authors declare that there is no conflict of interest.

451

452

#### 453 **Highlights**

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

456

457

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## **2. Bukti manuskrip perlu direvisi**



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

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

42 The conventional smoking processes generally use hardwood as fuel, such as melon wood (Umar *et al.*,  
43 2018), mangrove wood (Cissoko *et al.*, 2020), teak wood (Daramola *et al.*, 2020), coconut shells, and  
44 coconut husks (Nugroho *et al.*, 2018). Corn cobs have also been reported to be used in the fish smoking  
45 process (Sukowati, 2023). According to literature, traditional fish smoking in North Sulawesi,  
46 Indonesia, uses smoke from burning coconut shells or wood charcoal (Landangkasiang *et al.*, 2017;  
47 Primalasari *et al.*, 2019; Saediman *et al.*, 2021). Smoked fish processing has been known for a long time  
48 because of its traditional processing techniques, simplicity, ease of implementation, and low cost  
49 (Islamiyah, 2021). In North Sulawesi, Indonesia, smoked fish commonly known as *fufu* is traditionally  
50 made from skipjack (*Katsuwonus pelamis*); *julung julung* (*Hemiramphus far*) (Berhimpon *et al.*, 2018);  
51 and scad fish (*Decapterus* spp.) known as *pinekuhe* (Ansar & Ijong, 2021). The smoked fish product of  
52 *julung julung* is a typical product from North Sulawesi and Gorontalo, commonly called *roa* or *galavea*  
53 (Azis & Akolo, 2020).

54 The study's reported that the conventional smoking process has disadvantages such as smoking time,  
55 concentration of carcinogenic substances, temperature, and inconsistent product quality (Racovita *et al.*,  
56 2020). Conventional smoking of food products has been shown to produce carcinogenic components  
57 such as polycyclic aromatic hydrocarbons (Jinadasa *et al.*, 2020). Benzo(a)pyrene (C<sub>20</sub>H<sub>12</sub>) is one of the  
58 carcinogenic compounds produced from traditional smoking products (Jinadasa *et al.*, 2020). One  
59 method to reduce carcinogenic compounds in smoked fish products is to use liquid smoke in the  
60 smoking process (Nithin *et al.*, 2020; Xin *et al.*, 2021).

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

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

72 smoked fish requires a maximum total plate count of  $5 \times 10^4$  CFU/g and a maximum moisture content of  
73 60% (Indonesia Standardization Agency, 2013). Another study reported that smoked fish processed  
74 with liquid smoke had a moisture content of 47.63%, phenol content of 12.62%, and pH of 4.8  
75 (Berhimpon *et al.*, 2018). The results of these studies indicate that liquid smoke is good to apply to  
76 smoked fish products. This study characterized smoked fish fillets of *julung julung* (*Hemiramphus* sp.)  
77 produced with liquid smoke from corn cobs waste. The study was conducted by dipping the fish fillets  
78 of *julung julung* with liquid smoke, with drying and steaming treatments on the fish fillets. This study  
79 aims to obtain the best smoking method for fish fillets using liquid smoke from corn cobs waste based  
80 on the assessment of total volatile bases, moisture content, water activity, pH value, phenol content,  
81 polycyclic aromatic hydrocarbon content, and sensory assessment.

82

### 83 **Materials and methods**

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

92



93

94

95

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

96 The treatment in this study was applying liquid smoke from corn cobs waste to the smoking process  
97 of *julung julung* fish fillets, which was compared with the conventional method (using coconut wood,  
98 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.

99

#### 100 **Liquid smoke manufacturing process**

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

109

#### 110 **Total volatile bases assay**

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

$$118 \quad TVB \left( \text{mg} \frac{\text{N}}{100\text{g}} \right) = \frac{(V_1 - V_0) \times N.HCl \times dilution \times 100}{sample\ weight\ (g)}$$

119

120 **Moisture content assay**

121 The moisture content by method of Indonesia National Standard (Indonesia Standardization Agency,  
122 2006a). An empty porcelain cup is weighed (A). Then, the sample was weighed to  $\pm 2$  g in a porcelain  
123 cup of known mass (B). The samples were dried in an oven at  $105^{\circ}\text{C}$  for 20 hours. The samples were  
124 then cooled in a desiccator, after which the samples in the cup were weighed (C). Moisture content was  
125 expressed as %.

126 
$$\text{Moisture (\%)} = \frac{B (g) - C (g)}{B(g) - A (g)} \times 100\%$$

127

128 **Water activity (Aw) assay**

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

134

135 **pH assay**

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

140

141 **Phenol level assay**

142 Samples were extracted by adding 1 mL ethanol p.a., 5 mL distilled water, and 0.5 mL Folin Ciocalteu  
143 50%, homogenized, and allowed to stand for 5 minutes. Then added, 1 mL of 5%  $\text{Na}_2\text{CO}_3$  and left in  
144 the dark for  $\pm 60$  minutes with gallic acid as a standard. The absorbance value was read on a  
145 spectrophotometer (Thermo Scientific Genesys 50) with a wavelength of 725 nm. Total phenolics were  
146 expressed as mg/g (Indonesia Standardization Agency, 2004; Muliadin *et al.*, 2022).

147 
$$\text{Total Phenol } \left(\frac{\text{mg}}{\text{g}}\right) = \frac{\text{Gallic acid equivalence } \left(\frac{\text{mg}}{\text{L}}\right) \times \text{Volume (L)}}{\text{Sample weight (g)}}$$

148

149 **Polycyclic aromatic hydrocarbon (PAH) assay**

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

152 of internal standard solution (9,10 dimethylantracene), and some boiling stones were added. After that,  
153 the mixture was boiled for 4 hours in a soxhelet. The liquid phase was transferred to a separatory funnel  
154 and extracted with 100 mL of n-hexane with shaking for 3 min. The MeOH:KOH phase was drained  
155 and discarded. The combined n-hexane phase was rinsed twice, each with 50 mL of H<sub>2</sub>O-MeOH (8:1)  
156 and 50 mL of water. The methanol-water and water phases were dried and discarded. The organic phase  
157 was concentrated in a rotary evaporator (40°C) to a volume of about 10 mL. The concentrated extract  
158 was dried under nitrogen gas.

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

169  
170 **Sensory assessment assay**

171 Sensory assessment assay refers to Indonesia National Standard (Indonesia Standardization Agency,  
172 2006b). The sensory assessment was based on a hedonic scale (Table 1). Smoked fish samples were  
173 placed on a plastic plate with a glass of water, coded, and presented to 30 panelists randomly under  
174 light. The parameters observed were appearance, aroma, taste, and texture. Panelists rated acceptance  
175 of the samples on a scale of 1 – 9.

176  
177 **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

179 **Data analysis**

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

184 **Results and discussion**

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

192 **Total volatile bases**

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

201

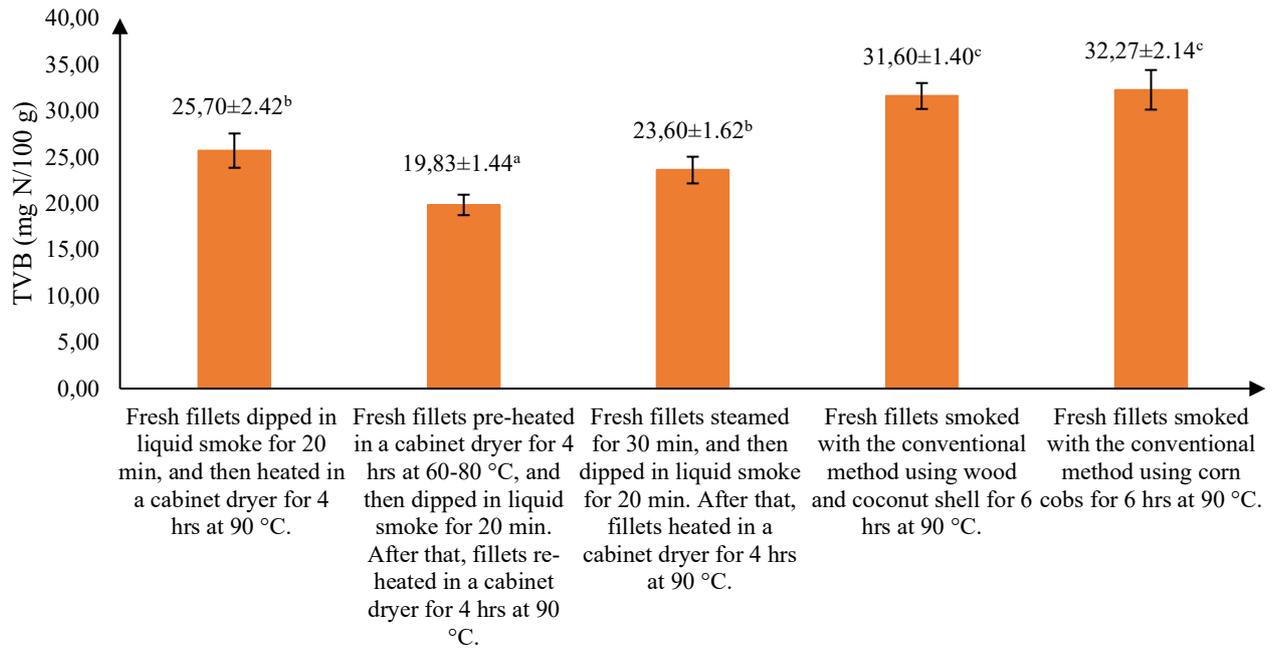


Fig. 2. Total volatile bases of *julung julung* smoked fillet

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).

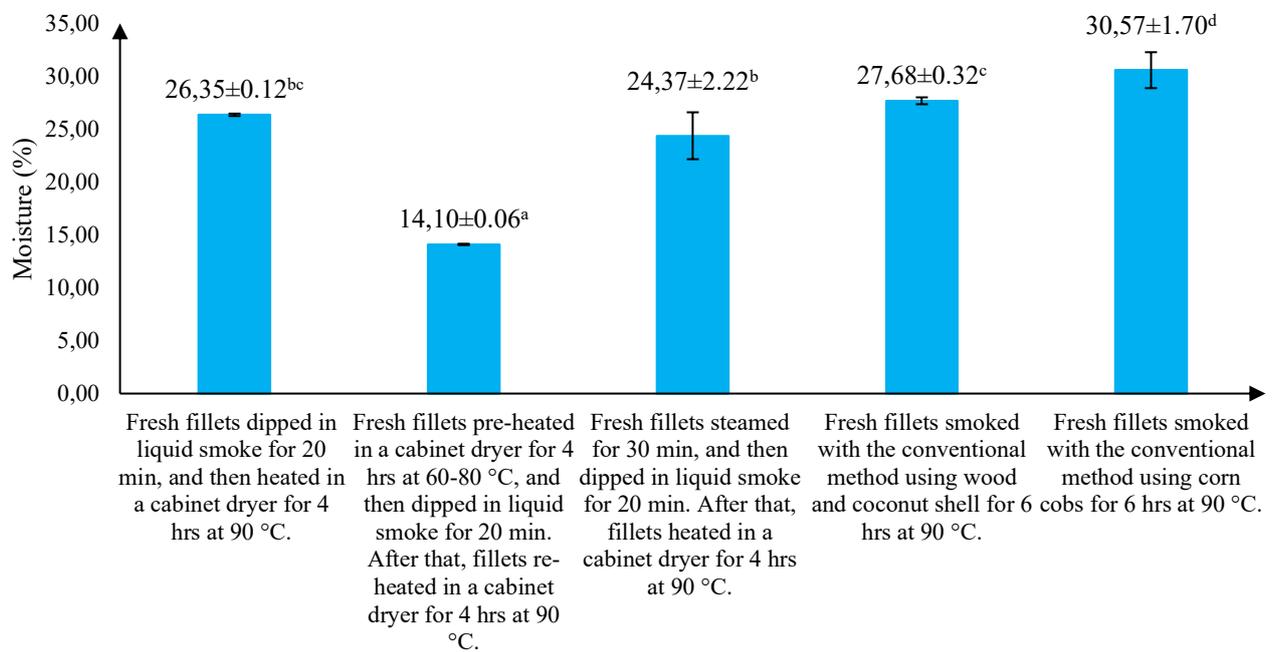
224 The European standard (European Market Observatory for Fisheries and Aquaculture, 2020) states that  
 225 the limit range of TVB value for smoked fish is 25 – 35 mg N/100 g. All treatments' TVB value of the  
 226 smoked fish fillets still met the European standard. Previous studies reported TVB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

233

234 **Moisture content**

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

240



241

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

243

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

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

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

267

### 268 **Water activity (Aw)**

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

278

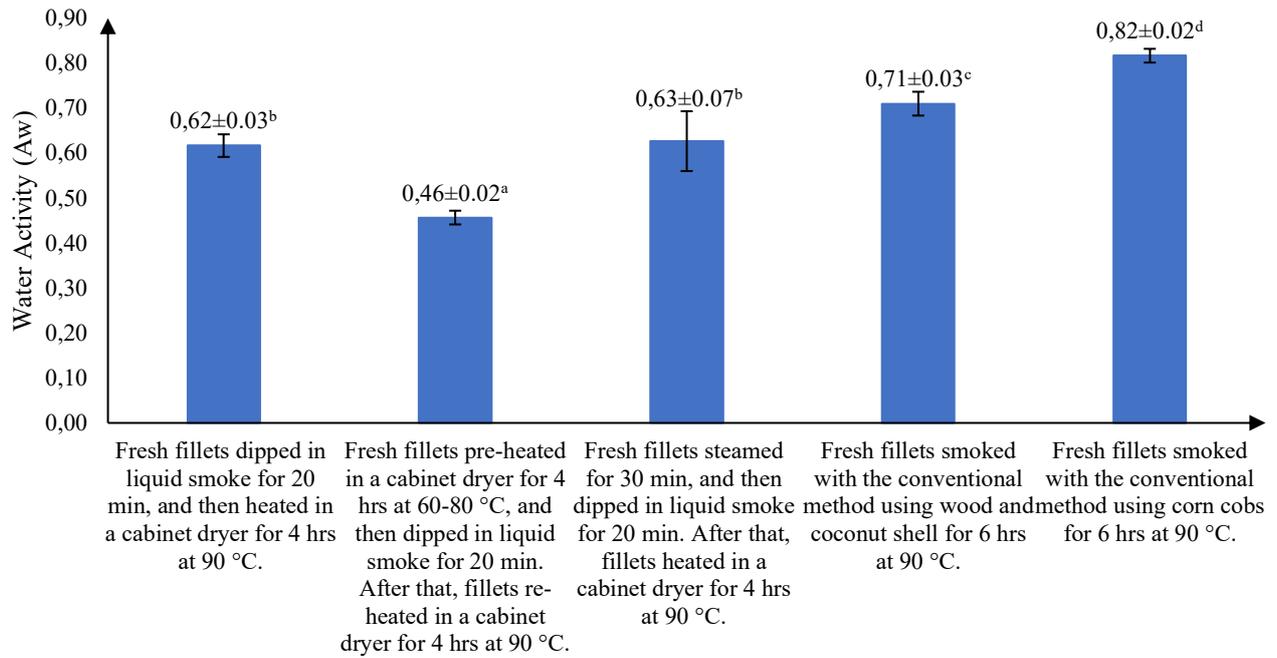


Fig. 4. Water activity of *julung julung* smoked fillet

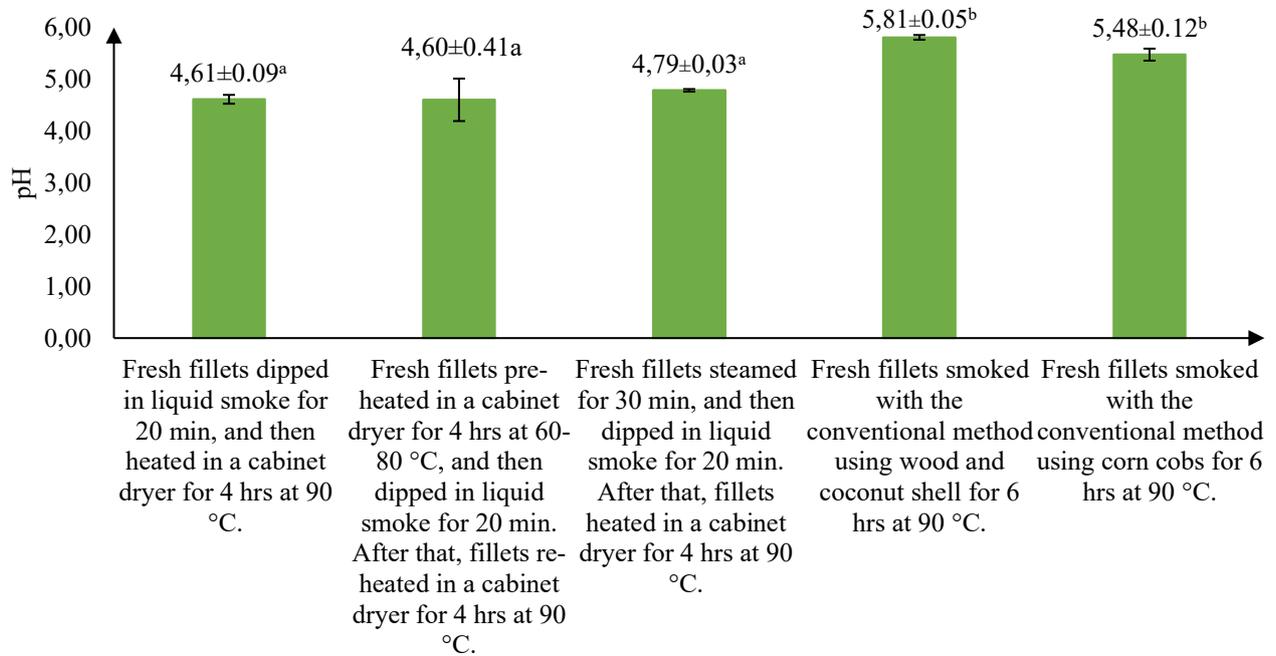
Fig. 4 shows the analysis of the variance of smoked fish fillet *julung julung* with different smoking method treatments affecting water activity ( $p < 0.05$ ). The water activity value of smoked fish fillet ranged from 0.46 – 0.82, with the lowest water activity in treatment B. The treatment of the 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

302 method. Another study also reported that using liquid smoke can reduce pH caused by the condensation  
 303 of organic acids in the smoking process (Puke & Galoburda, 2020). The pH value of smoked fish  
 304 correlates with the phenol content contained in the smoke component, and an increase in phenol content  
 305 causes a decrease in pH (Berhimpon *et al.*, 2018).  
 306



307

308

309

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

310 **Phenol level**

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

320

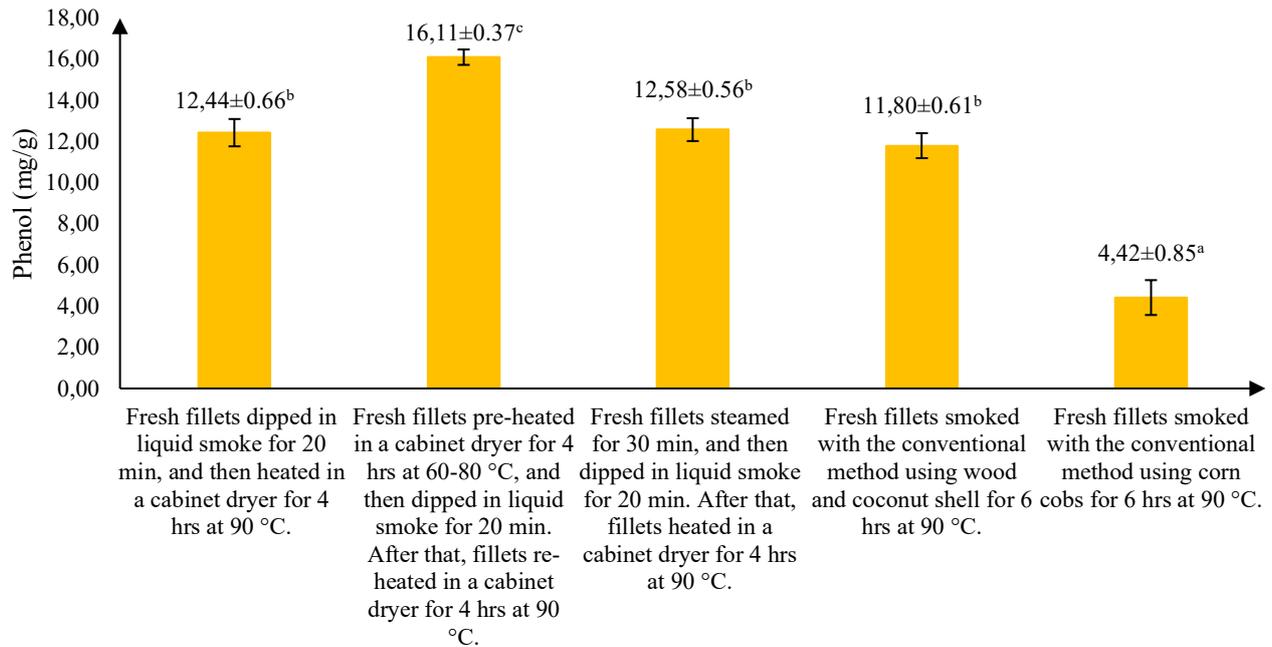


Fig. 6. Phenol level of *julung julung* smoked fillet

321

322

323

324 Duncan's analysis showed phenol levels in treatments A, C, and D were not different. Previous research  
 325 reported that the drying treatment of fish meat can increase phenolic compounds in smoked fish products  
 326 (Sérot *et al.*, 2004). The high phenol content in treatment B is thought to be because the pre-heated  
 327 process causes the fish meat to lose water so that when dipped in liquid smoke, the fish fillets absorb a  
 328 large amount of liquid smoke. Previous studies have reported that when the fish surface is dried, there  
 329 is less smoke condensation than products smoked at lower temperatures. The results of this study  
 330 indicate that a dry fish surface allows for better absorption of smoke components (Belichovska *et al.*,  
 331 2019).

332 The study reported phenol levels in liquid smoke from corn cob waste of 0.335 mg/g (Swastawati *et al.*,  
 333 2007) and 2.55% (Leviyani *et al.*, 2019). The phenol level of smoked fish with the conventional method  
 334 of smoking with corn cobs is lower due to the high acid content, which can potentially reduce the phenol  
 335 content of smoked fish (Swastawati *et al.*, 2012). Anggraini & Nurhazisa (2017) reported that the phenol  
 336 content of liquid smoke from coconut shells was 3.04%, while liquid smoke from corn cob was 1.38%.

337

338 **Polycyclic aromatic hydrocarbon (PAH) levels**

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

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

Polycyclic Aromatic Hydrocarbon	Smoked <i>Julung Julung</i> 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

345 nd = not detected

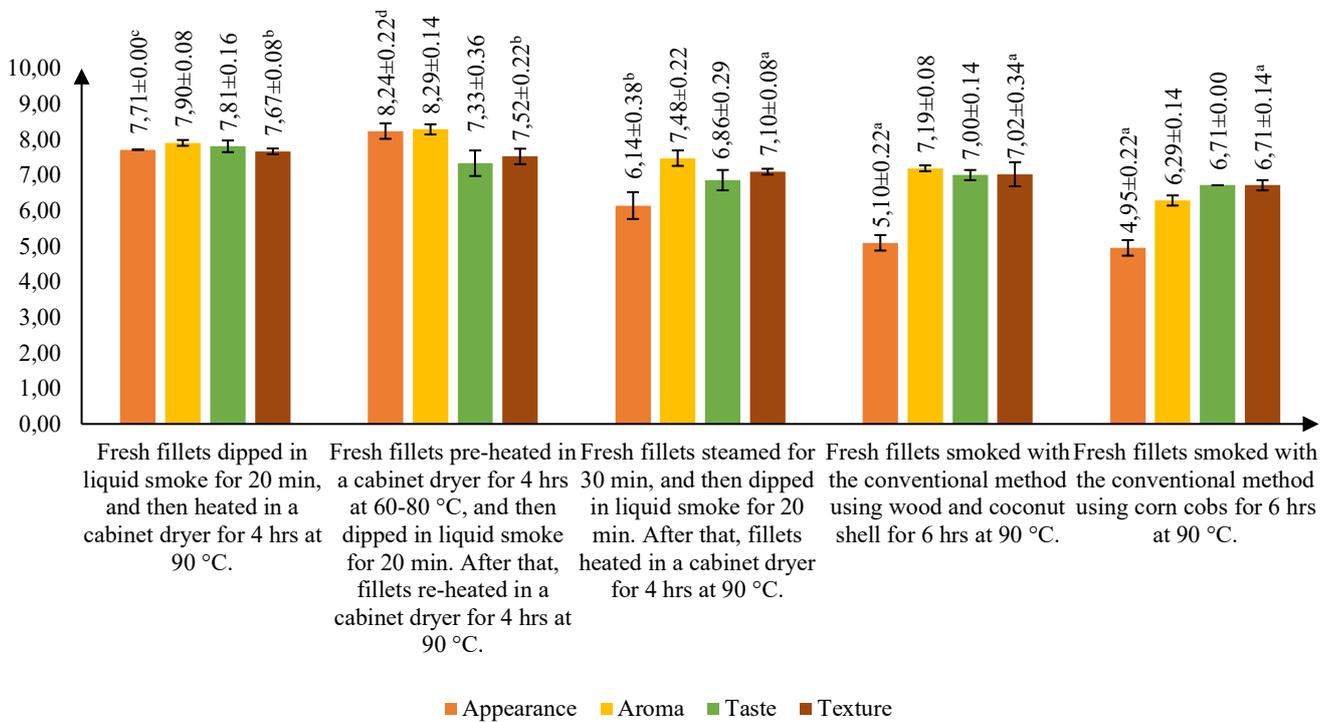
346  
 347 Table 2 shows that the PAH levels in smoked fish are still below the standard required in the Indonesian  
 348 National Standard No. 2725 of 2013 on smoked fish, which is benzo(a)pyrene max 5 µg/kg (Indonesia  
 349 Standardization Agency, 2013). The polycyclic aromatic hydrocarbon compound regulated in the  
 350 Indonesian National Standard is only benzo(a)pyrene. Stołyhwo & Sikorski (2005) reported  
 351 benzo(a)pyrene levels in smoked fish with hot and cold smoking processes ranging from 0.05 to about  
 352 60 µg/kg. Other studies have reported benzo(b,k)fluoranthene levels in salmon and rainbow trout  
 353 ranging from 1.83 to 9.55 µg/kg, while benzo(g,h)perylene levels in salmon were 0.44 µg/kg (Basak *et*  
 354 *al.*, 2010). Berhimpion *et al.* (2018) also reported benzo(a)pyrene levels in smoked skipjack fish of 0.25  
 355 µg/kg. The level of benzo(a)pyrene in smoked barracuda was reported to be 0.32 µg/kg,  
 356 benzo(b)fluoranthene 0.35 µg/kg, benzo(k)fluoranthene 0.21 µg/kg, benzo(a)anthracene 0.44 µg/kg,  
 357 and benzo(g,h)perylene 2.56 µg/kg (Asamoah *et al.*, 2021).

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

367  
 368 **Sensory assessment**

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

374 customize products according to consumer assessment (Ekelemu *et al.*, 2021). Indonesian National  
 375 Standard No. 2725:2013 regarding smoked fish requires a minimum sensory assessment of 7 (Score 1-  
 376 9) (Indonesia Standardization Agency, 2013). The results of the panelists' assessment of *julung julung*  
 377 smoked fillet from each treatment can be seen in Fig. 7.  
 378



379 **Fig. 7.** Sensory assesment of *julung julung* smoked fillet  
 380  
 381

382 **Appearance:** Fig. 7 shows the analysis of the variance of the sensory assessment of the appearance of  
 383 smoked *julung julung* fish fillets with different smoking method treatments affecting the appearance of  
 384 smoked fish ( $p < 0.05$ ). The panelists' assessment of the appearance of smoked fish fillets ranged from  
 385 4.95 to 8.24, with the highest panelists' assessment in treatment B. Based on the requirements of the  
 386 Indonesian National Standard, only treatments A and B met the minimum panelist assessment  
 387 requirement of 7. The moisture content factor is thought to have influenced the panelists' assessment of  
 388 the appearance of smoked fish, so panelists less favored treatment C with steaming. Moisture content  
 389 can affect the physical properties of fish, such as the appearance and texture of smoked fish (Baten *et al.*,  
 390 2020a). Smoked fish with high moisture content will make the color of smoked fish look paler  
 391 (Flick, 2010).

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

396 smoking process, the darker the color will be. The time of the smoking process for smoked fish using  
397 the liquid smoke method is shorter than the conventional method (Puke & Galoburda, 2020; Baten *et al.*,  
398 2020b). The smoking process can change the color of fish, giving it a distinctive golden color due  
399 to the interaction of carbonyls with amino components on the surface of the meat. The color and  
400 appearance of smoked fish also positively correlate with phenol content. Phenol compounds in smoke  
401 interact with amino acid components in fish meat, producing a distinctive golden color in smoked fish  
402 (Montazeri *et al.*, 2013).

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

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

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

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

436

#### 437 **Conclusion**

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

446

#### 447 **Acknowledgement (bold)**

#### 448 **Author contributions**

#### 449 **Conflicts of interest**

450 The authors declare that there is no conflict of interest.

451

452

#### 453 **Highlights**

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

456

457

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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.

# 1 **Characteristics of *Julung Julung* Smoked Fish Fillets (*Hemiramphus* sp.) using** 2 **Liquid Smoke from Corn Cobs Waste**

## 3 **Abstract**

4 Corn cobs can be utilized as a shell ingredient for liquid smoke. Liquid smoke can be used for the smoked fish  
5 industry in North Sulawesi, Indonesia. However, the utilization of liquid smoke for the smoked fish industry has  
6 yet to be optimal. This study aimed to obtain the best smoking method for *julung julung* fish fillets (*Hemiramphus*  
7 sp.) using liquid smoke from corn cob waste. The treatment in this study was to apply liquid smoke from corn cob  
8 waste in the smoking process, which was compared with the conventional method (using shell, coconut wood, and  
9 corn cob). Parameters to determine the quality of smoked fish were total volatile bases (TVB), moisture, water  
10 activity, pH, phenol levels, polycyclic aromatic hydrocarbon (PAH), and sensory assessment. The results showed  
11 that the TVB of smoked *julung julung* fish fillets ranged from 19.83 – 32.27 mg N/100g. The moisture ranged  
12 from 14.10 – 30.57%. Water activity ranged from 0.46 – 0.82. pH ranged from 4.60 – 5.81. Phenol levels ranged  
13 from 4.42 – 16.11 mg/g. PAH levels are still below the standard required in the Indonesian National Standard.  
14 Panelists rated neutral to really like the appearance, aroma, taste, and texture of smoked fish. From these results,  
15 it can be concluded that the best treatment is fresh fillets preheated in a cabinet dryer for 4 hours at 60 – 80 °C and  
16 then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated in a cabinet dryer for 4 hours at 90  
17 °C.

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

**Commented [u1]:** Corn cobs  
Liquid smoke

## 20 **Introduction**

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

26 One of the corn cobs was used to make liquid smoke. Previous research reported that corn cobs can  
27 produce liquid smoke, a by-product of the pyrolysis of corn cob waste. The yield of liquid smoke from  
28 corn cobs is about 28.37%, with a pH value of 3.5 (Sriharti *et al.*, 2020). The particle size of corn cobs  
29 affects the yield of liquid smoke, with smaller particle sizes resulting in higher yields (Aladin *et al.*,  
30 2018). Another study reported a phenolic content of 335 mg/L in liquid smoke produced from corn cobs  
31 (Swastawati *et al.*, 2007).

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

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

42 The conventional smoking processes generally use hardwood as fuel, such as melon wood (Umar *et al.*,  
43 2018), mangrove wood (Cissoko *et al.*, 2020), teak wood (Daramola *et al.*, 2020), coconut shells, and  
44 coconut husks (Nugroho *et al.*, 2018). Corn cobs have also been reported to be used in the fish smoking  
45 process (Sukowati, 2023). According to literature, traditional fish smoking in North Sulawesi,  
46 Indonesia, uses smoke from burning coconut shells or wood charcoal (Landangkasiang *et al.*, 2017;  
47 Primalasari *et al.*, 2019; Saediman *et al.*, 2021). Smoked fish processing has been known for a long time  
48 because of its traditional processing techniques, simplicity, ease of implementation, and low cost  
49 (Islamiyah, 2021). In North Sulawesi, Indonesia, smoked fish commonly known as *fufu* is traditionally  
50 made from skipjack (*Katsuwonus pelamis*); *julung julung* (*Hemiramphus far*) (Berhimpon *et al.*, 2018);  
51 and scad fish (*Decapterus* spp.) known as *pinekuhe* (Ansar & Ijong, 2021). The smoked fish product of  
52 *julung julung* is a typical product from North Sulawesi and Gorontalo, commonly called *roa* or *galavea*  
53 (Azis & Akolo, 2020).

54 The study's reported that the conventional smoking process has disadvantages such as smoking time,  
55 concentration of carcinogenic substances, temperature, and inconsistent product quality (Racovita *et al.*,  
56 2020). Conventional smoking of food products has been shown to produce carcinogenic components  
57 such as polycyclic aromatic hydrocarbons (Jinadasa *et al.*, 2020). Benzo(a)pyrene (C<sub>20</sub>H<sub>12</sub>) is one of the  
58 carcinogenic compounds produced from traditional smoking products (Jinadasa *et al.*, 2020). One  
59 method to reduce carcinogenic compounds in smoked fish products is to use liquid smoke in the  
60 smoking process (Nithin *et al.*, 2020; Xin *et al.*, 2021).

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

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

72 smoked fish requires a maximum total plate count of  $5 \times 10^4$  CFU/g and a maximum moisture content of  
73 60% (Indonesia Standardization Agency, 2013). Another study reported that smoked fish processed  
74 with liquid smoke had a moisture content of 47.63%, phenol content of 12.62%, and pH of 4.8  
75 (Berhimpon *et al.*, 2018). ~~The results of these studies indicate that liquid smoke is good to apply to~~  
76 ~~smoked fish products.~~ This study characterized smoked fish fillets of *julung julung* (*Hemiramphus* sp.)  
77 produced with liquid smoke from corn cobs waste. The study was conducted by dipping the fish fillets  
78 of *julung julung* with liquid smoke, with drying and steaming treatments on the fish fillets. This study  
79 aims to obtain the best smoking method for fish fillets using liquid smoke from corn cobs waste based  
80 on the assessment of total volatile bases, moisture content, water activity, pH value, phenol content,  
81 polycyclic aromatic hydrocarbon content, and sensory assessment.

82

### 83 Materials and methods

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

92



93

94

95

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

Commented [u2]: Why used this concentration??

96 The treatment in this study was applying liquid smoke from corn cobs waste to the smoking process  
97 of *julung julung* fish fillets, which was compared with the conventional method (using coconut wood,  
98 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.

Commented [u3]: Refrences?

Commented [u4]: Refrences?

99

#### 100 Liquid smoke manufacturing process

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

Commented [u5]: Temperature?

Commented [u6]: Reference?

Commented [u7]: How?? To separate what?? how about benzopyrene?

109

#### 110 Total volatile bases assay

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

Commented [u8]: Volatile Base

118

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

119

120 **Moisture content assay**

121 The moisture content by method of Indonesia National Standard (Indonesia Standardization Agency,  
122 2006a). An empty porcelain cup is weighed (A). Then, the sample was weighed to  $\pm 2$  g in a porcelain  
123 cup of known mass (B). The samples were dried in an oven at 105°C for 20 hours. The samples were  
124 then cooled in a desiccator, after which the samples in the cup were weighed (C). Moisture content was  
125 expressed as %.

126 
$$\text{Moisture (\%)} = \frac{B (g) - C (g)}{B(g) - A (g)} \times 100\%$$

127

128 **Water activity (Aw) assay**

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

134

135 **pH assay**

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

140

141 **Phenol level assay**

142 Samples were extracted by adding 1 mL ethanol p.a., 5 mL distilled water, and 0.5 mL Folin Ciocalteu  
143 50%, homogenized, and allowed to stand for 5 minutes. Then added, 1 mL of 5% Na<sub>2</sub>CO<sub>3</sub> and left in  
144 the dark for  $\pm 60$  minutes with gallic acid as a standard. The absorbance value was read on a  
145 spectrophotometer (Thermo Scientific Genesys 50) with a wavelength of 725 nm. Total phenolics were  
146 expressed as mg/g (Indonesia Standardization Agency, 2004; Muliadin *et al.*, 2022).

147 
$$\text{Total Phenol } \left(\frac{mg}{g}\right) = \frac{\text{Gallic acid equivalence } \left(\frac{mg}{L}\right) \times \text{Volume (L)}}{\text{Sample weight (g)}}$$

148

149 **Polycyclic aromatic hydrocarbon (PAH) assay**

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

152 of internal standard solution (9,10 dimethylantracene), and some boiling stones were added. After that,  
153 the mixture was boiled for 4 hours in a soxhlet. The liquid phase was transferred to a separatory funnel  
154 and extracted with 100 mL of n-hexane with shaking for 3 min. The MeOH:KOH phase was drained  
155 and discarded. The combined n-hexane phase was rinsed twice, each with 50 mL of H<sub>2</sub>O-MeOH (8:1)  
156 and 50 mL of water. The methanol-water and water phases were dried and discarded. The organic phase  
157 was concentrated in a rotary evaporator (40°C) to a volume of about 10 mL. The concentrated extract  
158 was dried under nitrogen gas.

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

169

#### 170 Sensory assessment assay

171 Sensory assessment assay refers to Indonesia National Standard (Indonesia Standardization Agency,  
172 2006b). The sensory assessment was based on a hedonic scale (Table 1). Smoked fish samples were  
173 placed on a plastic plate with a glass of water, coded, and presented to 30 panelists randomly under  
174 light. The parameters observed were appearance, aroma, taste, and texture. Panelists rated acceptance  
175 of the samples on a scale of 1 – 9.

176

177

**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

178

Commented [u9]: Without any preparation??

179 **Data analysis**

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

184 **Results and discussion**

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

192 **Total volatile bases**

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

201

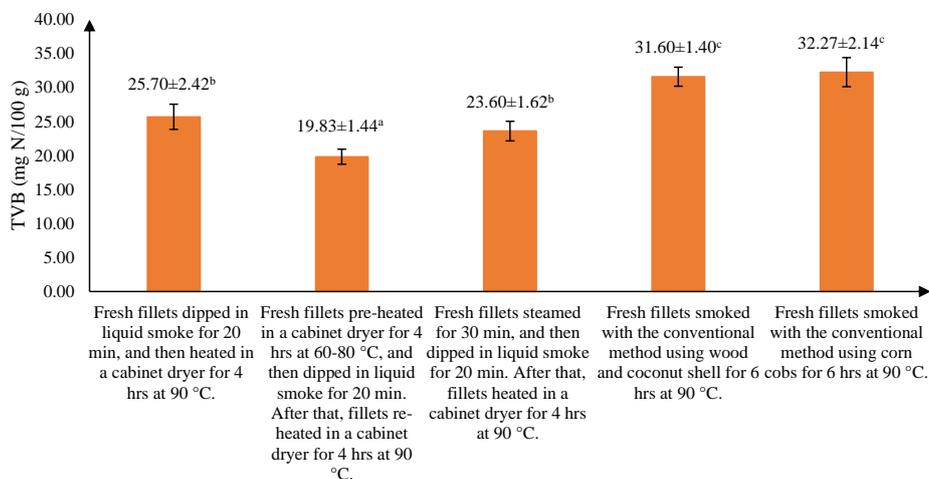


Fig. 2. Total volatile bases of *julung julung* smoked fillet

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).

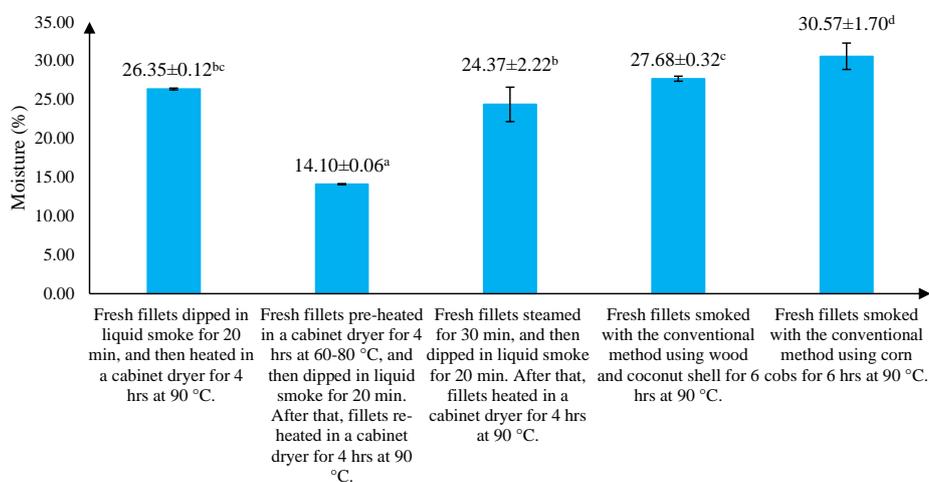
224 The European standard (European Market Observatory for Fisheries and Aquaculture, 2020) states that  
 225 the limit range of TVB value for smoked fish is 25 – 35 mg N/100 g. All treatments' TVB value of the  
 226 smoked fish fillets still met the European standard. Previous studies reported TVB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

233

234 **Moisture content**

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

240



241

242

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

243

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

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

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

#### 268 **Water activity (Aw)**

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

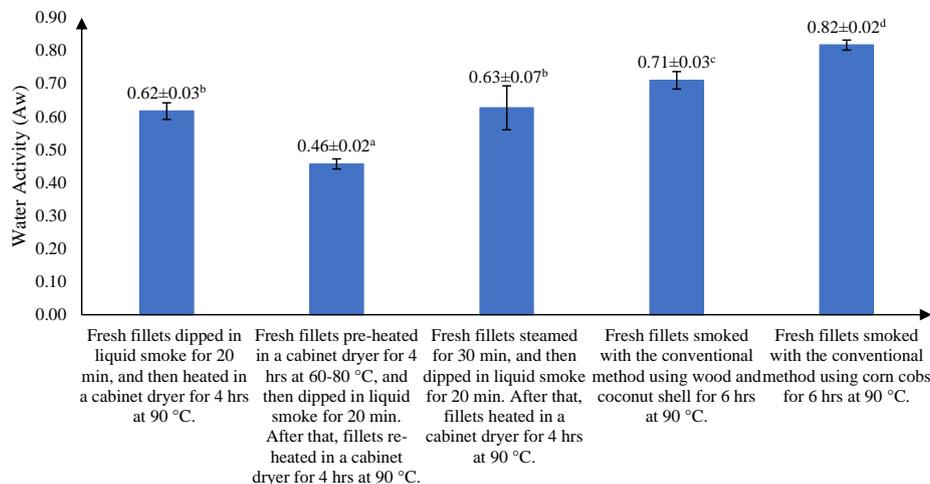


Fig. 4. Water activity of *julung julung* smoked fillet

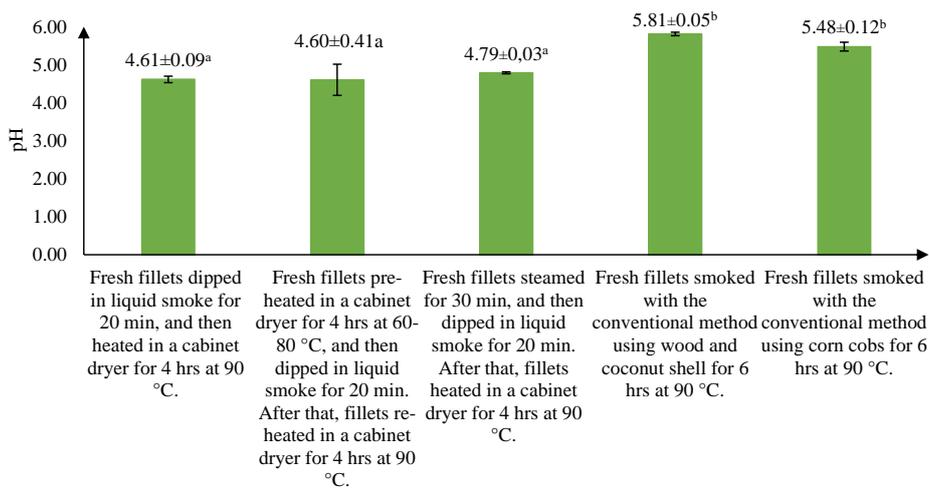
Fig. 4 shows the analysis of the variance of smoked fish fillet *julung julung* with different smoking method treatments affecting water activity ( $p < 0.05$ ). The water activity value of smoked fish fillet ranged from 0.46 – 0.82, with the lowest water activity in treatment B. The treatment of the 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

302 method. Another study also reported that using liquid smoke can reduce pH caused by the condensation  
 303 of organic acids in the smoking process (Puke & Galoburda, 2020). The pH value of smoked fish  
 304 correlates with the phenol content contained in the smoke component, and an increase in phenol content  
 305 causes a decrease in pH (Berhimpon *et al.*, 2018).  
 306



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

308  
 309  
 310 **Phenol level**

311 Phenol is a compound found in wood smoke. Phenol compounds enter food through diffusion and  
 312 capillary action, affecting taste, color, and aroma and extending shelf life (Remy *et al.*, 2016). In  
 313 addition, phenol compounds are also reported to act as antioxidants that can prevent rancidity in fish  
 314 meat (Sérot *et al.*, 2004). Leksono *et al.* (2020) also noted that the higher the phenol level in smoke, the  
 315 stronger the flavor and aroma of smoked fish. The analysis of the variance of smoked fish fillets *julung*  
 316 *julung* with different smoking method treatments affecting phenol level ( $p < 0.05$ ). The phenol level of  
 317 smoked fish fillet ranged from 4.42 to 16.11 mg/g (Fig. 6). The treatment of the smoking method with  
 318 corn cob liquid smoke (treatments A, B, and C) had higher phenol level when compared to the  
 319 conventional smoking method (treatments D and E).  
 320

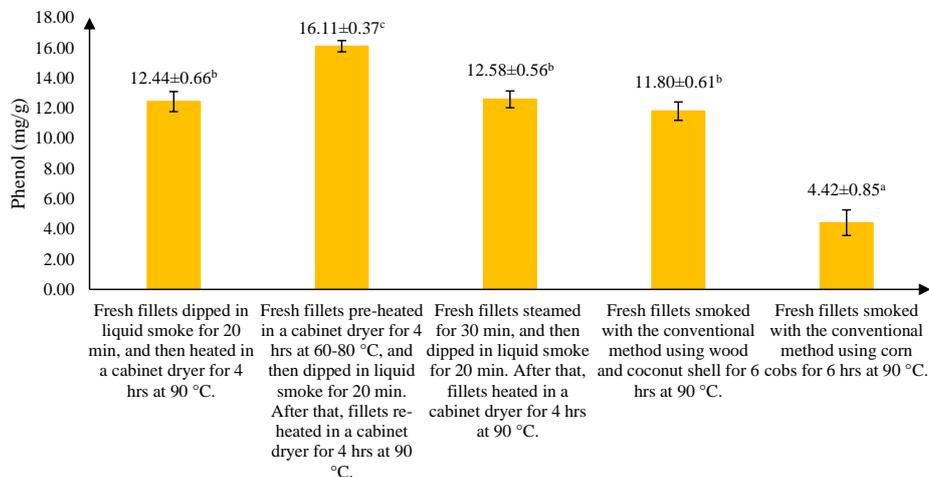


Fig. 6. Phenol level of *julung julung* smoked fillet

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

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

Polycyclic Aromatic Hydrocarbon	Smoked <i>Julung Julung</i> fish (µg/kg)					Corn Cob Liquid Smoke
	A	B	C	D	E	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

345 nd = not detected

346

347 Table 2 shows that the PAH levels in smoked fish are still below the standard required in the Indonesian  
 348 National Standard No. 2725 of 2013 on smoked fish, which is benzo(a)pyrene max 5 µg/kg (Indonesia  
 349 Standardization Agency, 2013). The polycyclic aromatic hydrocarbon compound regulated in the  
 350 Indonesian National Standard is only benzo(a)pyrene. Stołyhwo & Sikorski (2005) reported  
 351 benzo(a)pyrene levels in smoked fish with hot and cold smoking processes ranging from 0.05 to about  
 352 60 µg/kg. Other studies have reported benzo(b,k)fluoranthene levels in salmon and rainbow trout  
 353 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.*,  
 354 2010). Berhimpon *et al.* (2018) also reported benzo(a)pyrene levels in smoked skipjack fish of 0.25  
 355 µg/kg. The level of benzo(a)pyrene in smoked barracuda was reported to be 0.32 µg/kg,  
 356 benzo(b)fluoranthene 0.35 µg/kg, benzo(k)fluoranthene 0.21 µg/kg, benzo(a)anthracene 0.44 µg/kg,  
 357 and benzo(g,h)perylene 2.56 µg/kg (Asamoah *et al.*, 2021).

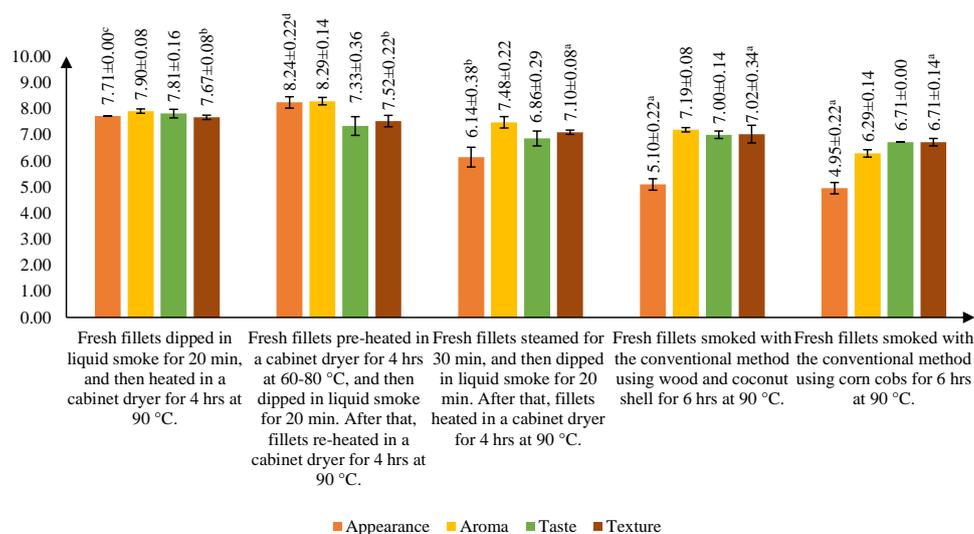
358 High PAH levels in smoked fish are due to the lignin content of the material used as fuel in the smoking  
 359 process (Asamoah *et al.*, 2021). The lignin content in corn cobs is reported to be about 17 – 21%  
 360 (Thangavelu *et al.*, 2018; Olajuyigbe *et al.*, 2019; Gandam *et al.*, 2022).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

367

368 **Sensory assessment**

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

374 customize products according to consumer assessment (Ekelemu *et al.*, 2021). Indonesian National  
 375 Standard No. 2725:2013 regarding smoked fish requires a minimum sensory assessment of 7 (Score 1-  
 376 9) (Indonesia Standardization Agency, 2013). The results of the panelists' assessment of *julung julung*  
 377 smoked fillet from each treatment can be seen in Fig. 7.  
 378



379  
 380 **Fig. 7.** Sensory assesment of *julung julung* smoked fillet  
 381

382 **Appearance:** Fig. 7 shows the analysis of the variance of the sensory assessment of the appearance of  
 383 smoked *julung julung* fish fillets with different smoking method treatments affecting the appearance of  
 384 smoked fish ( $p < 0.05$ ). The panelists' assessment of the appearance of smoked fish fillets ranged from  
 385 4.95 to 8.24, with the highest panelists' assessment in treatment B. Based on the requirements of the  
 386 Indonesian National Standard, only treatments A and B met the minimum panelist assessment  
 387 requirement of 7. The moisture content factor is thought to have influenced the panelists' assessment of  
 388 the appearance of smoked fish, so panelists less favored treatment C with steaming. Moisture content  
 389 can affect the physical properties of fish, such as the appearance and texture of smoked fish (Baten *et al.*,  
 390 2020a). Smoked fish with high moisture content will make the color of smoked fish look paler  
 391 (Flick, 2010).  
 392 Treatments D and E were less favored by panelists, presumably because conventional smoking methods  
 393 produce darker products (dark brown), less bright and less shiny. Smoking fish with liquid smoke has  
 394 smoked fish products with a golden color and clean and shiny surface (Berhimpon *et al.*, 2018). Another  
 395 factor that affects the appearance of smoked fish is the length of the smoking process. The longer the

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

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

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

420 **Texture:** Fig. 7 shows the analysis of variance of the sensory assessment of the texture of smoked fish  
421 fillet *julung julung* with different smoking method treatments giving effect to the texture of smoked fish  
422 ( $p<0.05$ ). Panelists' assessment of the texture of smoked fish *julung julung*, ranged from 6.71 – 7.67,  
423 with the highest panelist assessment in treatments A and B. Duncan's test analysis showed that  
424 treatments A and B differed from treatments C, D, and E. This result was thought to be because the fish  
425 fillets were dipped in liquid smoke, giving a dry, compact, and tender texture. Whereas in treatment C  
426 (fish fillets subjected to steaming), the texture of the smoked fillets was rather sticky and not solid.  
427 Treatments D and E produced the texture of smoked fish fillets which were less dense and not compact.  
428 The texture of smoked fish is negatively correlated with its moisture content. The higher the moisture  
429 content in smoked fish, the softer and less firm the texture (Chan *et al.*, 2022). High moisture content  
430 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 °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 °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.

### 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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# 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 °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 °C.

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

## Introduction

Corn is the second most widely grown crop in Indonesia after rice. Indonesia is the 7<sup>th</sup> 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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37 while reducing production time (Indiarto *et al.*, 2020). Generally, various types of hardwood were used  
38 to manufacture liquid smoke (Diatmika *et al.*, 2019). The quality of the liquid smoke produced may  
39 vary depending on the type of wood used (Budaraga *et al.*, 2016). In addition to imparting specific  
40 flavors and aromas to foods (Maulina *et al.*, 2020), liquid smoke is reported to be antimicrobial (Septana  
41 *et al.*, 2020) and antioxidant (Budaraga & Putra, 2021).

42 The conventional smoking processes generally use hardwood as fuel, such as melon wood (Umar *et al.*,  
43 2018), mangrove wood (Cissoko *et al.*, 2020), teak wood (Daramola *et al.*, 2020), coconut shells, and  
44 coconut husks (Nugroho *et al.*, 2018). Corn cobs have also been reported to be used in the fish smoking  
45 process (Sukowati, 2023). According to literature, traditional fish smoking in North Sulawesi,  
46 Indonesia, uses smoke from burning coconut shells or wood charcoal (Landangkasiang *et al.*, 2017;  
47 Primalasari *et al.*, 2019; Saediman *et al.*, 2021). Smoked fish processing has been known for a long time  
48 because of its traditional processing techniques, simplicity, ease of implementation, and low cost  
49 (Islamiyah, 2021). In North Sulawesi, Indonesia, smoked fish commonly known as *fufu* is traditionally  
50 made from skipjack (*Katsuwonus pelamis*); *julung julung* (*Hemiramphus far*) (Berhimpon *et al.*, 2018);  
51 and scad fish (*Decapterus* spp.) known as *pinekuhe* (Ansar & Ijong, 2021). The smoked fish product of  
52 *julung julung* is a typical product from North Sulawesi and Gorontalo, commonly called *roa* or *galavea*  
53 (Azis & Akolo, 2020).

54 The study's reported that the conventional smoking process has disadvantages such as smoking time,  
55 concentration of carcinogenic substances, temperature, and inconsistent product quality (Racovita *et al.*,  
56 2020). Conventional smoking of food products has been shown to produce carcinogenic components  
57 such as polycyclic aromatic hydrocarbons (Jinadasa *et al.*, 2020). Benzo(a)pyrene (C<sub>20</sub>H<sub>12</sub>) is one of the  
58 carcinogenic compounds produced from traditional smoking products (Jinadasa *et al.*, 2020). One  
59 method to reduce carcinogenic compounds in smoked fish products is to use liquid smoke in the  
60 smoking process (Nithin *et al.*, 2020; Xin *et al.*, 2021).

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

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

72 smoked fish requires a maximum total plate count of  $5 \times 10^4$  CFU/g and a maximum moisture content of  
73 60% (Indonesia Standardization Agency, 2013). Another study reported that smoked fish processed  
74 with liquid smoke had a moisture content of 47.63%, phenol content of 12.62%, and pH of 4.8  
75 (Berhimpon *et al.*, 2018). The results of these studies indicate that liquid smoke is good to apply to  
76 smoked fish products. This study characterized smoked fish fillets of *julung julung* (*Hemiramphus* sp.)  
77 produced with liquid smoke from corn cobs waste. The study was conducted by dipping the fish fillets  
78 of *julung julung* with liquid smoke, with drying and steaming treatments on the fish fillets. This study  
79 aims to obtain the best smoking method for fish fillets using liquid smoke from corn cobs waste based  
80 on the assessment of total volatile bases, moisture content, water activity, pH value, phenol content,  
81 polycyclic aromatic hydrocarbon content, and sensory assessment.

82

### 83 Materials and methods

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

92



93

94

95

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

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96 The treatment in this study was applying liquid smoke from corn cobs waste to the smoking process  
97 of *julung julung* fish fillets, which was compared with the conventional method (using coconut wood,  
98 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.

99

#### 100 **Liquid smoke manufacturing process**

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

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#### 110 **Total volatile bases assay**

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

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

118

119

120 **Moisture content assay**

121 The moisture content by method of Indonesia National Standard (Indonesia Standardization Agency,  
122 2006a). An empty porcelain cup is weighed (A). Then, the sample was weighed to  $\pm 2$  g in a porcelain  
123 cup of known mass (B). The samples were dried in an oven at 105°C for 20 hours. The samples were  
124 then cooled in a desiccator, after which the samples in the cup were weighed (C). Moisture content was  
125 expressed as %.

126 
$$\text{Moisture (\%)} = \frac{B (g) - C (g)}{B(g) - A (g)} \times 100\%$$

127

128 **Water activity (Aw) assay**

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

134

135 **pH assay**

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

140

141 **Phenol level assay**

142 Samples were extracted by adding 1 mL ethanol p.a., 5 mL distilled water, and 0.5 mL Folin Ciocalteu  
143 50%, homogenized, and allowed to stand for 5 minutes. Then added, 1 mL of 5% Na<sub>2</sub>CO<sub>3</sub> and left in  
144 the dark for  $\pm 60$  minutes with gallic acid as a standard. The absorbance value was read on a  
145 spectrophotometer (Thermo Scientific Genesys 50) with a wavelength of 725 nm. Total phenolics were  
146 expressed as mg/g (Indonesia Standardization Agency, 2004; Muliadin *et al.*, 2022).

147 
$$\text{Total Phenol } \left(\frac{mg}{g}\right) = \frac{\text{Gallic acid equivalence } \left(\frac{mg}{L}\right) \times \text{Volume (L)}}{\text{Sample weight (g)}}$$

148

149 **Polycyclic aromatic hydrocarbon (PAH) assay**

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

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152 of internal standard solution (9,10 dimethylantracene), and some boiling stones were added. After that,  
153 the mixture was boiled for 4 hours in a soxhlet. The liquid phase was transferred to a separatory funnel  
154 and extracted with 100 mL of n-hexane with shaking for 3 min. The MeOH:KOH phase was drained  
155 and discarded. The combined n-hexane phase was rinsed twice, each with 50 mL of H<sub>2</sub>O-MeOH (8:1)  
156 and 50 mL of water. The methanol-water and water phases were dried and discarded. The organic phase  
157 was concentrated in a rotary evaporator (40°C) to a volume of about 10 mL. The concentrated extract  
158 was dried under nitrogen gas.

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

169  
170 **Sensory assessment assay**

171 Sensory assessment assay refers to Indonesia National Standard (Indonesia Standardization Agency,  
172 2006b). The sensory assessment was based on a hedonic scale (Table 1). Smoked fish samples were  
173 placed on a plastic plate with a glass of water, coded, and presented to 30 panelists randomly under  
174 light. The parameters observed were appearance, aroma, taste, and texture. Panelists rated acceptance  
175 of the samples on a scale of 1 – 9.

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176  
177 **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

178

179 **Data analysis**

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

183

184 **Results and discussion**

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

191

192 **Total volatile bases**

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

201

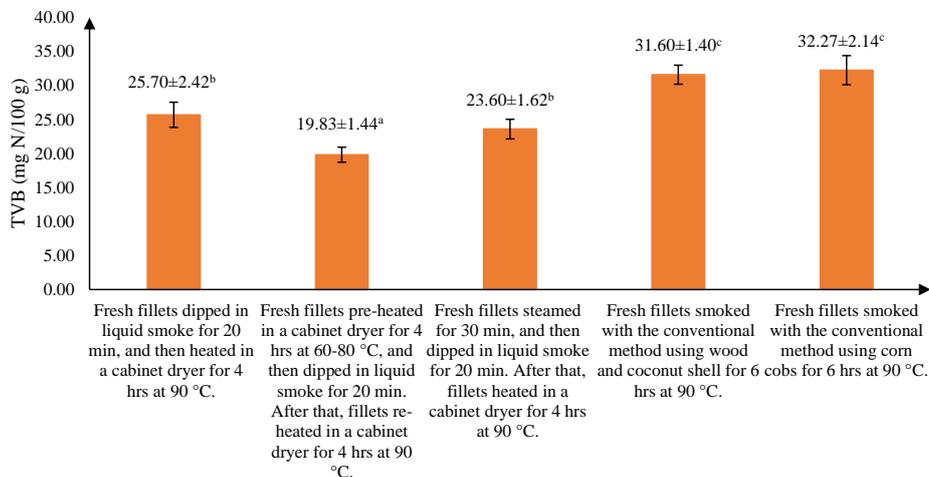


Fig. 2. Total volatile bases of *Julung Julung* smoked fillet

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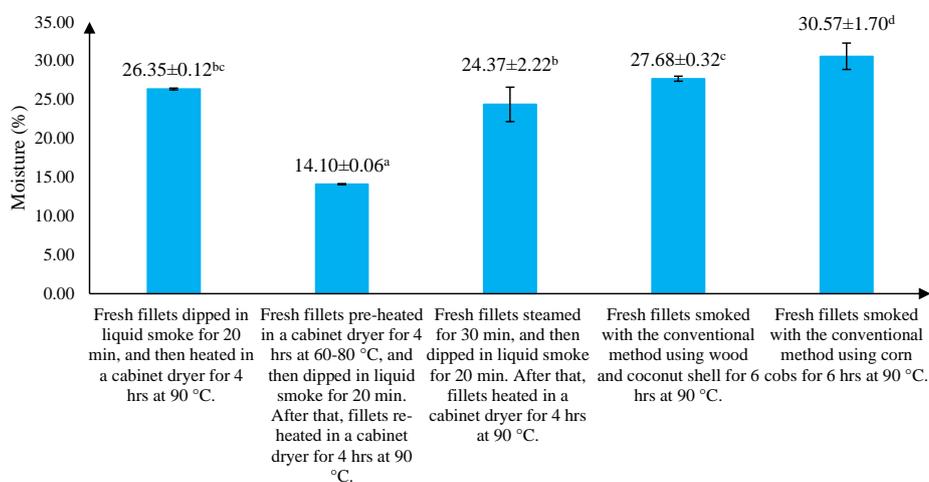
224 The European standard (European Market Observatory for Fisheries and Aquaculture, 2020) states that  
 225 the limit range of TVB value for smoked fish is 25 – 35 mg N/100 g. All treatments' TVB value of the  
 226 smoked fish fillets still met the European standard. Previous studies reported TVB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

233

234 **Moisture content**

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

240



241

242

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

243

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

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

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

#### 268 **Water activity (Aw)**

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

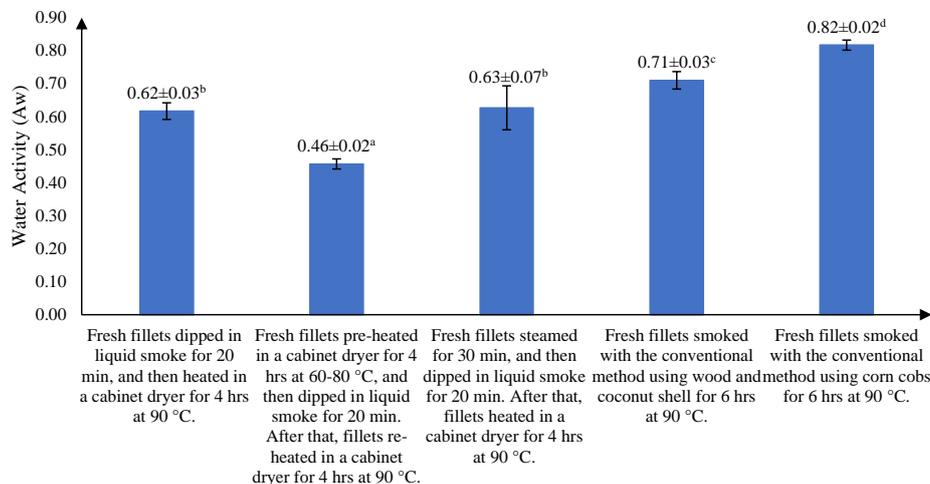


Fig. 4. Water activity of *julung julung* smoked fillet

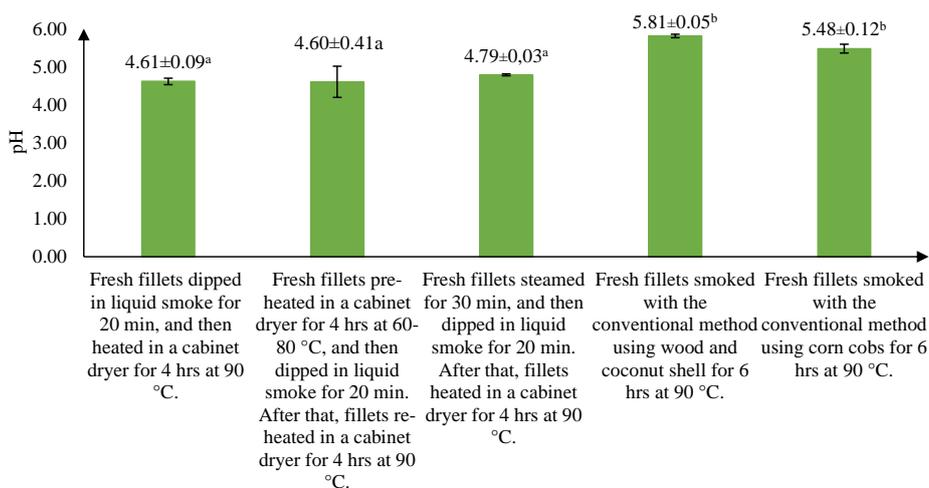
Fig. 4 shows the analysis of the variance of smoked fish fillet *julung julung* with different smoking method treatments affecting water activity ( $p < 0.05$ ). The water activity value of smoked fish fillet ranged from 0.46 – 0.82, with the lowest water activity in treatment B. The treatment of the 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

302 method. Another study also reported that using liquid smoke can reduce pH caused by the condensation  
 303 of organic acids in the smoking process (Puke & Galoburda, 2020). The pH value of smoked fish  
 304 correlates with the phenol content contained in the smoke component, and an increase in phenol content  
 305 causes a decrease in pH (Berhimpon *et al.*, 2018).  
 306



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

308  
 309  
 310 **Phenol level**

311 Phenol is a compound found in wood smoke. Phenol compounds enter food through diffusion and  
 312 capillary action, affecting taste, color, and aroma and extending shelf life (Remy *et al.*, 2016). In  
 313 addition, phenol compounds are also reported to act as antioxidants that can prevent rancidity in fish  
 314 meat (Sérot *et al.*, 2004). Leksono *et al.* (2020) also noted that the higher the phenol level in smoke, the  
 315 stronger the flavor and aroma of smoked fish. The analysis of the variance of smoked fish fillets *julung*  
 316 *julung* with different smoking method treatments affecting phenol level ( $p < 0.05$ ). The phenol level of  
 317 smoked fish fillet ranged from 4.42 to 16.11 mg/g (Fig. 6). The treatment of the smoking method with  
 318 corn cob liquid smoke (treatments A, B, and C) had higher phenol level when compared to the  
 319 conventional smoking method (treatments D and E).  
 320

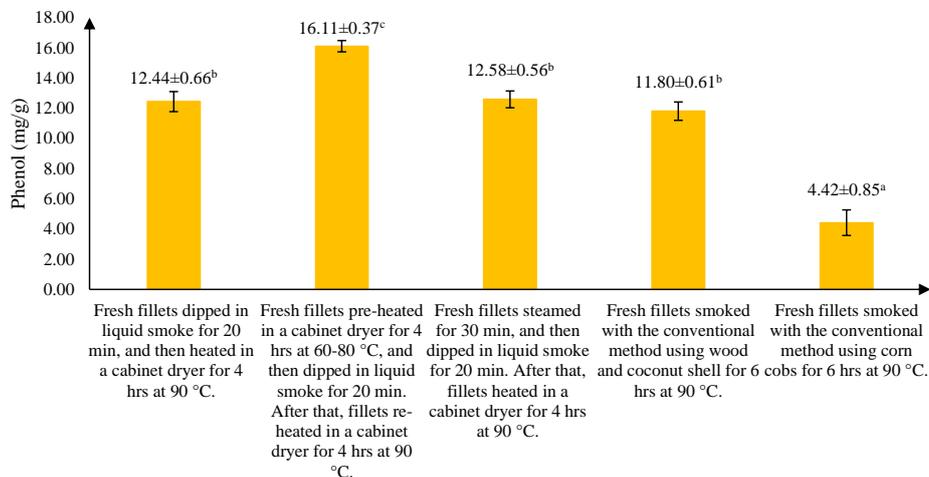


Fig. 6. Phenol level of *julung julung* smoked fillet

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

344 **Table 2.** Polycyclic aromatic hydrocarbon levels in *Julung Julung* smoked fillet

Polycyclic Aromatic Hydrocarbon	Smoked <i>Julung Julung</i> fish (µg/kg)					Corn Cob Liquid Smoke
	A	B	C	D	E	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

345 nd = not detected

346

347 Table 2 shows that the PAH levels in smoked fish are still below the standard required in the Indonesian  
 348 National Standard No. 2725 of 2013 on smoked fish, which is benzo(a)pyrene max 5 µg/kg (Indonesia  
 349 Standardization Agency, 2013). The polycyclic aromatic hydrocarbon compound regulated in the  
 350 Indonesian National Standard is only benzo(a)pyrene. Stołyhwo & Sikorski (2005) reported  
 351 benzo(a)pyrene levels in smoked fish with hot and cold smoking processes ranging from 0.05 to about  
 352 60 µg/kg. Other studies have reported benzo(b,k)fluoranthene levels in salmon and rainbow trout  
 353 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.*,  
 354 2010). Berhimpon *et al.* (2018) also reported benzo(a)pyrene levels in smoked skipjack fish of 0.25  
 355 µg/kg. The level of benzo(a)pyrene in smoked barracuda was reported to be 0.32 µg/kg,  
 356 benzo(b)fluoranthene 0.35 µg/kg, benzo(k)fluoranthene 0.21 µg/kg, benzo(a)anthracene 0.44 µg/kg,  
 357 and benzo(g,h)perylene 2.56 µg/kg (Asamoah *et al.*, 2021).

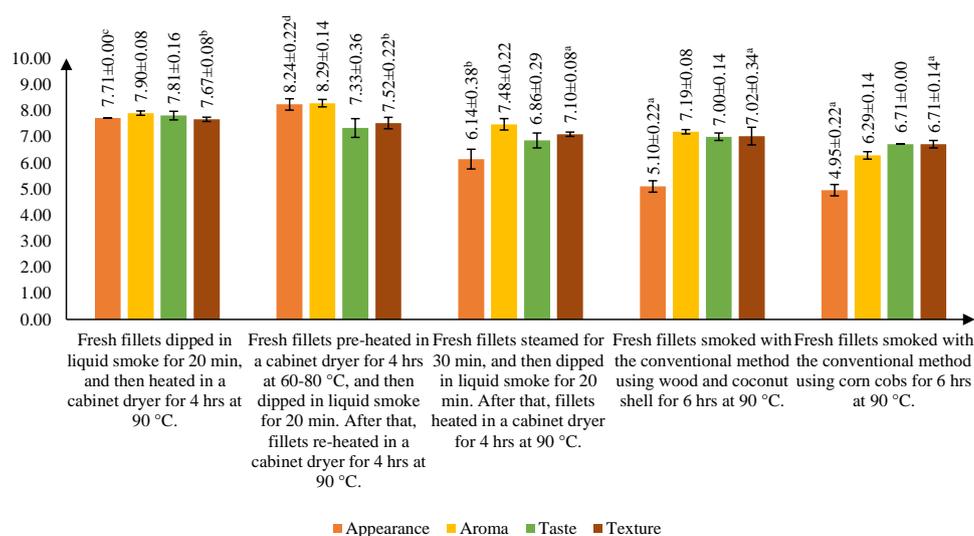
358 High PAH levels in smoked fish are due to the lignin content of the material used as fuel in the smoking  
 359 process (Asamoah *et al.*, 2021). The lignin content in corn cobs is reported to be about 17 – 21%  
 360 (Thangavelu *et al.*, 2018; Olajuyigbe *et al.*, 2019; Gandam *et al.*, 2022).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

367

368 **Sensory assessment**

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

374 customize products according to consumer assessment (Ekelemu *et al.*, 2021). Indonesian National  
 375 Standard No. 2725:2013 regarding smoked fish requires a minimum sensory assessment of 7 (Score 1-  
 376 9) (Indonesia Standardization Agency, 2013). The results of the panelists' assessment of *julung julung*  
 377 smoked fillet from each treatment can be seen in Fig. 7.  
 378



379  
 380 **Fig. 7.** Sensory assesment of *julung julung* smoked fillet  
 381

382 **Appearance:** Fig. 7 shows the analysis of the variance of the sensory assessment of the appearance of  
 383 smoked *julung julung* fish fillets with different smoking method treatments affecting the appearance of  
 384 smoked fish ( $p < 0.05$ ). The panelists' assessment of the appearance of smoked fish fillets ranged from  
 385 4.95 to 8.24, with the highest panelists' assessment in treatment B. Based on the requirements of the  
 386 Indonesian National Standard, only treatments A and B met the minimum panelist assessment  
 387 requirement of 7. The moisture content factor is thought to have influenced the panelists' assessment of  
 388 the appearance of smoked fish, so panelists less favored treatment C with steaming. Moisture content  
 389 can affect the physical properties of fish, such as the appearance and texture of smoked fish (Baten *et*  
 390 *al.*, 2020a). Smoked fish with high moisture content will make the color of smoked fish look paler  
 391 (Flick, 2010).  
 392 Treatments D and E were less favored by panelists, presumably because conventional smoking methods  
 393 produce darker products (dark brown), less bright and less shiny. Smoking fish with liquid smoke has  
 394 smoked fish products with a golden color and clean and shiny surface (Berhimpon *et al.*, 2018). Another  
 395 factor that affects the appearance of smoked fish is the length of the smoking process. The longer the

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

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

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

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420 **Texture:** Fig. 7 shows the analysis of variance of the sensory assessment of the texture of smoked fish  
421 fillet *julung julung* with different smoking method treatments giving effect to the texture of smoked fish  
422 ( $p<0.05$ ). Panelists' assessment of the texture of smoked fish *julung julung*, ranged from 6.71 – 7.67,  
423 with the highest panelist assessment in treatments A and B. Duncan's test analysis showed that  
424 treatments A and B differed from treatments C, D, and E. This result was thought to be because the fish  
425 fillets were dipped in liquid smoke, giving a dry, compact, and tender texture. Whereas in treatment C  
426 (fish fillets subjected to steaming), the texture of the smoked fillets was rather sticky and not solid.  
427 Treatments D and E produced the texture of smoked fish fillets which were less dense and not compact.  
428 The texture of smoked fish is negatively correlated with its moisture content. The higher the moisture  
429 content in smoked fish, the softer and less firm the texture (Chan *et al.*, 2022). High moisture content  
430 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 °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 °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.

### 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**

# 1 Characteristics of *Julung Julung* Smoked Fillets (*Hemiramphus* sp.) using Liquid 2 Smoke from Corn Cobs Waste

## 3 Abstract

4 This study aimed to obtain the best smoking method for *Julung Julung* fillets (*Hemiramphus* sp.) using liquid  
5 smoke from corn cob waste. This study used three different fillet treatment methods (dried and steamed) before  
6 liquid smoke application. This study also compared fish smoking between using liquid smoke with conventional  
7 methods (using shell, coconut wood, and corn cob). The parameters to determine the quality of smoked fish were  
8 Total Volatile Bases (TVB), moisture, water activity, pH, phenol levels, polycyclic aromatic hydrocarbon (PAH),  
9 and sensory assessment. The results showed that the TVB of smoked *Julung Julung* fillets ranged from 19.83 –  
10 32.27 mg N/100g. The moisture ranged from 14.10 – 30.57%. Water activity ranged from 0.46 – 0.82. pH ranged  
11 from 4.60 – 5.81. Phenol levels ranged from 4.42 – 16.11 mg/g. PAH levels are still below the standard required  
12 in the Indonesian National Standard. Panelists rated neutral to really like the appearance, aroma, taste, and texture  
13 of smoked fish. From these research, it can be concluded that treatment B is the best treatment, namely fresh fillets  
14 are first heated for 4 hours at 60 – 80 °C and then dipped in liquid smoke for 20 minutes. After that, the fillets were  
15 reheated for 4 hours at 90 °C.

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

## 18 Introduction

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

24 One of the corn cobs was used to make liquid smoke. Previous research reported that corn cobs can  
25 produce liquid smoke, a by-product of the pyrolysis of corn cob waste. The yield of liquid smoke from  
26 corn cobs is about 28.37%, with a pH value of 3.5 (Sriharti *et al.*, 2020). The particle size of corn cobs  
27 affects the yield of liquid smoke, with smaller particle sizes resulting in higher yields (Aladin *et al.*,  
28 2018). Swastawati *et al.* (2007) reported the phenolic content in liquid smoke produced from 335 mg/L  
29 corn cobs.

30 Liquid smoke is a natural product made from the condensation of smoke from burning wood (Andy *et al.*,  
31 2021). Liquid smoke is commonly used as a flavoring in food to provide a smoked flavor without  
32 the food undergoing the actual smoking process (Sari *et al.*, 2006). Using liquid smoke in food will save  
33 time, energy, and labor, so that can reduce production costs (Krah *et al.*, 2019). In addition, using liquid  
34 smoke in food can speed up and standardize the smoking process, adding flavor and microbiological  
35 safety while reducing production time (Indiarto *et al.*, 2020). Generally, various types of hardwood were  
36 used to manufacture liquid smoke (Diatmika *et al.*, 2019). The quality of the liquid smoke produced

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

40 The conventional smoking processes generally use hardwood as fuel, such as melon wood (Umar *et al.*,  
41 2018), mangrove wood (Cissoko *et al.*, 2020), teak wood (Daramola *et al.*, 2020), coconut shells, and  
42 coconut husks (Nugroho *et al.*, 2018). Corn cobs have also been reported to be used in the fish smoking  
43 process (Sukowati, 2023). According to literature, traditional fish smoking in North Sulawesi,  
44 Indonesia, uses smoke from burning coconut shells or wood charcoal (Landangkasiang *et al.*, 2017;  
45 Primalasari *et al.*, 2019; Saediman *et al.*, 2021). Smoked fish processing has been known for a long time  
46 because of its traditional processing techniques, simplicity, ease of implementation, and low cost  
47 (Islamiyah, 2021). In North Sulawesi, Indonesia, smoked fish commonly known as *fufu* is traditionally  
48 made from skipjack (*Katsuwonus pelamis*); *julung julung* (*Hemiramphus far*) (Berhimpon *et al.*, 2018);  
49 and scad fish (*Decapterus* spp.) known as *pinekuhe* (Ansar & Ijong, 2021). The smoked fish product of  
50 *julung julung* is a typical product from North Sulawesi and Gorontalo, commonly called *roa* or *galavea*  
51 (Azis & Akolo, 2020).

52 The study's reported that the conventional smoking process has disadvantages such as smoking time,  
53 concentration of carcinogenic substances, temperature, and inconsistent product quality (Racovita *et al.*,  
54 2020). Conventional smoking of food products has been shown to produce carcinogenic components  
55 such as polycyclic aromatic hydrocarbons (Jinadasa *et al.*, 2020). Benzo(a)pyrene (C<sub>20</sub>H<sub>12</sub>) is one of the  
56 carcinogenic compounds produced from traditional smoking products (Jinadasa *et al.*, 2020). One  
57 method to reduce carcinogenic compounds in smoked fish products is to use liquid smoke in the  
58 smoking process (Nithin *et al.*, 2020; Xin *et al.*, 2021).

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

66 Previous research reported that smoked fish dipped in liquid smoke for 15 minutes had a total plate  
67 count value of  $4.7 \times 10^4$  CFU/g on day six and moisture content below 60% during storage (Suroso *et*  
68 *al.*, 2018). Dipping fish in liquid smoke with a concentration of 15% and a dipping time of 60 minutes  
69 showed a bacterial colony of  $2.12 \times 10^2$  CFU/g (Ali *et al.*, 2021). The Indonesian National Standard on  
70 smoked fish requires a maximum total plate count of  $5 \times 10^4$  CFU/g and a maximum moisture content of  
71 60% (Indonesia Standardization Agency, 2013). Another study reported that smoked fish processed

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

80

#### 81 Materials and methods

82 The main materials of the study were *julung julung* fish (*Hemiramphus* sp.), corn cob (*Zea mays* L.)  
83 from Gangga Island in Likupang, North Sulawesi, Indonesia. This study includes the preparation of  
84 liquid smoke from corn cobs waste, preparing fish (weeding and filleting), and treating fish fillets by  
85 dip in liquid smoke. Fresh *julung julung* fish was obtained from traditional fishermen in Manado, North  
86 Sulawesi, Indonesia (Fig. 1). The fish was placed in a cool box and given ice in a ratio of 1:1 (ice:fish)  
87 (Mishra *et al.*, 2021). Then, the fish was washed, cleaned, and filleted. The cleaned fillets were dipped  
88 in a 5% salt solution for 30 minutes. The concentration of liquid smoke from corn cob waste was 0,8%  
89 (Berhimpon *et al.*, 2018).

90



91

92

93

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

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%.

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

**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).

97

#### 98 Liquid smoke manufacturing process

99 Making liquid smoke refers to modified research (Aladin *et al.*, 2018). Modifications were made to the  
100 equipment used in the pyrolysis process. Dry corn cobs were weighed to determine fuel efficiency. Corn  
101 cobs are gradually burned into the combustion furnace, at 400 °C and have a pyrolysis time of about  
102 120 minutes. The furnace was closed to prevent smoke from escaping from the tank. The smoke  
103 generated from the combustion flows through a pipe connected to a storage tank covered with ice cubes.  
104 The smoke that passes through the pipe will become cold, so condensation occurs, turning the smoke  
105 into liquid. The smoke that has been formed is collected into a container attached to the end of the pipe.  
106 The liquid smoke obtained is then allowed to settle the tar formed for three weeks and filtered (using  
107 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.

#### 108 Total volatile bases assay

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

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

117

118

119 **Moisture content assay**

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

$$125 \quad \text{Moisture (\%)} = \frac{B (g) - C (g)}{B (g) - A (g)} \times 100\%$$

126  
127 **Water activity (Aw) assay**

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

133  
134 **pH assay**

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

139  
140 **Phenol level assay**

141 Samples were extracted by adding 1 mL ethanol p.a., 5 mL distilled water, and 0.5 mL Folin Ciocalteu  
142 50%, homogenized, and allowed to stand for 5 minutes. Then, 1 mL of 5% Na<sub>2</sub>CO<sub>3</sub> and left in the dark  
143 for  $\pm 60$  minutes with gallic acid as a standard. The absorbance value was read on a spectrophotometer  
144 (Thermo Scientific Genesys 50) with a wavelength of 725 nm. Total phenolics were expressed as mg/g  
145 (Indonesia Standardization Agency, 2004; Muliadin *et al.*, 2022).

$$146 \quad \text{Total Phenol } \left(\frac{mg}{g}\right) = \frac{\text{Gallic acid equivalence } \left(\frac{mg}{L}\right) \times \text{Volume (L)}}{\text{Sample weight (g)}}$$

147  
148 **Polycyclic aromatic hydrocarbon (PAH) assay**

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

the mixture was boiled for 4 hours in a soxhlet. 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<sub>2</sub>O-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<sup>-1</sup> 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<sup>-1</sup> 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

179 **Statistical analysis**

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

185  
186 **Results and discussion**

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

193  
194 **Total volatile bases**

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

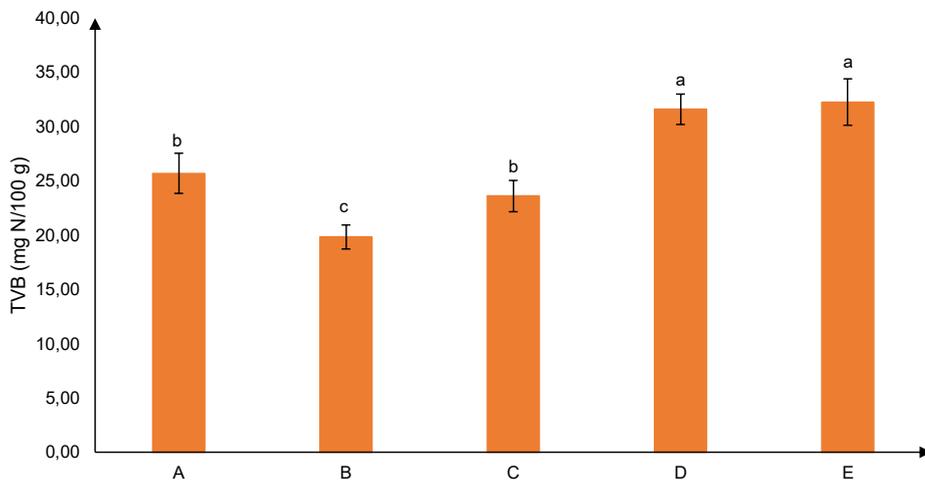


Fig. 2. Total volatile bases of *julung julung* smoked fillet

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 & Yuniarta, 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).

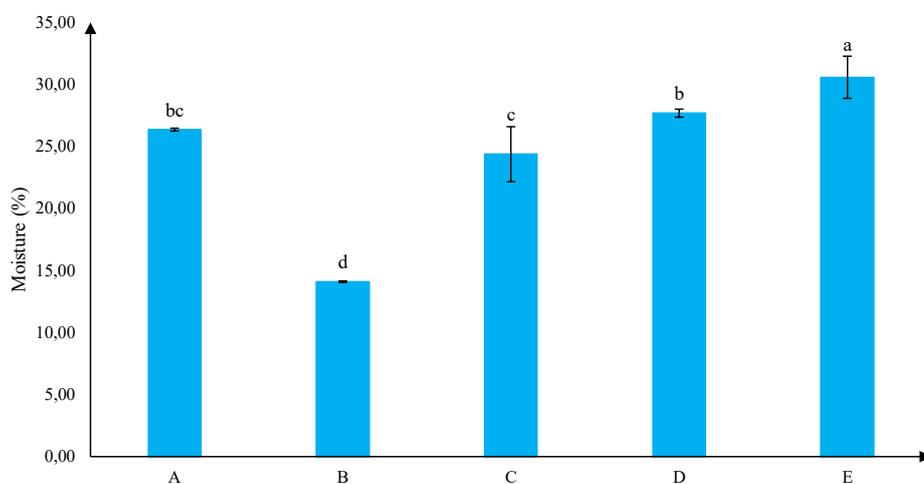
226 The European standard (European Market Observatory for Fisheries and Aquaculture, 2020) states that  
 227 the limit range of TVB value for smoked fish is 25 – 35 mg N/100 g. All treatments' TVB value of the  
 228 smoked fish fillets still met the European standard. Previous studies reported TVB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

235

236 **Moisture content**

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

242



243

244

245

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

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

249 value in smoked fish products from all treatments still meets the Indonesian National Standard No.  
250 2725:2013 on smoked fish, which is a maximum of 60% (Indonesia Standardization Agency, 2013).  
251 Darianto *et al.* (2018) reported that the smoking process can reduce the moisture content of fish to below  
252 40%, which can help preserve it longer. The treatment of the smoking method with liquid smoke with  
253 corn cob (Treatment A, B, C) has a lower moisture content when compared to the treatment of the  
254 conventional smoking method (Treatment D and E). This result is because the smoking chamber is not  
255 fully enclosed in the conventional smoking method, so the heat generated could be more optimal.  
256 Suboptimal heat can increase moisture content and cause the moisture content of smoked fish to  
257 decrease only slightly (Amos & Paulina, 2017). Whereas in the liquid smoke smoking method,  
258 temperature and humidity can be controlled better so that the moisture content of the product can be  
259 reduced efficiently (Salindeho & Lumoindong, 2017).

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

270

#### 271 **Water activity (Aw)**

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

281

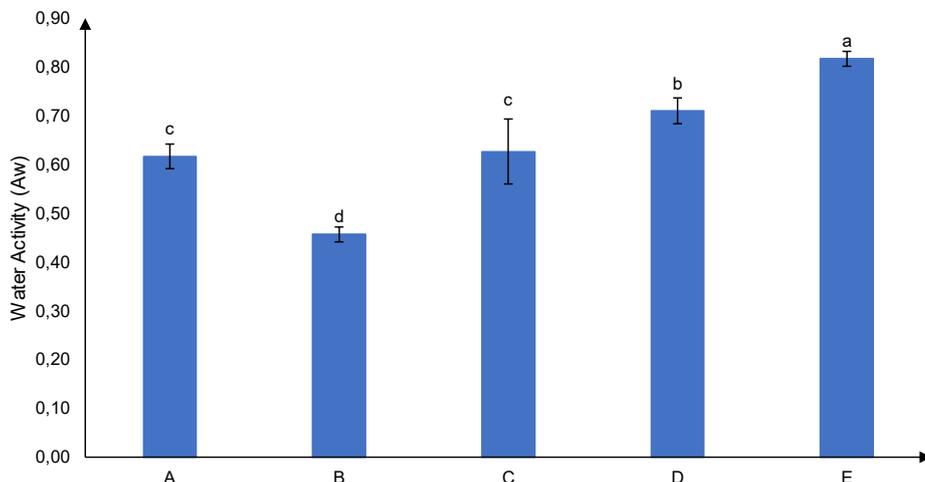


Fig. 4. Water activity of *julung julung* smoked fillet

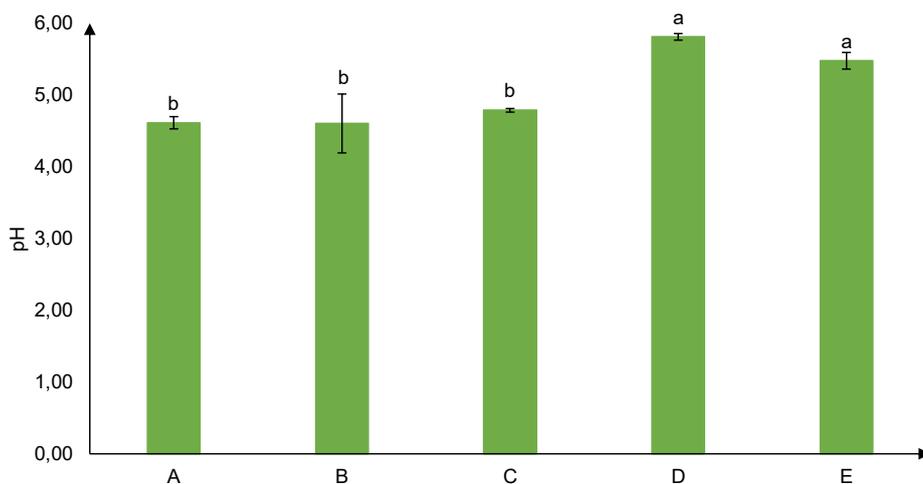
Fig. 4 shows the analysis of the variance of smoked fish fillet *julung julung* with different smoking method treatments affecting water activity ( $p < 0.05$ ). The water activity value of smoked fish fillet ranged from 0.46 – 0.82, with the lowest water activity in treatment B. The treatment of the 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

305 method. Another study also reported that using liquid smoke can reduce pH caused by the condensation  
 306 of organic acids in the smoking process (Puke & Galoburda, 2020). The pH value of smoked fish  
 307 correlates with the phenol content contained in the smoke component, and an increase in phenol content  
 308 causes a decrease in pH (Berhimpon *et al.*, 2018).  
 309



310  
 311 **Fig. 5. pH of julung julung smoked fillet**  
 312

313 **Phenol level**

314 Phenol is a compound found in wood smoke. Phenol compounds enter food through diffusion and  
 315 capillary action, affecting taste, color, and aroma and extending shelf life (Remy *et al.*, 2016). In  
 316 addition, phenol compounds are also reported to act as antioxidants that can prevent rancidity in fish  
 317 meat (Sérot *et al.*, 2004). Leksono *et al.* (2020) also noted that the higher the phenol level in smoke, the  
 318 stronger the flavor and aroma of smoked fish. The analysis of the variance of smoked fish fillets julung  
 319 julung with different smoking method treatments affecting phenol level ( $p < 0.05$ ). The phenol level of  
 320 smoked fish fillet ranged from 4.42 to 16.11 mg/g (Fig. 6). The treatment of the smoking method with  
 321 corn cob liquid smoke (treatments A, B, and C) had higher phenol level when compared to the  
 322 conventional smoking method (treatments D and E).  
 323

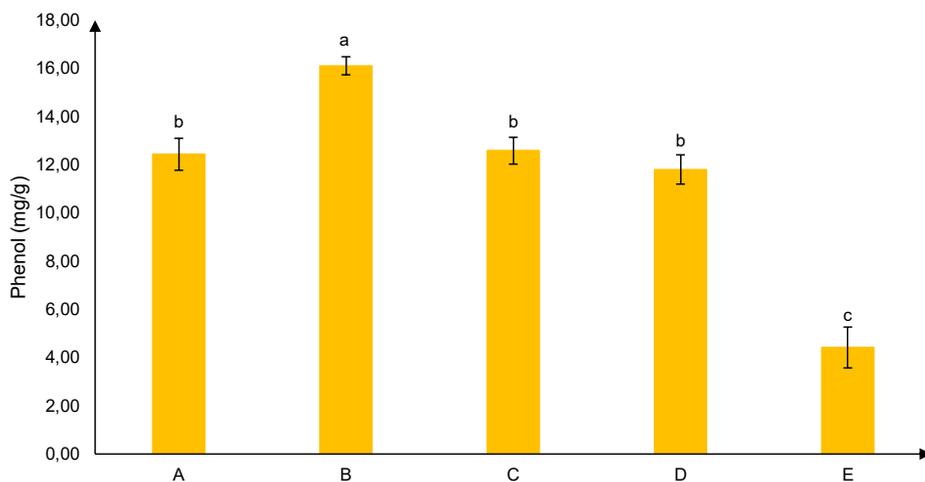


Fig. 6. Phenol level of *julung julung* smoked fillet

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 <i>Julung Julung</i> 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.

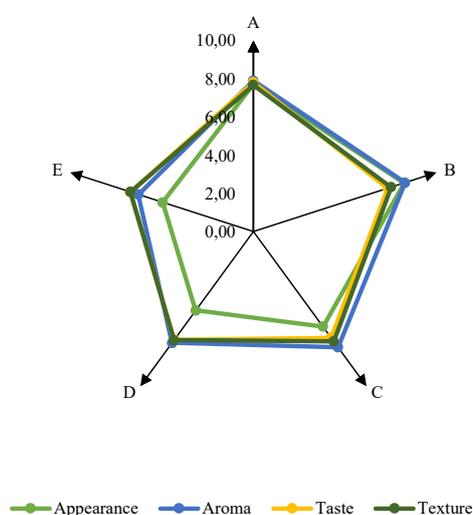
### Sensory assessment

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

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

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

382



383

384

385

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

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

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

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

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

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

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

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

#### 439 440 **Conclusion**

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

#### 448 449 **Acknowledgment**

#### 450 451 **Author contributions**

#### 452 453 **Conflicts of interest**

454 The authors declare that there is no conflict of interest.

#### 455 456 **Highlights**

457 Effectiveness of using liquid smoke derived from corn cob waste in the smoking process of *julung*  
458 *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 °C and then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated for 4 hours at 90 °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

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#### 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.

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

40 The conventional smoking processes generally use hardwood as fuel, such as melon wood (Umar *et al.*,  
41 2018), mangrove wood (Cissoko *et al.*, 2020), teak wood (Daramola *et al.*, 2020), coconut shells, and  
42 coconut husks (Nugroho *et al.*, 2018). Corn cobs have also been reported to be used in the fish smoking  
43 process (Sukowati, 2023). According to literature, traditional fish smoking in North Sulawesi,  
44 Indonesia, uses smoke from burning coconut shells or wood charcoal (Landangkasiang *et al.*, 2017;  
45 Primalasari *et al.*, 2019; Saediman *et al.*, 2021). Smoked fish processing has been known for a long time  
46 because of its traditional processing techniques, simplicity, ease of implementation, and low cost  
47 (Islamiyah, 2021). In North Sulawesi, Indonesia, smoked fish commonly known as *fufu* is traditionally  
48 made from skipjack (*Katsuwonus pelamis*); *julung julung* (*Hemiramphus far*) (Berhimpon *et al.*, 2018);  
49 and scad fish (*Decapterus* spp.) known as *pinekuhe* (Ansar & Ijong, 2021). The smoked fish product of  
50 *julung julung* is a typical product from North Sulawesi and Gorontalo, commonly called *roa* or *galavea*  
51 (Azis & Akolo, 2020).

52 The study's reported that the conventional smoking process has disadvantages such as smoking time,  
53 concentration of carcinogenic substances, temperature, and inconsistent product quality (Racovita *et al.*,  
54 2020). Conventional smoking of food products has been shown to produce carcinogenic components  
55 such as polycyclic aromatic hydrocarbons (Jinadasa *et al.*, 2020). Benzo(a)pyrene (C<sub>20</sub>H<sub>12</sub>) is one of the  
56 carcinogenic compounds produced from traditional smoking products (Jinadasa *et al.*, 2020). One  
57 method to reduce carcinogenic compounds in smoked fish products is to use liquid smoke in the  
58 smoking process (Nithin *et al.*, 2020; Xin *et al.*, 2021).

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

66 Previous research reported that smoked fish dipped in liquid smoke for 15 minutes had a total plate  
67 count value of  $4.7 \times 10^4$  CFU/g on day six and moisture content below 60% during storage (Suroso *et al.*,  
68 2018). Dipping fish in liquid smoke with a concentration of 15% and a dipping time of 60 minutes  
69 showed a bacterial colony of  $2.12 \times 10^2$  CFU/g (Ali *et al.*, 2021). The Indonesian National Standard on  
70 smoked fish requires a maximum total plate count of  $5 \times 10^4$  CFU/g and a maximum moisture content of  
71 60% (Indonesia Standardization Agency, 2013). Another study reported that smoked fish processed

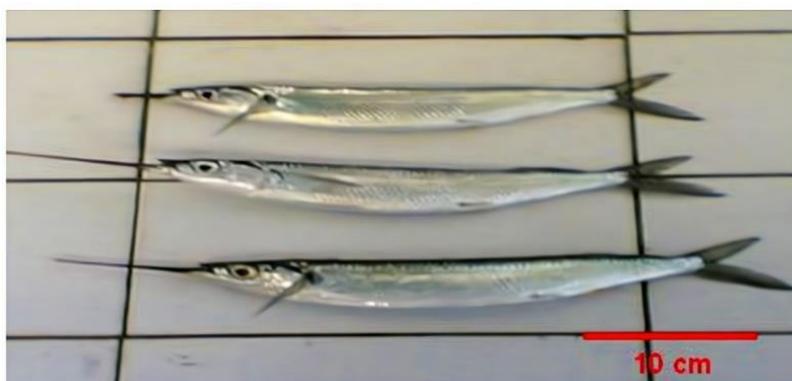
72 with liquid smoke had a moisture content of 47.63%, phenol content of 12.62%, and pH of 4.8  
73 (Berhimpon *et al.*, 2018). The results of these studies indicate that liquid smoke is good to apply to  
74 smoked fish products. This study characterized smoked fish fillets of *julung julung* (*Hemiramphus*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

80

#### 81 **Materials and methods**

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

90



91

92

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

93

94 The treatment in this study was applying liquid smoke from corn cobs waste to the smoking process  
95 of *julung julung* fillets, which was compared with the conventional method (using coconut wood,  
96 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 °C.
- (E) : Fresh fillets were smoked with the conventional method using corncob for 6 hours at 90 °C.

97

98 **Liquid smoke manufacturing process**

99 Making liquid smoke refers to modified research (Aladin *et al.*, 2018). Modifications were made to the  
100 equipment used in the pyrolysis process. Dry corn cobs were weighed to determine fuel efficiency. Corn  
101 cobs are gradually burned into the combustion furnace, at 400 °C and have a pyrolysis time of about  
102 120 minutes. The furnace was closed to prevent smoke from escaping from the tank. The smoke  
103 generated from the combustion flows through a pipe connected to a storage tank covered with ice cubes.  
104 The smoke that passes through the pipe will become cold, so condensation occurs, turning the smoke  
105 into liquid. The smoke that has been formed is collected into a container attached to the end of the pipe.  
106 The liquid smoke obtained is then allowed to settle the tar formed for three weeks and filtered (using  
107 Whatman filter paper No. 10) to obtain clear liquid smoke.

108 **Total volatile bases assay**

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

116 
$$TVB \left( \text{mg} \frac{\text{N}}{100\text{g}} \right) = \frac{(V_1 - V_0) \times N.HCl \times dilution \times 100}{sample\ weight\ (g)}$$

117

118

119 **Moisture content assay**

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

125 
$$\text{Moisture (\%)} = \frac{B (g) - C (g)}{B(g) - A (g)} \times 100\%$$

126

127 **Water activity (Aw) assay**

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

133

134 **pH assay**

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

139

140 **Phenol level assay**

141 Samples were extracted by adding 1 mL ethanol p.a., 5 mL distilled water, and 0.5 mL Folin Ciocalteu  
142 50%, homogenized, and allowed to stand for 5 minutes. Then, 1 mL of 5% Na<sub>2</sub>CO<sub>3</sub> and left in the dark  
143 for  $\pm 60$  minutes with gallic acid as a standard. The absorbance value was read on a spectrophotometer  
144 (Thermo Scientific Genesys 50) with a wavelength of 725 nm. Total phenolics were expressed as mg/g  
145 (Indonesia Standardization Agency, 2004; Muliadin *et al.*, 2022).

146 
$$\text{Total Phenol } \left(\frac{mg}{g}\right) = \frac{\text{Gallic acid equivalence } \left(\frac{mg}{L}\right) \times \text{Volume (L)}}{\text{Sample weight (g)}}$$

147

148 **Polycyclic aromatic hydrocarbon (PAH) assay**

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

the mixture was boiled for 4 hours in a soxhlet. 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<sub>2</sub>O-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<sup>-1</sup> 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<sup>-1</sup> 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

179 **Statistical analysis**

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

185  
186 **Results and discussion**

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

193  
194 **Total volatile bases**

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

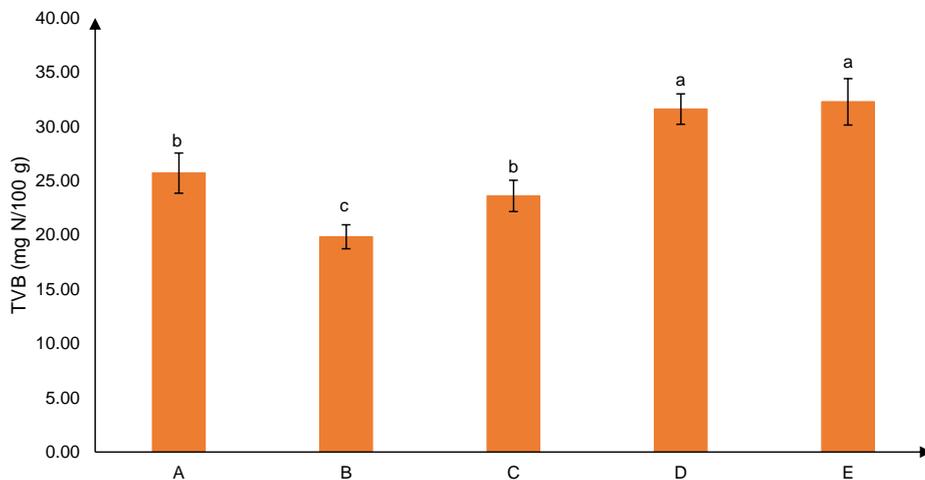


Fig. 2. Total volatile bases of *julung julung* smoked fillet

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).

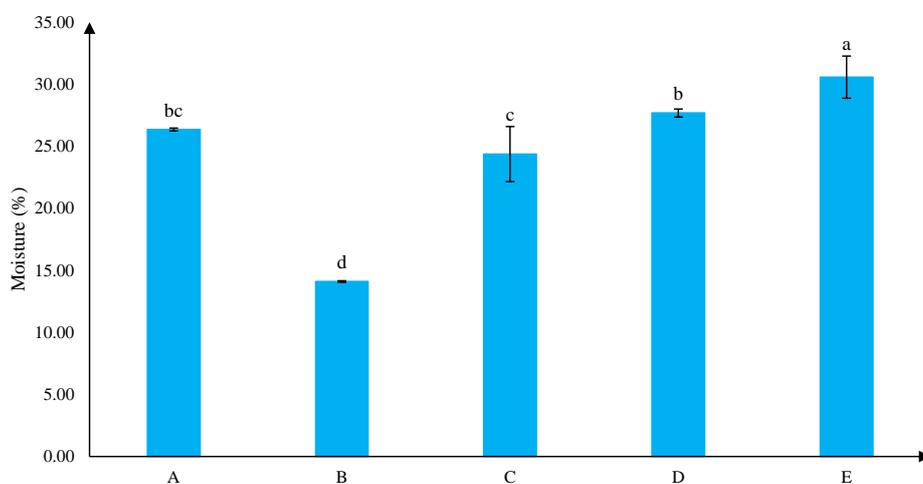
226 The European standard (European Market Observatory for Fisheries and Aquaculture, 2020) states that  
 227 the limit range of TVB value for smoked fish is 25 – 35 mg N/100 g. All treatments' TVB value of the  
 228 smoked fish fillets still met the European standard. Previous studies reported TVB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

235

236 **Moisture content**

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

242



243

244

245

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

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

249 value in smoked fish products from all treatments still meets the Indonesian National Standard No.  
250 2725:2013 on smoked fish, which is a maximum of 60% (Indonesia Standardization Agency, 2013).  
251 Darianto *et al.* (2018) reported that the smoking process can reduce the moisture content of fish to below  
252 40%, which can help preserve it longer. The treatment of the smoking method with liquid smoke with  
253 corn cob (Treatment A, B, C) has a lower moisture content when compared to the treatment of the  
254 conventional smoking method (Treatment D and E). This result is because the smoking chamber is not  
255 fully enclosed in the conventional smoking method, so the heat generated could be more optimal.  
256 Suboptimal heat can increase moisture content and cause the moisture content of smoked fish to  
257 decrease only slightly (Amos & Paulina, 2017). Whereas in the liquid smoke smoking method,  
258 temperature and humidity can be controlled better so that the moisture content of the product can be  
259 reduced efficiently (Salindeho & Lumoindong, 2017).

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

270

#### 271 **Water activity (Aw)**

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

281

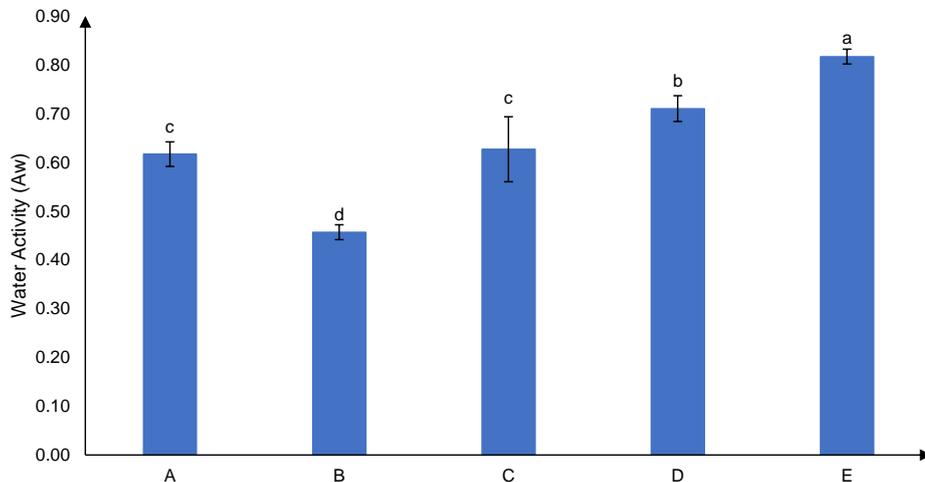


Fig. 4. Water activity of *julung julung* smoked fillet

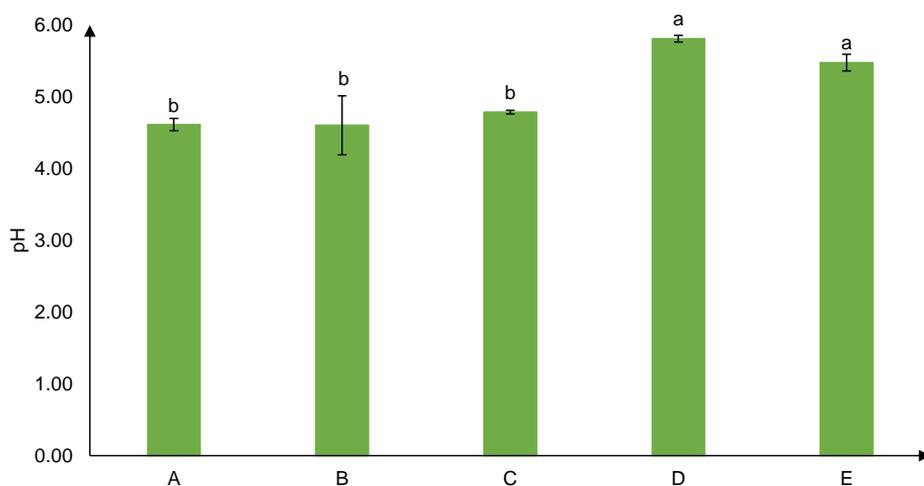
Fig. 4 shows the analysis of the variance of smoked fish fillet *julung julung* with different smoking method treatments affecting water activity ( $p < 0.05$ ). The water activity value of smoked fish fillet ranged from 0.46 – 0.82, with the lowest water activity in treatment B. The treatment of the 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

305 method. Another study also reported that using liquid smoke can reduce pH caused by the condensation  
 306 of organic acids in the smoking process (Puke & Galoburda, 2020). The pH value of smoked fish  
 307 correlates with the phenol content contained in the smoke component, and an increase in phenol content  
 308 causes a decrease in pH (Berhimpon *et al.*, 2018).  
 309



310  
 311 **Fig. 5.** pH of *julung julung* smoked fillet  
 312

313 **Phenol level**

314 Phenol is a compound found in wood smoke. Phenol compounds enter food through diffusion and  
 315 capillary action, affecting taste, color, and aroma and extending shelf life (Remy *et al.*, 2016). In  
 316 addition, phenol compounds are also reported to act as antioxidants that can prevent rancidity in fish  
 317 meat (Sérot *et al.*, 2004). Leksono *et al.* (2020) also noted that the higher the phenol level in smoke, the  
 318 stronger the flavor and aroma of smoked fish. The analysis of the variance of smoked fish fillets *julung*  
 319 *julung* with different smoking method treatments affecting phenol level ( $p < 0.05$ ). The phenol level of  
 320 smoked fish fillet ranged from 4.42 to 16.11 mg/g (Fig. 6). The treatment of the smoking method with  
 321 corn cob liquid smoke (treatments A, B, and C) had higher phenol level when compared to the  
 322 conventional smoking method (treatments D and E).  
 323

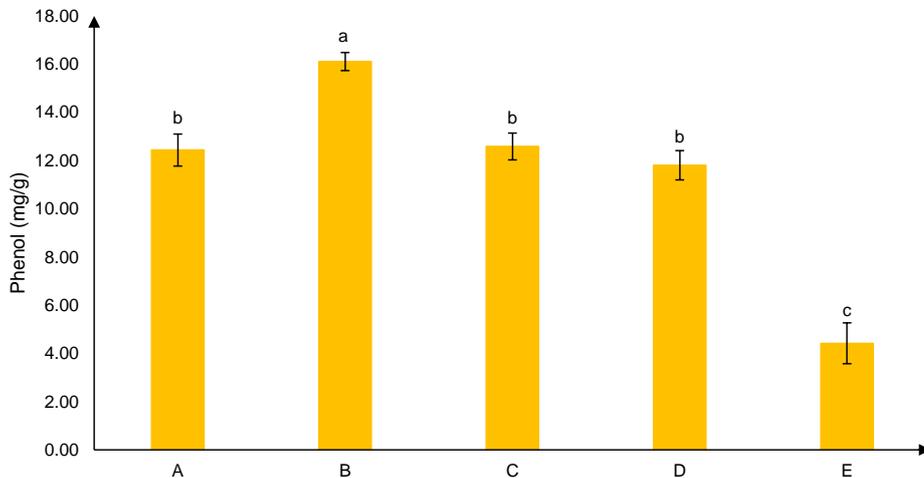


Fig. 6. Phenol level of *julung julung* smoked fillet

324  
325  
326

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

336 The study reported phenol levels in liquid smoke from corn cob waste of 0.335 mg/g (Swastawati *et al.*,  
337 2007) and 2.55% (Leviyani *et al.*, 2019). The phenol level of smoked fish with the conventional method  
338 of smoking with corn cobs is lower due to the high acid content, which can potentially reduce the phenol  
339 content of smoked fish (Swastawati *et al.*, 2012). Anggraini & Nurhazisa (2017) reported that the phenol  
340 content of liquid smoke from coconut shells was 3.04%, while liquid smoke from corn cob was 1.38%.  
341

342 **Polycyclic aromatic hydrocarbon (PAH) levels**

343 Polycyclic aromatic hydrocarbons are a group of organic compounds formed by incomplete combustion,  
344 such as wood. Therefore, the type of fuel used in the fish smoking process affects the PAH content of  
345 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 <i>Julung Julung</i> 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).

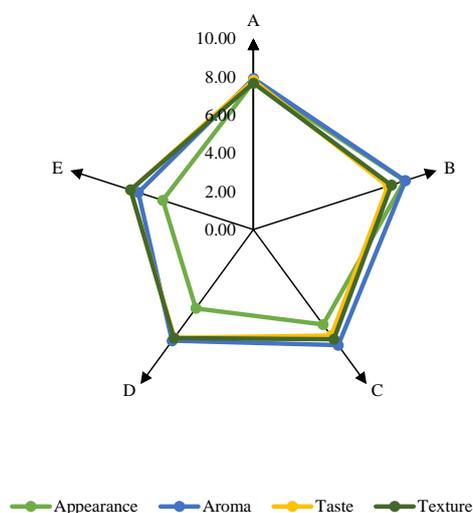
High PAH levels in smoked fish are due to the lignin content of the material used as fuel in the smoking process (Asamoah *et al.*, 2021). The lignin content in corn cobs is reported to be about 17 – 21% (Thangavelu *et al.*, 2018; Olajuyigbe *et al.*, 2019; Gandam *et al.*, 2022). The lignin content 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

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

382



383

384

385

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

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

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

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

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

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

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

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

439

#### 440 **Conclusion**

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

448

#### 449 **Acknowledgment**

450

#### 451 **Author contributions**

452

#### 453 **Conflicts of interest**

454 The authors declare that there is no conflict of interest.

455

#### 456 **Highlights**

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

459

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## **6. Bukti respons kepada reviewer terkait revisi manuskrip**

# 1 Characteristics of *Julung Julung* Smoked Fillets (*Hemiramphus* sp.) using Liquid 2 Smoke from Corn Cobs Waste

## 3 Abstract

4 This study aimed to obtain the best smoking method for *Julung Julung* fillets (*Hemiramphus* sp.) using liquid  
5 smoke from corn cob waste. This study used three different fillet treatment methods (dried and steamed) before  
6 liquid smoke application. This study also compared fish smoking between using liquid smoke with conventional  
7 methods (using shell, coconut wood, and corn cob). The parameters to determine the quality of smoked fish were  
8 Total Volatile Bases (TVB), moisture, water activity, pH, phenol levels, polycyclic aromatic hydrocarbon (PAH),  
9 and sensory assessment. The results showed that the TVB of smoked *Julung Julung* fillets ranged from 19.83 –  
10 32.27 mg N/100g. The moisture ranged from 14.10 – 30.57%. Water activity ranged from 0.46 – 0.82. pH ranged  
11 from 4.60 – 5.81. Phenol levels ranged from 4.42 – 16.11 mg/g. PAH levels are still below the standard required  
12 in the Indonesian National Standard. Panelists rated neutral to really like the appearance, aroma, taste, and texture  
13 of smoked fish. From these research, it can be concluded that treatment B is the best treatment, namely fresh fillets  
14 are first heated for 4 hours at 60 – 80 °C and then dipped in liquid smoke for 20 minutes. After that, the fillets were  
15 reheated for 4 hours at 90 °C.

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

## 18 Introduction

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

24 One of the corn cobs was used to make liquid smoke. Previous research reported that corn cobs can  
25 produce liquid smoke, a by-product of the pyrolysis of corn cob waste. The yield of liquid smoke from  
26 corn cobs is about 28.37%, with a pH value of 3.5 (Sriharti *et al.*, 2020). The particle size of corn cobs  
27 affects the yield of liquid smoke, with smaller particle sizes resulting in higher yields (Aladin *et al.*,  
28 2018). Swastawati *et al.* (2007) reported the phenolic content in liquid smoke produced from 335 mg/L  
29 corn cobs.

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

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

40 The conventional smoking processes generally use hardwood as fuel, such as melon wood (Umar *et al.*,  
41 2018), mangrove wood (Cissoko *et al.*, 2020), teak wood (Daramola *et al.*, 2020), coconut shells, and  
42 coconut husks (Nugroho *et al.*, 2018). Corn cobs have also been reported to be used in the fish smoking  
43 process (Sukowati, 2023). According to literature, traditional fish smoking in North Sulawesi,  
44 Indonesia, uses smoke from burning coconut shells or wood charcoal (Landangkasiang *et al.*, 2017;  
45 Primalasari *et al.*, 2019; Saediman *et al.*, 2021). Smoked fish processing has been known for a long time  
46 because of its traditional processing techniques, simplicity, ease of implementation, and low cost  
47 (Islamiyah, 2021). In North Sulawesi, Indonesia, smoked fish commonly known as *fufu* is traditionally  
48 made from skipjack (*Katsuwonus pelamis*); *julung julung* (*Hemiramphus far*) (Berhimpon *et al.*, 2018);  
49 and scad fish (*Decapterus* spp.) known as *pinekuhe* (Ansar & Ijong, 2021). The smoked fish product of  
50 *julung julung* is a typical product from North Sulawesi and Gorontalo, commonly called *roa* or *galavea*  
51 (Azis & Akolo, 2020).

52 The study's reported that the conventional smoking process has disadvantages such as smoking time,  
53 concentration of carcinogenic substances, temperature, and inconsistent product quality (Racovita *et al.*,  
54 2020). Conventional smoking of food products has been shown to produce carcinogenic components  
55 such as polycyclic aromatic hydrocarbons (Jinadasa *et al.*, 2020). Benzo(a)pyrene (C<sub>20</sub>H<sub>12</sub>) is one of the  
56 carcinogenic compounds produced from traditional smoking products (Jinadasa *et al.*, 2020). One  
57 method to reduce carcinogenic compounds in smoked fish products is to use liquid smoke in the  
58 smoking process (Nithin *et al.*, 2020; Xin *et al.*, 2021).

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

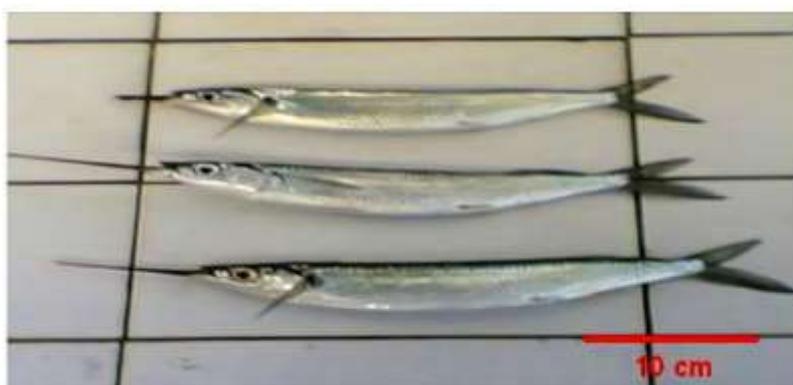
66 Previous research reported that smoked fish dipped in liquid smoke for 15 minutes had a total plate  
67 count value of  $4.7 \times 10^4$  CFU/g on day six and moisture content below 60% during storage (Suroso *et*  
68 *al.*, 2018). Dipping fish in liquid smoke with a concentration of 15% and a dipping time of 60 minutes  
69 showed a bacterial colony of  $2.12 \times 10^2$  CFU/g (Ali *et al.*, 2021). The Indonesian National Standard on  
70 smoked fish requires a maximum total plate count of  $5 \times 10^4$  CFU/g and a maximum moisture content of  
71 60% (Indonesia Standardization Agency, 2013). Another study reported that smoked fish processed

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

### 81 Materials and methods

82 The main materials of the study were *julung julung* fish (*Hemiramphus* sp.), corn cob (*Zea mays* L.)  
83 from Gangga Island in Likupang, North Sulawesi, Indonesia. This study includes the preparation of  
84 liquid smoke from corn cobs waste, preparing fish (weeding and filleting), and treating fish fillets by  
85 dip in liquid smoke. Fresh *julung julung* fish was obtained from traditional fishermen in Manado, North  
86 Sulawesi, Indonesia (Fig. 1). The fish was placed in a cool box and given ice in a ratio of 1:1 (ice:fish)  
87 (Mishra *et al.*, 2021). Then, the fish was washed, cleaned, and filleted. The cleaned fillets were dipped  
88 in a 5% salt solution for 30 minutes. The concentration of liquid smoke from corn cob waste was 0.8%  
89 (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%.



91  
92 **Fig. 1.** *Julung julung* (*Hemiramphus* sp.)

93  
94 The treatment in this study was applying liquid smoke from corn cobs waste to the smoking process  
95 of *julung julung* fillets, which was compared with the conventional method (using coconut wood,  
96 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 °C.
- (E) : Fresh fillets were smoked with the conventional method using corncob for 6 hours at 90 °C.

**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).

97

## 98 Liquid smoke manufacturing process

99 Making liquid smoke refers to modified research (Aladin *et al.*, 2018). Modifications were made to the  
100 equipment used in the pyrolysis process. Dry corn cobs were weighed to determine fuel efficiency. Corn  
101 cobs are gradually burned into the combustion furnace, at 400 °C and have a pyrolysis time of about  
102 120 minutes. The furnace was closed to prevent smoke from escaping from the tank. The smoke  
103 generated from the combustion flows through a pipe connected to a storage tank covered with ice cubes.  
104 The smoke that passes through the pipe will become cold, so condensation occurs, turning the smoke  
105 into liquid. The smoke that has been formed is collected into a container attached to the end of the pipe.  
106 The liquid smoke obtained is then allowed to settle the tar formed for three weeks and filtered (using  
107 Whatman filter paper No. 10) to obtain clear liquid smoke.

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

## 108 Total volatile bases assay

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

**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.

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

117

118

119 **Moisture content assay**

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

$$125 \quad \text{Moisture (\%)} = \frac{B (g) - C (g)}{B (g) - A (g)} \times 100\%$$

126  
127 **Water activity (Aw) assay**

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

133  
134 **pH assay**

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

139  
140 **Phenol level assay**

141 Samples were extracted by adding 1 mL ethanol p.a., 5 mL distilled water, and 0.5 mL Folin Ciocalteu  
142 50%, homogenized, and allowed to stand for 5 minutes. Then, 1 mL of 5% Na<sub>2</sub>CO<sub>3</sub> and left in the dark  
143 for  $\pm 60$  minutes with gallic acid as a standard. The absorbance value was read on a spectrophotometer  
144 (Thermo Scientific Genesys 50) with a wavelength of 725 nm. Total phenolics were expressed as mg/g  
145 (Indonesia Standardization Agency, 2004; Muliadin *et al.*, 2022).

$$146 \quad \text{Total Phenol } \left(\frac{mg}{g}\right) = \frac{\text{Gallic acid equivalence } \left(\frac{mg}{L}\right) \times \text{Volume (L)}}{\text{Sample weight (g)}}$$

147  
148 **Polycyclic aromatic hydrocarbon (PAH) assay**

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

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

the mixture was boiled for 4 hours in a soxhlet. 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<sub>2</sub>O-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<sup>-1</sup> 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<sup>-1</sup> 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

179 **Statistical analysis**

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

185  
186 **Results and discussion**

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

193  
194 **Total volatile bases**

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

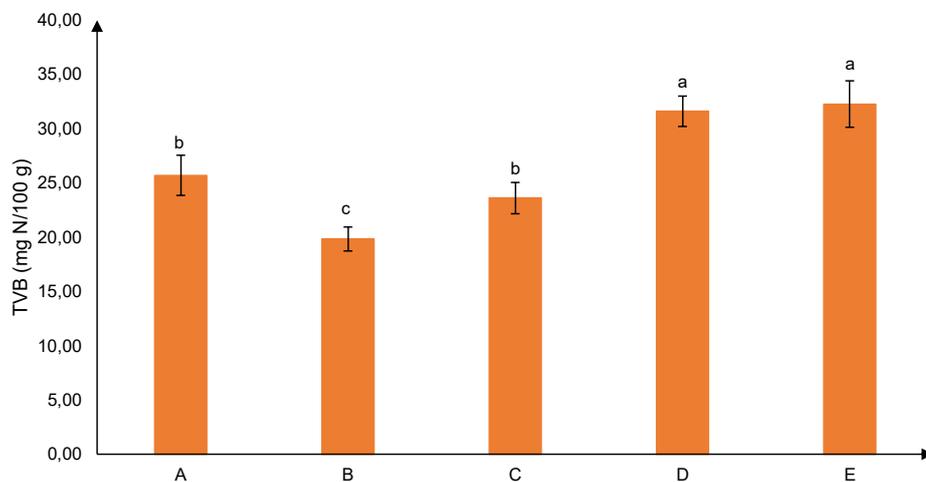


Fig. 2. Total volatile bases of *julung julung* smoked fillet

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 & Yuniarta, 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).

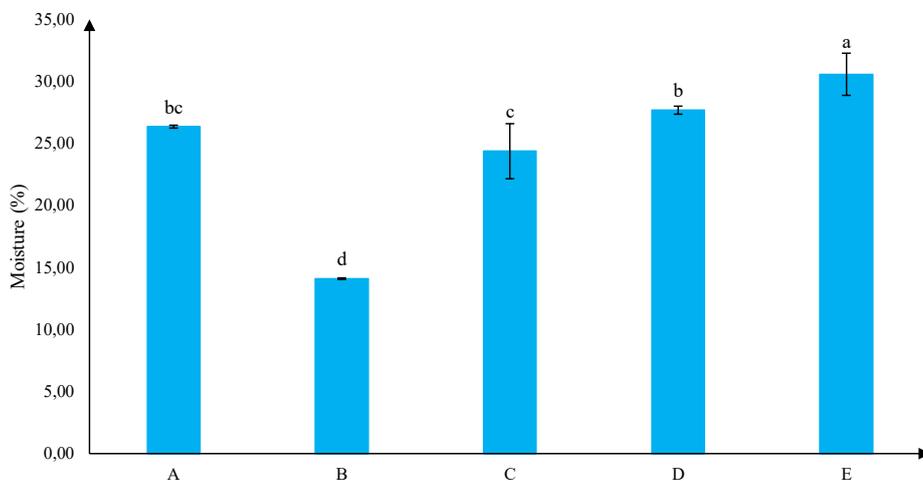
226 The European standard (European Market Observatory for Fisheries and Aquaculture, 2020) states that  
 227 the limit range of TVB value for smoked fish is 25 – 35 mg N/100 g. All treatments' TVB value of the  
 228 smoked fish fillets still met the European standard. Previous studies reported TVB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

235

236 **Moisture content**

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

242



243

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

245

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

249 value in smoked fish products from all treatments still meets the Indonesian National Standard No.  
250 2725:2013 on smoked fish, which is a maximum of 60% (Indonesia Standardization Agency, 2013).  
251 Darianto *et al.* (2018) reported that the smoking process can reduce the moisture content of fish to below  
252 40%, which can help preserve it longer. The treatment of the smoking method with liquid smoke with  
253 corn cob (Treatment A, B, C) has a lower moisture content when compared to the treatment of the  
254 conventional smoking method (Treatment D and E). This result is because the smoking chamber is not  
255 fully enclosed in the conventional smoking method, so the heat generated could be more optimal.  
256 Suboptimal heat can increase moisture content and cause the moisture content of smoked fish to  
257 decrease only slightly (Amos & Paulina, 2017). Whereas in the liquid smoke smoking method,  
258 temperature and humidity can be controlled better so that the moisture content of the product can be  
259 reduced efficiently (Salindeho & Lumoindong, 2017).

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

270

#### 271 **Water activity (Aw)**

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

281

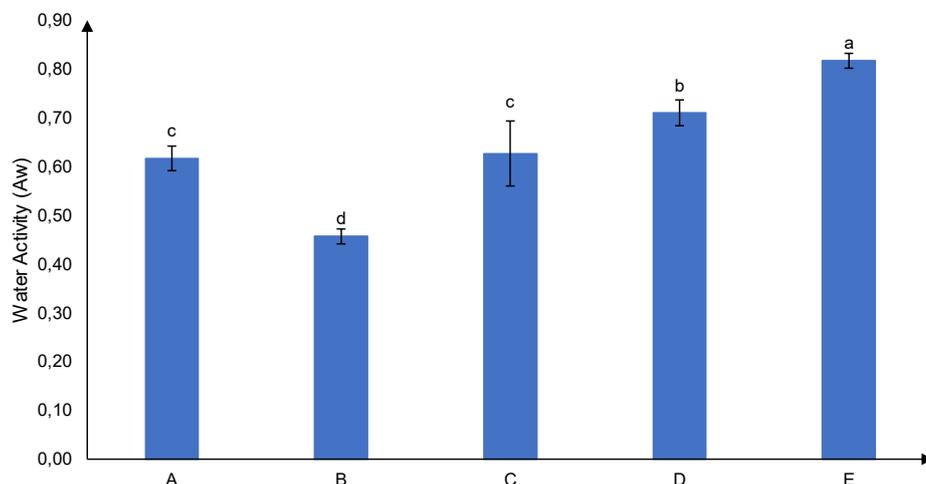


Fig. 4. Water activity of *julung julung* smoked fillet

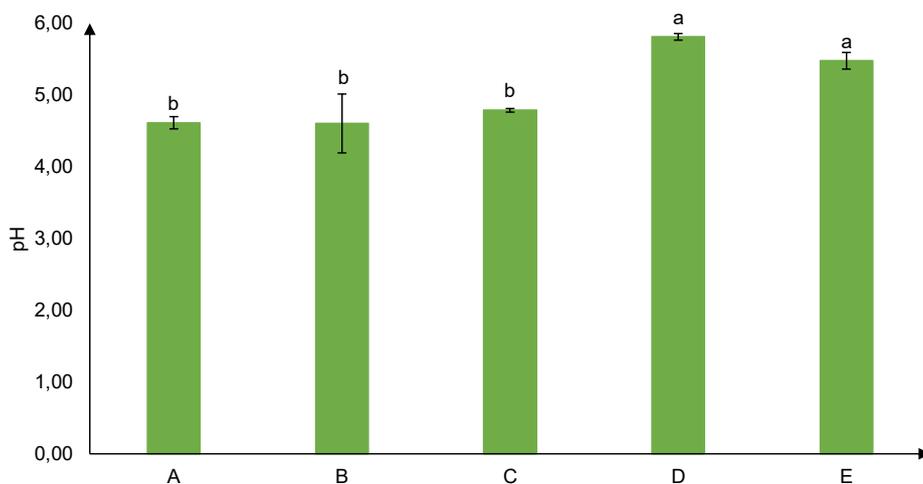
Fig. 4 shows the analysis of the variance of smoked fish fillet *julung julung* with different smoking method treatments affecting water activity ( $p < 0.05$ ). The water activity value of smoked fish fillet ranged from 0.46 – 0.82, with the lowest water activity in treatment B. The treatment of the 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

305 method. Another study also reported that using liquid smoke can reduce pH caused by the condensation  
 306 of organic acids in the smoking process (Puke & Galoburda, 2020). The pH value of smoked fish  
 307 correlates with the phenol content contained in the smoke component, and an increase in phenol content  
 308 causes a decrease in pH (Berhimpon *et al.*, 2018).  
 309



310  
 311 **Fig. 5. pH of julung julung smoked fillet**  
 312

313 **Phenol level**

314 Phenol is a compound found in wood smoke. Phenol compounds enter food through diffusion and  
 315 capillary action, affecting taste, color, and aroma and extending shelf life (Remy *et al.*, 2016). In  
 316 addition, phenol compounds are also reported to act as antioxidants that can prevent rancidity in fish  
 317 meat (Sérot *et al.*, 2004). Leksono *et al.* (2020) also noted that the higher the phenol level in smoke, the  
 318 stronger the flavor and aroma of smoked fish. The analysis of the variance of smoked fish fillets julung  
 319 julung with different smoking method treatments affecting phenol level ( $p < 0.05$ ). The phenol level of  
 320 smoked fish fillet ranged from 4.42 to 16.11 mg/g (Fig. 6). The treatment of the smoking method with  
 321 corn cob liquid smoke (treatments A, B, and C) had higher phenol level when compared to the  
 322 conventional smoking method (treatments D and E).  
 323

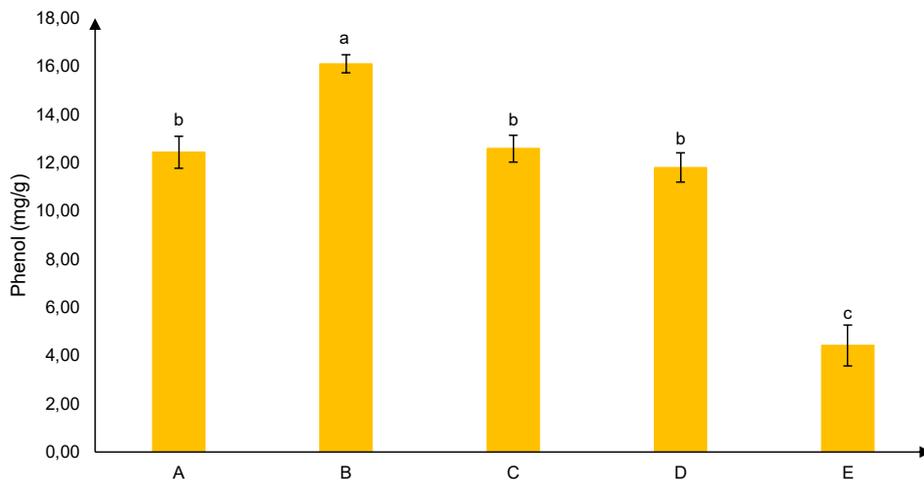


Fig. 6. Phenol level of *julung julung* smoked fillet

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 <i>Julung Julung</i> 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).

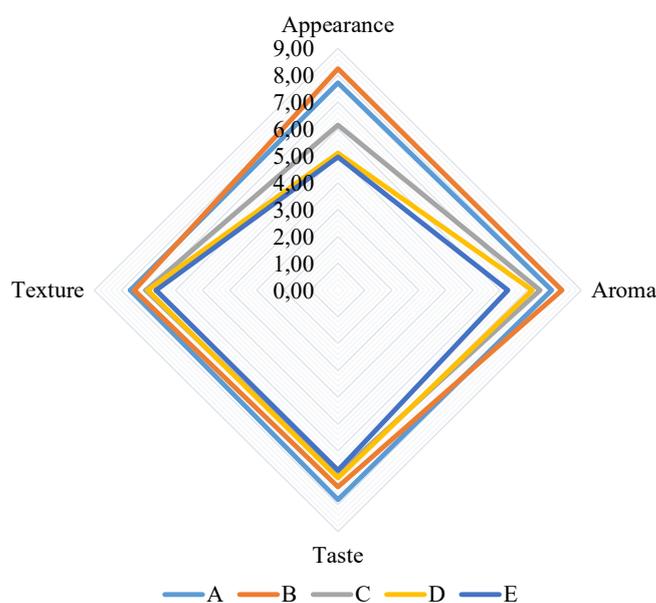
High PAH levels in smoked fish are due to the lignin content of the material used as fuel in the smoking process (Asamoah *et al.*, 2021). The lignin content in corn cobs is reported to be about 17 – 21% (Thangavelu *et al.*, 2018; Olajuyigbe *et al.*, 2019; Gandam *et al.*, 2022). The lignin content 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

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

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



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

383  
 384  
 385 Fig. 7 shows the analysis of the variance of the sensory assessment of the appearance of smoked *julung*  
 386 *julung fillets* with different smoking method treatments affecting the appearance of smoked fish  
 387 ( $p < 0.05$ ). The panelists' assessment of the appearance of smoked fish fillets ranged from 4.95 to 8.24,  
 388 with the highest panelists' assessment in treatment B. Based on the requirements of the Indonesian  
 389 National Standard, only treatments A and B met the minimum panelist assessment requirement of 7.  
 390 The moisture content factor is thought to have influenced the panelists' assessment of the appearance of  
 391 smoked fish, so panelists less favored treatment C with steaming. Moisture content can affect the  
 392 physical properties of fish, such as the appearance and texture of smoked fish (Baten *et al.*, 2020a).  
 393 Smoked fish with high moisture content will make the color of smoked fish look paler (Flick, 2010).  
 394 Treatments D and E were less favored by panelists, presumably because conventional smoking methods  
 395 produce darker products (dark brown), less bright and less shiny. Smoking fish with liquid smoke has

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

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

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

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

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

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

#### 438 439 **Conclusion**

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

#### 448 449 **Acknowledgment**

#### 450 451 **Author contributions**

#### 452 453 **Conflicts of interest**

454 The authors declare that there is no conflict of interest.

#### 455 456 **Highlights**

457 Effectiveness of using liquid smoke derived from corn cob waste in the smoking process of *julung*  
458 *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**

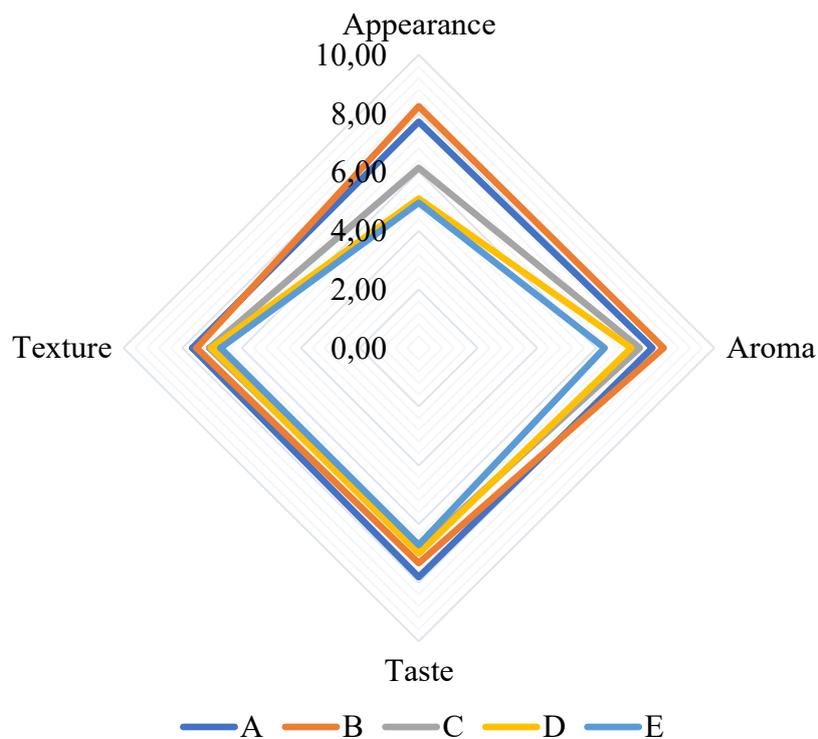
PAH testing is not replicated because because Data in table 2 using the HPLC method

### **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 °C and then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated for 4 hours at 90 °C.

### 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 °C 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 preparation??

**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 9<sup>th</sup> 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 routine 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 use standard curve?

**Response**

Yes, using gallic acid as standard 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**



Jefri Mand &lt;jefri.mandeno76@gmail.com&gt;

---

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

1 pesan

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**Research and Innovation in Food Science and Technology** <editorial@e-mail.sinaweb.net>

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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 Sambbeka, 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.

If there is anything else, please do not hesitate to contact us.

Truly yours,

Executive Managing Director of **Research and Innovation in Food Science and Technology**

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**8. Bukti manuskrip telah diterima dan akan dipublikasikan**



Jefri Mand &lt;jefri.mandeno76@gmail.com&gt;

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

1 pesan

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Balas Ke: Research and Innovation in Food Science and Technology &lt;jrifst@rifst.ac.ir&gt;

Kepada: jefri.mandeno76@gmail.com, tanodwendy@gmail.com

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## Certificate of Acceptance

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

Accepted: February 16, 2025

Manuscript ID: JRIFST-2406-1571 (R2)

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

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 (*Hemiramphus* sp.) from Corn Cobs Waste** ” has been accepted as " Original Paper " for publication and it will now be transferred to our production department.

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

Yours sincerely,

Dr. Bahareh Emadzadeh, PhD

Editor-in-Chief

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

Yours sincerely,

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Editor-in-Chief

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**9. Bukti permohonan revisi final sebelum  
dipublikasikan**



Jefri Mand &lt;jefri.mandeno76@gmail.com&gt;

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**JRIFST (#JRIFST-2406-1571)**

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21 Juni 2025 pukul 15.18

Kepada: jefri.mandeno76@gmail.com

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](mailto:jrifst@gmail.com) within **2 days**.

I look forward to hearing from you.

Best wishes,

Editorial Office

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## **10. Artikel published**

# 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 °C and then dipped in liquid smoke for 20 minutes. After that, the fillets were reheated for 4 hours at 90 °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

### 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.

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

40 The conventional smoking processes generally use hardwood as fuel, such as melon wood (Umar *et al.*,  
41 2018), mangrove wood (Cissoko *et al.*, 2020), teak wood (Daramola *et al.*, 2020), coconut shells, and  
42 coconut husks (Nugroho *et al.*, 2018). Corn cobs have also been reported to be used in the fish smoking  
43 process (Sukowati, 2023). According to literature, traditional fish smoking in North Sulawesi,  
44 Indonesia, uses smoke from burning coconut shells or wood charcoal (Landangkasiang *et al.*, 2017;  
45 Primalasari *et al.*, 2019; Saediman *et al.*, 2021). Smoked fish processing has been known for a long time  
46 because of its traditional processing techniques, simplicity, ease of implementation, and low cost  
47 (Islamiyah, 2021). In North Sulawesi, Indonesia, smoked fish commonly known as *fufu* is traditionally  
48 made from skipjack (*Katsuwonus pelamis*); *julung julung* (*Hemiramphus far*) (Berhimpon *et al.*, 2018);  
49 and scad fish (*Decapterus* spp.) known as *pinekuhe* (Ansar & Ijong, 2021). The smoked fish product of  
50 *julung julung* is a typical product from North Sulawesi and Gorontalo, commonly called *roa* or *galavea*  
51 (Azis & Akolo, 2020).

52 The study's reported that the conventional smoking process has disadvantages such as smoking time,  
53 concentration of carcinogenic substances, temperature, and inconsistent product quality (Racovita *et al.*,  
54 2020). Conventional smoking of food products has been shown to produce carcinogenic components  
55 such as polycyclic aromatic hydrocarbons (Jinadasa *et al.*, 2020). Benzo(a)pyrene (C<sub>20</sub>H<sub>12</sub>) is one of the  
56 carcinogenic compounds produced from traditional smoking products (Jinadasa *et al.*, 2020). One  
57 method to reduce carcinogenic compounds in smoked fish products is to use liquid smoke in the  
58 smoking process (Nithin *et al.*, 2020; Xin *et al.*, 2021).

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

66 Previous research reported that smoked fish dipped in liquid smoke for 15 minutes had a total plate  
67 count value of  $4.7 \times 10^4$  CFU/g on day six and moisture content below 60% during storage (Suroso *et al.*,  
68 2018). Dipping fish in liquid smoke with a concentration of 15% and a dipping time of 60 minutes  
69 showed a bacterial colony of  $2.12 \times 10^2$  CFU/g (Ali *et al.*, 2021). The Indonesian National Standard on  
70 smoked fish requires a maximum total plate count of  $5 \times 10^4$  CFU/g and a maximum moisture content of  
71 60% (Indonesia Standardization Agency, 2013). Another study reported that smoked fish processed

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

80

#### 81 **Materials and methods**

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

90



91

92

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

93

94 The treatment in this study was applying liquid smoke from corn cobs waste to the smoking process  
95 of *julung julung* fillets, which was compared with the conventional method (using coconut wood,  
96 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 °C.
- (E) : Fresh fillets were smoked with the conventional method using corncob for 6 hours at 90 °C.

97

98 **Liquid smoke manufacturing process**

99 Making liquid smoke refers to modified research (Aladin *et al.*, 2018). Modifications were made to the  
100 equipment used in the pyrolysis process. Dry corn cobs were weighed to determine fuel efficiency. Corn  
101 cobs are gradually burned into the combustion furnace, at 400 °C and have a pyrolysis time of about  
102 120 minutes. The furnace was closed to prevent smoke from escaping from the tank. The smoke  
103 generated from the combustion flows through a pipe connected to a storage tank covered with ice cubes.  
104 The smoke that passes through the pipe will become cold, so condensation occurs, turning the smoke  
105 into liquid. The smoke that has been formed is collected into a container attached to the end of the pipe.  
106 The liquid smoke obtained is then allowed to settle the tar formed for three weeks and filtered (using  
107 Whatman filter paper No. 10) to obtain clear liquid smoke.

108 **Total volatile bases assay**

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

116 
$$TVB \left( \text{mg} \frac{\text{N}}{100\text{g}} \right) = \frac{(V_1 - V_0) \times N.HCl \times dilution \times 100}{sample\ weight\ (g)}$$

117

118

119 **Moisture content assay**

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

125 
$$\text{Moisture (\%)} = \frac{B(g) - C(g)}{B(g) - A(g)} \times 100\%$$

126

127 **Water activity (Aw) assay**

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

133

134 **pH assay**

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

139

140 **Phenol level assay**

141 Samples were extracted by adding 1 mL ethanol p.a., 5 mL distilled water, and 0.5 mL Folin Ciocalteu  
142 50%, homogenized, and allowed to stand for 5 minutes. Then, 1 mL of 5% Na<sub>2</sub>CO<sub>3</sub> and left in the dark  
143 for  $\pm 60$  minutes with gallic acid as a standard. The absorbance value was read on a spectrophotometer  
144 (Thermo Scientific Genesys 50) with a wavelength of 725 nm. Total phenolics were expressed as mg/g  
145 (Indonesia Standardization Agency, 2004; Muliadin *et al.*, 2022).

146 
$$\text{Total Phenol } \left(\frac{mg}{g}\right) = \frac{\text{Gallic acid equivalence } \left(\frac{mg}{L}\right) \times \text{Volume (L)}}{\text{Sample weight (g)}}$$

147

148 **Polycyclic aromatic hydrocarbon (PAH) assay**

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

the mixture was boiled for 4 hours in a soxhlet. 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<sub>2</sub>O-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<sup>-1</sup> 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<sup>-1</sup> 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

179 **Statistical analysis**

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

185  
186 **Results and discussion**

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

193  
194 **Total volatile bases**

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

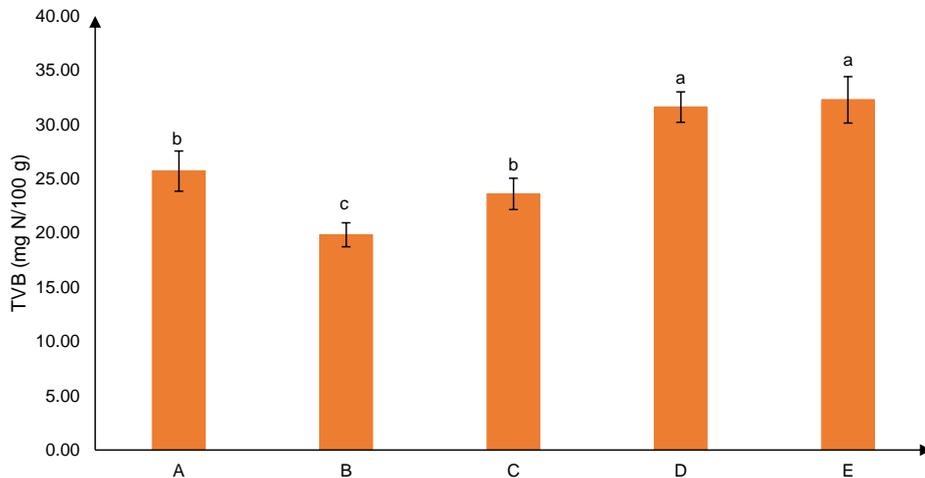


Fig. 2. Total volatile bases of *julung julung* smoked fillet

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).

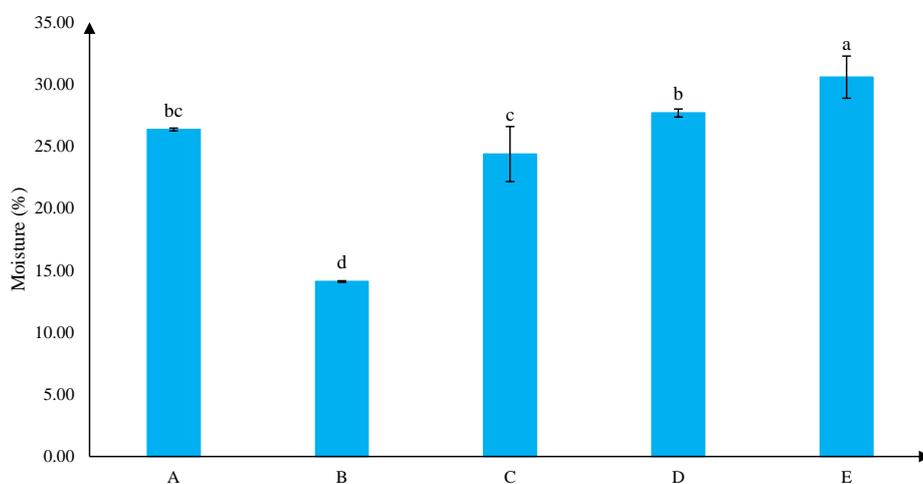
226 The European standard (European Market Observatory for Fisheries and Aquaculture, 2020) states that  
 227 the limit range of TVB value for smoked fish is 25 – 35 mg N/100 g. All treatments' TVB value of the  
 228 smoked fish fillets still met the European standard. Previous studies reported TVB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

235

236 **Moisture content**

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

242



243

244

245

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

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

249 value in smoked fish products from all treatments still meets the Indonesian National Standard No.  
250 2725:2013 on smoked fish, which is a maximum of 60% (Indonesia Standardization Agency, 2013).  
251 Darianto *et al.* (2018) reported that the smoking process can reduce the moisture content of fish to below  
252 40%, which can help preserve it longer. The treatment of the smoking method with liquid smoke with  
253 corn cob (Treatment A, B, C) has a lower moisture content when compared to the treatment of the  
254 conventional smoking method (Treatment D and E). This result is because the smoking chamber is not  
255 fully enclosed in the conventional smoking method, so the heat generated could be more optimal.  
256 Suboptimal heat can increase moisture content and cause the moisture content of smoked fish to  
257 decrease only slightly (Amos & Paulina, 2017). Whereas in the liquid smoke smoking method,  
258 temperature and humidity can be controlled better so that the moisture content of the product can be  
259 reduced efficiently (Salindeho & Lumoindong, 2017).

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

270

#### 271 **Water activity (Aw)**

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

281

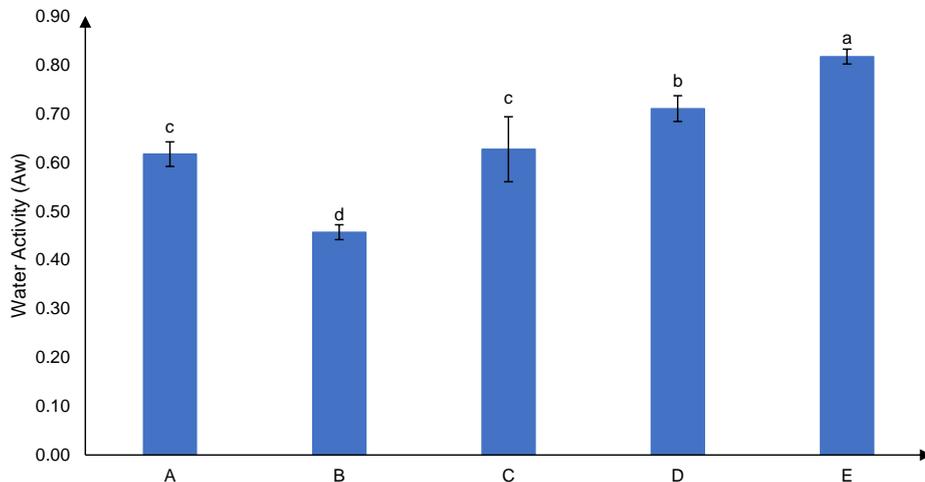


Fig. 4. Water activity of *julung julung* smoked fillet

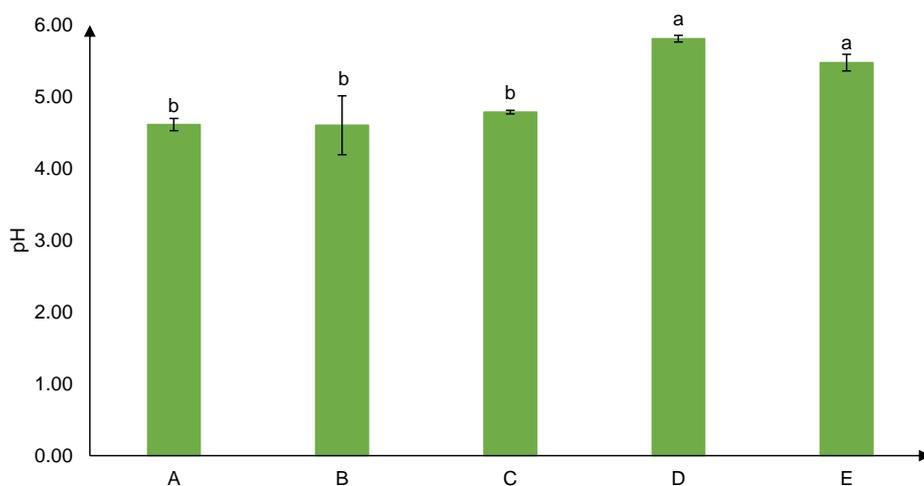
Fig. 4 shows the analysis of the variance of smoked fish fillet *julung julung* with different smoking method treatments affecting water activity ( $p < 0.05$ ). The water activity value of smoked fish fillet ranged from 0.46 – 0.82, with the lowest water activity in treatment B. The treatment of the 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

305 method. Another study also reported that using liquid smoke can reduce pH caused by the condensation  
 306 of organic acids in the smoking process (Puke & Galoburda, 2020). The pH value of smoked fish  
 307 correlates with the phenol content contained in the smoke component, and an increase in phenol content  
 308 causes a decrease in pH (Berhimpon *et al.*, 2018).  
 309



310  
 311 **Fig. 5.** pH of *julung julung* smoked fillet  
 312

313 **Phenol level**

314 Phenol is a compound found in wood smoke. Phenol compounds enter food through diffusion and  
 315 capillary action, affecting taste, color, and aroma and extending shelf life (Remy *et al.*, 2016). In  
 316 addition, phenol compounds are also reported to act as antioxidants that can prevent rancidity in fish  
 317 meat (Sérot *et al.*, 2004). Leksono *et al.* (2020) also noted that the higher the phenol level in smoke, the  
 318 stronger the flavor and aroma of smoked fish. The analysis of the variance of smoked fish fillets *julung*  
 319 *julung* with different smoking method treatments affecting phenol level ( $p < 0.05$ ). The phenol level of  
 320 smoked fish fillet ranged from 4.42 to 16.11 mg/g (Fig. 6). The treatment of the smoking method with  
 321 corn cob liquid smoke (treatments A, B, and C) had higher phenol level when compared to the  
 322 conventional smoking method (treatments D and E).  
 323

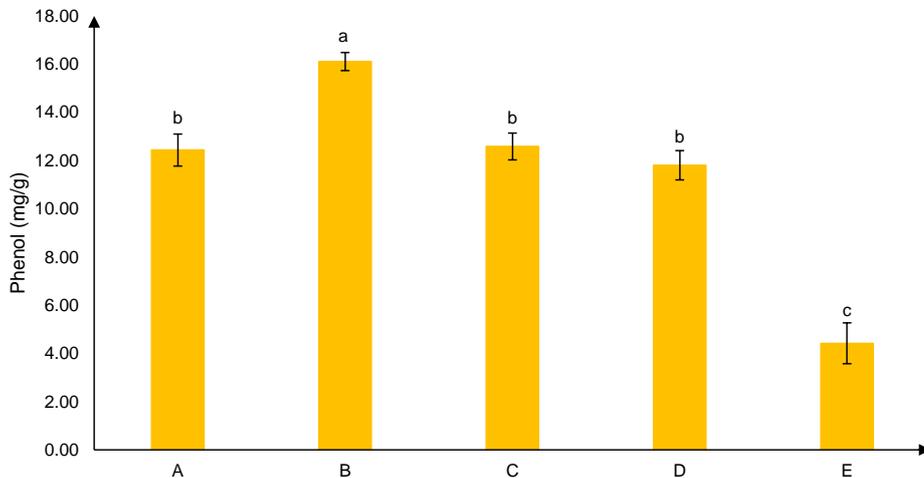


Fig. 6. Phenol level of *julung julung* smoked fillet

324  
325  
326

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%.

342 **Polycyclic aromatic hydrocarbon (PAH) levels**

343 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

345

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 <i>Julung Julung</i> 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).

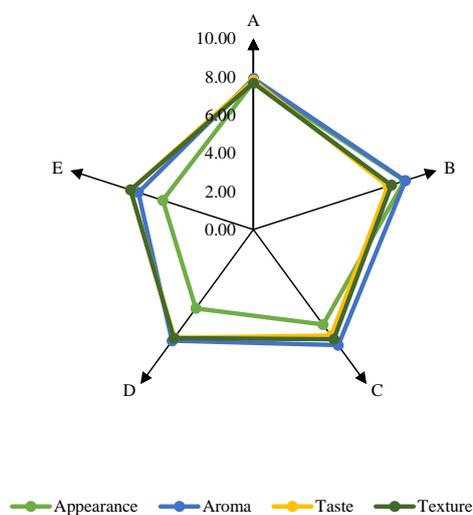
High PAH levels in smoked fish are due to the lignin content of the material used as fuel in the smoking process (Asamoah *et al.*, 2021). The lignin content in corn cobs is reported to be about 17 – 21% (Thangavelu *et al.*, 2018; Olajuyigbe *et al.*, 2019; Gandam *et al.*, 2022). The lignin content 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

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

382



383

384

385

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

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

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

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

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

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

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

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

439

#### 440 **Conclusion**

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

448

#### 449 **Acknowledgment**

450

#### 451 **Author contributions**

452

#### 453 **Conflicts of interest**

454 The authors declare that there is no conflict of interest.

455

#### 456 **Highlights**

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

459

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Authors: Jefri Anthonius Mandeno, Wendy Alexander Tanod, Eko Cahyono, Yana Sammbeka, Frets Jonas Rieuwpassa, Novalina Prianto 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 (Hemiramphus sp.) using Liquid Smoke from Corn Cobs Waste** has been published online via **Research and Innovation in Food Science and Technology**

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