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Physicochemical characterization of coconut shell liquid smoke and its potential as a natural preservative for fish balls

Karakterisasi fisikokimia asap cair dari tempurung kelapa serta potensinya sebagai pengawet alami bakso ikan

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ABSTRACT

Synthetic preservatives in food products pose potential health risks and environmental concerns, leading to an increasing demand for natural alternatives. This study evaluates the physicochemical properties of liquid smoke derived from coconut shells and its potential as a natural preservative for fish balls. The production of liquid smoke involved torrefaction at 250°C, followed by purification through distillation and adsorption with activated carbon. The liquid smoke was characterized by a pH of 2.86, a density of 1.058 g/mL, and a total titratable acidity of 8.95%, meeting the Indonesian National Standard for Crude Lignocellulose Liquid Smoke (SNI 8985:2021). GC-MS analysis revealed that acetic acid (80.87%) and phenol (8.90%) were the predominant compounds, contributing to its antimicrobial properties. The efficacy of liquid smoke as a preservative was tested on fish balls at concentrations of 3%, 5%, and 7%. The best preservation effect was observed at 7% concentration, which resulted in the lowest total plate count (2.35×10^6 CFU/g) after one day of storage at room temperature. These findings suggest that coconut shell-derived liquid smoke exhibits promising physicochemical characteristics and antimicrobial properties, making it a potential natural alternative to synthetic preservatives for food preservation.

ABSTRAK

Penggunaan pengawet sintetis dalam produk makanan menimbulkan potensi risiko kesehatan dan dampak lingkungan, sehingga meningkatkan permintaan akan alternatif alami. Penelitian ini mengevaluasi sifat fisikokimia asap cair yang berasal dari tempurung kelapa serta potensinya sebagai pengawet alami untuk bakso ikan. Produksi asap cair melibatkan proses torrefaksi pada suhu 250°C, diikuti dengan pemurnian melalui distilasi dan adsorpsi menggunakan karbon aktif. Asap cair yang dihasilkan memiliki pH 2.86, densitas 1.058 g/mL, dan total keasaman titrasi sebesar 8.95%, yang memenuhi Standar Nasional Indonesia untuk Asap Cair Lignoselulosa Mentah (SNI 8985:2021). Analisis GC-MS menunjukkan bahwa asam asetat (80.87%) dan fenol (8.90%) merupakan senyawa dominan yang berkontribusi terhadap sifat antimikrobanya. Efektivitas asap cair sebagai pengawet diuji pada bakso ikan dengan konsentrasi 3%, 5%, dan 7%. Efek pengawetan terbaik diamati pada konsentrasi 7%, yang menghasilkan jumlah total koloni bakteri terendah (2.35×10^6 CFU/g) setelah satu hari penyimpanan pada suhu ruang. Temuan ini menunjukkan bahwa asap cair dari tempurung kelapa memiliki karakteristik fisikokimia yang menjanjikan serta sifat antimikroba, sehingga berpotensi menjadi alternatif alami pengawet sintetis dalam pengawetan makanan.

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INTRODUCTION

Liquid smoke is a product obtained from a thermochemical process, such as pyrolysis or torrefaction, which involves heating organic materials in the absence of oxygen (Abdullah, 2017; Risfaheri, 2018; Alfarisi et al., 2023; Sunarno et al., 2024a; Sunarno et al., 2024b). This process produces gas, a solid (known as biochar), and a liquid referred to as liquid smoke, also known as wood vinegar or pyroligneous acid. Liquid smoke consisted of various bioactive compounds, such as phenols, carboxylic acids, and ketones, which provide several functional properties, including antioxidant, antimicrobial (Tuesta-Chavez et al., 2022), and anti-inflammatory (Arundina et al., 2022) effects. These properties enable its application as a biopesticide (Dewi et al., 2021; Purnama et al., 2024), food preservative (Handayani et al., 2020; Al'farisi et al., 2024; Mutamima et al., 2024), and disinfectant (Fardiaz & Rifqi, 2022). In terms of food preservation, liquid smoke has proven effective in extending the shelf life of food products, particularly meat, fish, and its processed products (Sunarno et al., 2024a).

In Indonesia, coconut shells are a plentiful agricultural waste that has not been fully utilized or explored for potential uses. Coconut shells consist of approximately 34% cellulose, 21% hemicellulose, 27% lignin, and 18% ash, making them a suitable material for the production of liquid smoke (Rasi & Seda, 2017). The lignin content contributes to the formation of phenolic compounds, which have antimicrobial and antioxidant properties (Ali et al., 2024). On the other hand, cellulose and hemicellulose contribute to the production of organic acids, particularly acetic acid, which is well-suited for food preservation (de Souza & Kawaguti, 2021; Wali & Abed, 2019).

Several studies have explored liquid smoke production and its application as a natural food preservative. Izza et al. (2022) investigated the production of liquid smoke from teak and pine wood sawdust through pyrolysis and applied it for tofu preservation. The study varied the wood ratios (1:0, 0:1, 1:1), distillation temperatures (100℃, 101-125℃, 126-150℃), and observation periods (2-6 days). The best results were obtained with a 1:1 teak to pine ratio, a distillation temperature of 126℃-150℃, and tofu preservation lasting up to 4 days. Frida et al. (2018) examined liquid smoke from corn cobs for tilapia preservation, with pyrolysis times of 1, 2, and 3 h. The best yield was achieved with a 2-h pyrolysis time, which was able to preserve tilapia for up to 20 days. Alamsyah et al. (2021) investigated the use of liquid smoke from coconut shells, corn cobs, and sugarcane bagasse to preserve yellowfin tuna fish balls. The most effective treatment obtained from sugarcane bagasse liquid smoke, which resulted in a 30-day shelf life and minimal bacterial growth at 4℃. Additionally, Zuraida et al. (2009) investigated the antibacterial activity of coconut shell liquid smoke as a potential substitute for traditional smoking methods in preserving fish balls. The study determined the Minimum Inhibitory Concentration (MIC) of coconut shell derived liquid smoke against *Pseudomonas aeruginosa* and *Staphylococcus aureus*. This study showed that liquid smoke effectively inhibited microbial activity. The application of 2.5% liquid smoke extended the shelf life of fish balls from 16 to 32 hours at 27–28℃ and up to 20 days at 4±1 ℃.

Previous studies on liquid smoke focused on its application as a food preservative, exploring its antimicrobial properties and effectiveness in extending the shelf life of food products. However, research discussing the use of coconut shell derived liquid smoke as a food preservative accompanied by its physico-chemical characterization to ensure its safety for consumption remains limited. This research addresses this gap by producing liquid smoke from coconut shells through a combination of torrefaction (a low-temperature pyrolysis process), distillation, and adsorption, followed by detailed physical and chemical analyses to verify its composition and safety. The resulting liquid smoke will be physically and chemically analyzed before being applied to preserve catfish (*Pangasius sp.*) fish balls. This approach not only aims to ensure the liquid smoke's efficiency as a natural preservative but also to confirm its safety for human consumption.

MATERIALS & METHODS

Materials and equipment

The materials used in this study include coconut shells, H_3PO_4 solution, distilled water, catfish (*Pangasius sp.*) fillet, tapioca flour, fine salt, shallots, white pepper, ice cubes, egg whites, 96% alcohol, phenolphthalein indicator, NaCl, Nutrient Agar (NA) media, and NaOH p.a (Emsure, Merck, Germany). The equipment used includes a torrefaction unit consisting of a reactor, condenser, and collection container, as illustrated in Figure 1. This unit was designed for the controlled pyrolysis process to produce liquid smoke. Additional equipment includes a distillation apparatus (Pyrex, Corning Inc., USA) for liquid smoke purification, a pH meter (Yinmik EZ-9901, Yinmik, China) for acidity measurement, an incubator (Memmert IN110, Memmert GmbH, Germany) for microbial analysis, an autoclave (Tomy SX-700, Tomy Digital Biology Co., Japan) for sterilization, a hot plate (IKA C-MAG HS 7, IKA-Werke, Germany) for heating, a colony counter (Suntex 570, Suntex Instruments Co., Taiwan) for bacterial count assessment, and a Gas Chromatography-Mass Spectrometry (GC-MS) (Shimadzu QP2010S, Shimadzu Corporation, Japan) for the chemical composition analysis of liquid smoke.

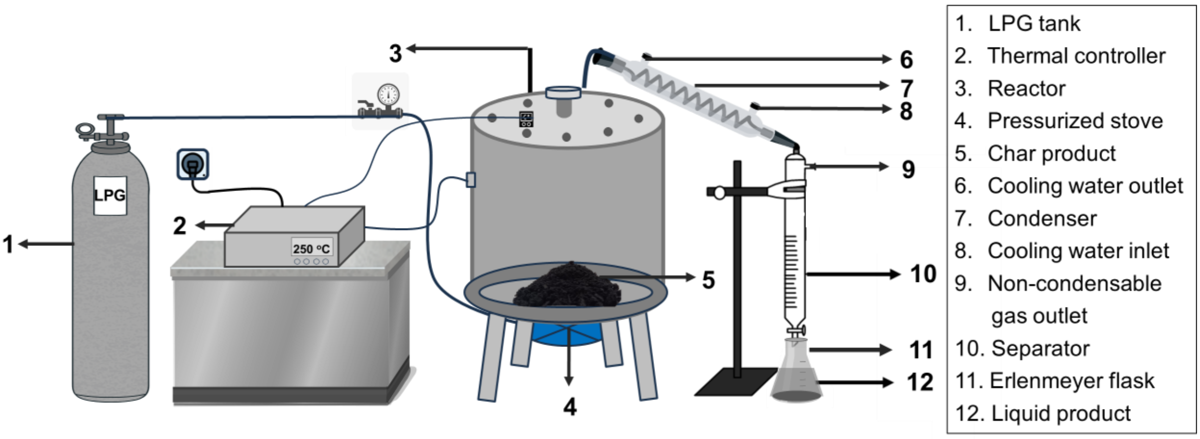

Figure 1. Schematic diagram of the torrefaction unit

Experimental methods

Raw material preparation

The coconut shells used in this study were obtained from local plantation in Garuda Sakti, Pekanbaru. Following the method of Nasruddin (2015), the coconut shells were sun-dried for 7 days before being crushed to approximately 2.5 × 2.5 cm in size to facilitate further processing.

Liquid smoke production and purification

Following the method of Arda et al. (2019), 1 kg of coconut shells were weighed and placed into a reactor equipped with a condenser and a pressurized stove as the heat source for the torrefaction process. The torrefaction was carried out at 250°C for 1 h. The char and the liquid product from the torrefaction process were collected for purification in the next step.

The liquid smoke was separated through a distillation process at 125°C (Pamori et al., 2015). The distilled liquid smoke was then adsorbed using activated carbon obtained from the torrefaction by-products in the previous step. The activated carbon was prepared by impregnating char (solid product of co-torrefaction process) with a saturated H_3PO_4 solution at a ratio of 1:10 for 24 h (Izza et al., 2022).

Liquid smoke yield

As outlined by Arda et al. (2019), the yield of liquid smoke was calculated by dividing the mass of produced liquid smoke (g) by the mass of raw materials (g) before the torrefaction process. The formula used to determine the yield is as follows:

$$\text{Yield (\%)} = \frac{Mass\ of\ produced\ liquid\ smoke}{Mass\ of\ raw\ material} \times 100\% \quad (1)$$

Liquid smoke physicochemical characterization

Liquid smoke was analyzed by measuring its pH, density, and total titratable acidity (TTA) following the procedures outlined in SNI 8985:2021 (Badan Standardisasi Nasional [BSN], 2021). The total titratable acidity of the liquid smoke was calculated using equation (2).

$$\% \text{ TTA} = \frac{V_{NaOH} \, x \, N_{NaOH} \, x \, Mw_{acetic\,acid} \, x \, F_p}{V_{sample} \, x \, 1000} \times 100\% \quad (2)$$

where V represents the volume of the NaOH titrant, N is the normality of the NaOH titrant, M_w is the molecular weight of acetic acid, and F_p denotes the dilution factor. GC-MS analysis was conducted to identify the chemical compounds contained in the liquid smoke.

Fish balls production and preservation

Following the method of Cahyaningati and Sulistiyati (2019) with modifications, the production of catfish fish balls began by mincing 900 g of catfish (*Pangasius sp.*) fillet. Salt, spices, and tapioca flour were then added and mixed until uniform. Ice cube was added during the final mixing stage. The dough was processed until it became a homogeneous mixture ready for shaping. The fish balls were boiled in water for approximately 7 min until they floated, and drained.

Following Parnanto and Atmaka (2010), the boiled fish balls were evenly sprayed with a liquid smoke solution at concentrations of 3, 5, and 7% with fish balls and liquid smoke ratio of 1:4 (w/v). This study involved four different treatment groups. F0 served as the control group, consisting of fish balls without any preservatives. F1 contained fish balls treated with 3% liquid smoke, while F2 included fish balls with 5% liquid smoke. Lastly, F3 represented fish balls treated with the highest concentration of liquid smoke at 7%. These treatments were designed to assess the effects of liquid smoke on the preservation and quality of the fish balls.

Sensory and microbial analysis of fish balls

According to the study by Parnanto and Atmaka (2010), organoleptic testing was conducted using a preference test based on a scoring method. This test involved 20 selected untrained panelists, utilizing visual, olfactory, and tactile sense to assess the product's quality, including the color, aroma, and texture of fish balls that had been treated with liquid smoke from coconut shells. The criteria for evaluating the organoleptic test are presented in Table 1. The mean score for each aspect was calculated. Additionally, Total Plate Count (TPC) analysis of fish ball samples was conducted in accordance with the SNI 7266:2017 method (BSN, 2017).

The hedonic score represents the panelists' level of preference for each attribute, with higher scores indicating greater acceptability. A score of 4 signifies the most desirable characteristics, such as a white color, normal aroma, and springy texture, while a score of 1 indicates the least favorable attributes, such as a dark brown color, spoiled aroma, and mushy texture. The scoring system allows for an objective comparison of sensory qualities based on the panelists' perceptions.

Table 1 Organoleptic test score

Score	Hedonic Characteristic		
	Color	Aroma	Texture
4	White	Normal	Springy
3	Grey	Mild smoky	Slightly springy
2	Brown	Tangy	Slightly mushy
1	Dark brown	Spoiled	Mushy

RESULTS & DISCUSSIONS

Liquid smoke physicochemical characterization

The results of the physico-chemical analysis for liquid smoke, which was produced through torrefaction, distillation, and adsorption processes, are shown in Table 2. The yield of the liquid smoke was recorded at 39.9%, indicating a highly efficient production process. Although SNI 8985:2021 (BSN, 2021) does not specify a yield requirement, this percentage reflects the effectiveness of the conversion of raw materials into liquid smoke, suggesting a favorable process efficiency. When comparing these results with previous studies, Anggraini (2017) reported a yield of 40% for liquid smoke produced at a pyrolysis temperature of 250°C, while Permanasari et al. (2020) achieved yield of 34.32% at pyrolysis temperatures of 400°C. The yield observed in this study (39.9%) is comparable to these findings.

Table 2. Liquid smoke analysis

Analysis	Unit	Value	SNI 8985:2021	Appropriateness level
pH	-	2.86	1.5 – 3	Within Standard
Density	g/mL	1.058	1.015 – 1.060	Within Standard
Total Titrated Acid (TTA)	%	8.95	8 - 15	Within Standard
Phenolic Content	%	8.90	2.00	Exceed Standard
Yield	%	39.9	-	-

Additionally, the pH value of 2.86 is within the acceptable range of 1.5 to 3 as specified by SNI, indicating that the liquid smoke possesses an appropriate level of acidity. According to Sahrum et al. (2021), the pH value of liquid smoke is related to the total titrated acid content; a higher total titrated acid content results in a lower pH value, and conversely, a lower total titrated acid content leads to a higher pH value. Izza et al. (2022) further emphasize that high-quality liquid smoke typically exhibits a low pH value. A lower pH indicates better quality of the produced liquid smoke and shelf life of preserved products. At lower pH values, bacterial growth is inhibited, enhancing the liquid smoke's effectiveness as a food preservative. The liquid smoke density was 1.058 g/mL, which also complies with the SNI standard of 1.015 to 1.060 g/mL. Furthermore, the total titrated acid content was 8.95%, falling within the SNI standard range of 8–15%.

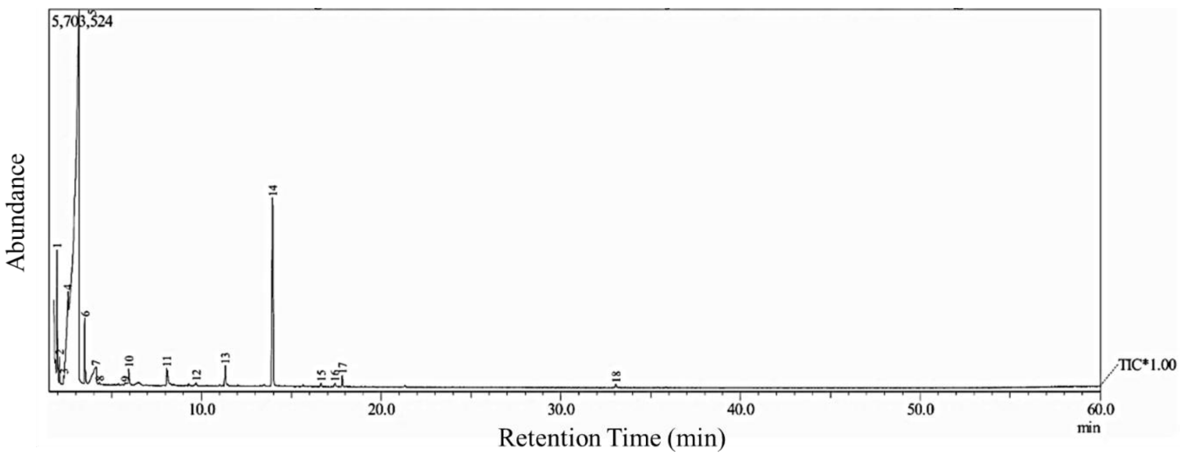

Figure 2. GC-MS chromatogram of liquid smoke in this study

Figure 2 shows the results from the GC-MS analysis of liquid smoke after purification, with details on the compounds and their percentage areas of liquid smoke constituents provided in Table 3. The most abundant compound was acetic acid, constituting 80.87% of the total composition. Acetic acid plays a crucial role as a natural preservative in liquid smoke, effectively inhibiting the growth of bacteria and other harmful microorganisms (Febriani et al., 2023). Additionally, phenol, which constitutes 8.90% of the liquid smoke, is a key component known for its antimicrobial properties and its contribution to the characteristic smoky flavor (Abdel-Naeem et al., 2021). However, its concentration should be carefully managed to ensure safe consumption. This can be achieved through controlled purification processes, such as re-distillation or selective adsorption using activated carbon, to reduce excessive phenol content while retaining its beneficial properties.

Table 3. GC-MS analysis of liquid smoke

Compounds	% Area
2-propanone-acetone	3.15
methyl-acetate	0.60
formic acid	0.19
acetic acid	80.87
propanoic acid	4.03
2-propanol	0.13
propylene-oxide	0.21
3-furaldehyde	0.84
butanoic acid	0.83
phenol	8.90
3-methyl-phenol	0.28
mequinol	0.43
4-nonen-2-yne, (E)	0.15

According to SNI 8985:2021 (BSN, 2021), the recommended maximum phenol content in liquid smoke for food applications is 2%, suggesting that further processing or dilution may be necessary for higher concentrations. Similarly, the European Food Safety Authority (EFSA) has adjusted the tolerable daily intake (TDI) of phenol from 1.5 mg/kg body weight per day to 0.5 mg/kg body weight per day, highlighting the importance of monitoring intake levels for long-term safety. In the context of food preservation, previous studies have reported that the acceptable phenol content in liquid smoke ranges from 0.1% to 16% (Maulina & br Karo, 2021). However, to comply with the SNI 8985:2021 (BSN, 2021) and minimize potential health risks, the phenol concentration should be diluted to below 2% or further purified before use in food applications.

Sensory and microbial analysis of fish balls

The organoleptic test results for the fish balls are shown in Figure 3. Organoleptic test results reveal that the control sample (F0) without preservatives received the highest scores across all parameters. For color, F0 scored 3.85, indicating a white and fresh appearance, while samples treated with liquid smoke showed a gradual decline in color scores as the concentration increased. F1 (3% liquid smoke) scored 3.7, while F2 (5%) and F3 (7%) scored 3.3 and 3.35, respectively, with the color becoming slightly browner as the concentration increased.

In terms of aroma, the control sample (F0) scored 3.8, reflecting a normal scent, while F1, F2, and F3 had progressively lower scores (3.25, 2.75, and 2.6, respectively), indicating a stronger smoky and pungent smell as the liquid smoke concentration increased. This suggests that higher concentrations of liquid smoke can negatively affect the aroma, making it less appealing. For texture, F0 also scored the highest at 3.8, indicating an ideal chewy texture. The texture remained acceptable in F1 (3.6) and F2 (3.5), but F3 (3.35) showed a slight decline, with the texture becoming slightly mushy at the highest concentration of liquid smoke. Overall, the addition of liquid smoke, especially at higher concentrations, had a noticeable impact on the organoleptic qualities, particularly in aroma and texture. However, F1 (3% liquid smoke) still maintained relatively high scores, indicating a balance between preservation and sensory quality.

A TPC analysis was conducted to determine the effect of liquid smoke addition on the bacterial growth of fish balls, with the results presented in Table 4. Table 4 shows that all fish ball samples, both with and without liquid smoke, have bacterial counts exceeding the maximum limit of SNI, which is 1 x 10^6 CFU/g. The control sample (fish balls without liquid smoke) had the highest bacterial count of 6.5 x 10^6 CFU/g on day 0, indicating rapid bacterial growth without any preservative. F1 (fish balls with 3% liquid smoke) had a lower bacterial count of 1.57 x 10^6 CFU/g on day 0, but this still exceeded the limit, and after one day, it increased to 3.38 x 10^6 CFU/g, showing that 3% liquid smoke did not sufficiently prevent bacterial growth. F2 (fish balls with 5% liquid smoke) also started above the limit with 2.18 x 10^6 CFU/g and rose to 7.4 x 10^6 CFU/g after one day, demonstrating that 5% liquid smoke was also inadequate for bacterial control. F3 (fish balls with 7% liquid smoke) had the lowest initial count at 1.35 x 10^6 CFU/g, closer to the SNI limit. On day 3, the count increased to 1.1 x 10^7 CFU/g. While 7% liquid smoke showed better bacterial inhibition than the lower concentrations, none of the treatments were able to keep bacterial levels below the SNI standard over time.

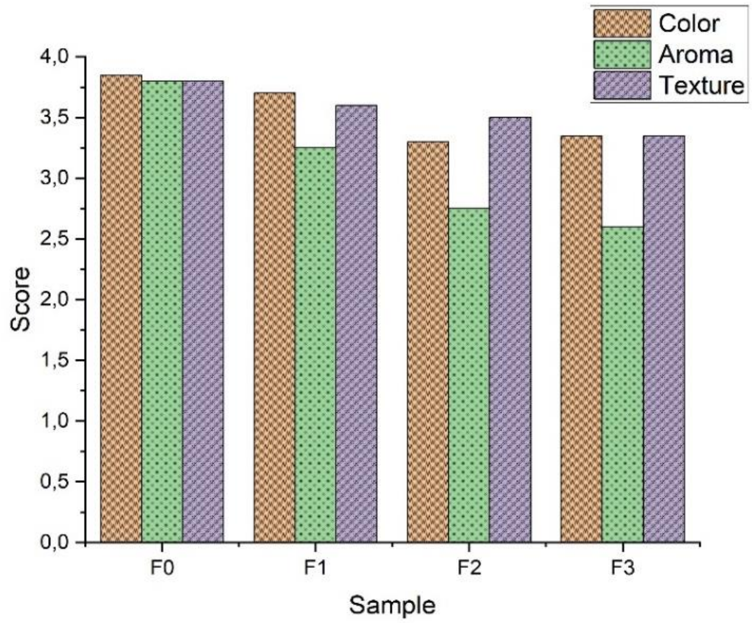

Figure 3. Fish balls organoleptic test result

Table 4. Total plate count analysis result

Sample	Unit	Preservation duration			
		0 Day	1 Days	3 Days	5 Days
F0	CFU/g	6.50×10^6	-	-	-
F1		2.18×10^6	7.40×10^6	-	-
F2		1.57×10^6	3.38×10^6	-	-
F3		1.35×10^6	2.35×10^6	1.1×10^7	-

Note. F0 : Control (Fish balls without preservative)
F1 : Fish balls + 3% liquid smoke
F2 : Fish balls + 5% liquid smoke
F3 : Fish balls + 7% liquid smoke

This analysis result indicates storing fish balls at room temperature is not recommended, as the high temperatures can accelerate bacterial growth, as demonstrated by the TPC results. To ensure the safety and quality of fish balls, it is essential to store them in a refrigerator or cooling unit. Cold storage slows bacterial growth, extending the shelf life and maintaining product safety. In regions with high daily temperatures and humidity levels, such as Pekanbaru, Indonesia, proper refrigeration is essential to prevent spoilage and maximize the effectiveness of preservatives like liquid smoke.

CONCLUSIONS

Based on the research conducted, it was found that the liquid smoke produced had properties meeting the SNI 8985:2021 standards, including a pH of 2.86, a density of 1.058 g/mL, and a total titrated acid content of 8.95%. However, the phenol content of 8.90% exceeds the maximum limit of 2% set by SNI for liquid smoke as raw material. This indicates that while the liquid smoke has desirable characteristics for preservation, further dilution or refinement may be necessary to ensure its safe application in food products. While the fish balls treated with liquid smoke exhibited favorable characteristics, the TPC slightly exceeded the SNI standard of 1 x 10^6 CFU/g. On day 0, the control fish balls (without preservatives) had a TPC of 6.50 x 10^6 CFU/g, whereas the treatment with 7% liquid smoke showed the most effective microbial reduction, achieving a TPC of 2.35 x 10^6 CFU/g after 1 day of storage on room temperature. These results highlight the potential of liquid smoke as a natural preservative, significantly reducing microbial growth. To enhance its effectiveness and align more closely with SNI standards, future research could explore optimized concentrations, combinations with other natural preservatives, or improved application methods. Additionally, adjusting storage conditions may further enhance microbial control while maintaining the sensory quality of the fish balls.

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