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The Use of Liquid Smoke from Coconut Shell as a Natural Food Preservative in the Processing of Flying Fish

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ABSTRACT

Liquid smoke is a product of condensation or vapor formation that occurs directly or indirectly from the combustion of materials containing lignin, cellulose, hemicellulose, and other carbon compounds. This research aims to understand the process of making liquid smoke from coconut shell, identify the components contained in the liquid smoke, and analyze the effect of storage duration on the quality of flying fish that has been soaked in liquid smoke, including tests for moisture content and phenol in the preservation process. Additionally, it evaluates the sensory quality, such as taste, smell, and texture, of the flying fish. The study used liquid smoke at various concentrations (0, 2, 4, 6%) with a 30-minute soaking period. Data analysis was performed using SPSS 26 with ANOVA testing. The results show that the addition of liquid smoke to flying fish with different concentration variations affects moisture content, phenol content, and organoleptic properties. The optimal concentration of liquid smoke for use as a preservative is 6%.

Keywords: Liquid smoke from coconut shell, flying fish, GC-MS.

INTRODUCTION

Indonesia, as a country with a tropical climate, possesses abundant natural resources, including coconuts (Cocos nucifera), which still have significant potential for further research and development to maximize their utilization. Although most parts of the coconut fruit have been utilized, a substantial amount is still wasted, such as the husk and shell. One way to make use of coconut shells is by converting them into materials for producing liquid smoke (Luh *et al*., 2020).

Liquid smoke is the result of condensation or the collection of vapor produced directly or indirectly from the combustion of materials containing lignin, cellulose, hemicellulose, and other carbon compounds (Hidayati & Setiono, 2020). According to (Rasi & Seda, 2017) liquid smoke produced from the pyrolysis of coconut shells contains 4.13% phenol compounds, 11.3% carbonyl compounds, and 10.2% acid compounds. This liquid smoke has the potential to be used as a preservative for foods such as fish and meat, as well as to enhance the flavor, aroma, and texture of these products. (Satria *et al*., 2021).

Mackerel scad is a small pelagic fish with high economic value and is abundant in Indonesian waters. This fish, known scientifically as 'Decapterus sp,' is named from the combination of the words 'deca,' meaning ten, and 'pteron,' meaning wing, so 'Decapterus' can be interpreted as a fish with ten wings (Kusumanigrum *et al.*, 2021). According to *(Umpain et al*., 2019) The chemical composition of mackerel scad consists of 76% water, 20.6% protein, 1.3% fat, and 1.4% ash. After the fish dies, various chemical, physical, and organoleptic changes occur rapidly, ultimately leading to spoilage. Fish is a type of food that easily spoils, and freshness is a crucial factor in maintaining the quality of this highly perishable product. Therefore, the use of smoking with liquid smoke can extend the shelf life of processed fish and maintain its freshness (Sutrisno *et al*., 2020). According to (Guarango, 2022) Several factors that affect the quality of smoked fish include the reduction of moisture content to below 40%, although the standard moisture content for smoked fish according to SNI (2013) is a maximum of 60%. Additionally, the compounds present in smoke can inhibit the growth of spoilage bacteria. According to (Litaay et al., 2022) the purpose of the smoking method is to reduce the moisture content in the fish, which helps inhibit bacterial growth and extend the shelf life of smoked fish.

MATERIALS & METHODS

The research was conducted at the Chemistry Laboratory, Faculty of Mathematics and Natural Sciences, Gorontalo State University, as well as at the Integrated Laboratory of Islamic University of Indonesia.

The equipment used in this research includes a distillation apparatus, reaction tubes, smoke delivery pipes, a condenser, a tar collector, a pyrolysis reactor cover, a liquid smoke collector, and a smoke discharge pipe, plastic containers, a desiccator, an analytical balance, dropper pipettes, stirring rods, spatulas, measuring flasks, graduated cylinders, beakers, porcelain dishes, a fume hood, stands and clamps, burettes, and an oven. Coconut shells, mackerel scad, aquadest, 0.1N $KBrO_3$, KBr, 3N HCl, starch indicator, and $Na_2S_2O_3$.

Process of Producing Liquid Smoke

Before the combustion process, the coconut shells are first cleaned of dirt and remaining fibers. After that, the coconut shells are crushed into smaller pieces. Then, drying is performed through sun-drying to reduce the moisture content in the shells. The dried shells are then burned in a pyrolyzer, which is tightly sealed and heated at temperatures ranging from 200 to 450°C. The smoke then passes through pipes and undergoes condensation, transforming into a liquid known as Grade III liquid smoke. The liquid smoke produced from pyrolysis is collected using a graduated cylinder, with the pyrolysis process lasting for 5 hours.

Purification of Liquid Smoke

The next step is the distillation process, where the Grade III liquid smoke sample is placed into a distillation flask that has been assembled. The distillation flask is then sealed and a thermometer is attached, followed by heating at a temperature between 95-100°C. The Grade II liquid smoke produced has a pale yellow color and a lighter aroma compared to Grade III liquid smoke. The distillation product is then collected and transferred into a clean bottle. The bottle is tightly sealed to prevent the evaporation of volatile compounds. The distillation of liquid smoke also aims to remove unwanted compounds such as tar and polycyclic aromatic hydrocarbons.

Identification of Liquid Smoke Components

The determination of chemical components in liquid smoke from coconut shell waste is conducted using a GC-MS spectrometer.

Sample Preparation of Mackerel Scad

The mackerel scad samples are washed and cleaned of any attached dirt, including the head and entrails, and then filleted. After that, the fish is drained for 5 minutes. The next step is to soak the fish in the liquid smoke obtained from coconut shells.

Application of Liquid Smoke to Mackerel Scad

1. Determination of Optimum Concentration of Grade II Liquid Smoke
   Cleaned mackerel scad is soaked in 250 ml of liquid smoke derived from coconut shells, with concentrations of 0, 2, 4, and 6% in plastic containers.
2. Determination of Storage Duration for Liquid Smoke
   Mackerel scad soaked in liquid smoke at concentrations of 0%, 2%, 4%, and 6% is stored for 1, 3, and 7 days. After soaking, the mackerel scad is drained and then analyzed for moisture content, phenol content, and organoleptic tests

Moisture Content Test for Smoked Mackerel Scad

$$\text{Moisture content (\%)} = \frac{b - c}{b - a} \times 100\%$$

The procedure for analyzing moisture content begins with drying a porcelain dish in an oven at 100-105°C for one hour. After drying, the dish is then cooled in a desiccator for about 30 minutes before weighing. A 1-gram sample that has been ground is placed in the dried dish, then put in the oven at 100-105°C for 3 hours. Once the drying process is complete, the dish is placed back in the desiccator and weighed with the sample, which should be free of moisture. The heating process in the oven is repeated until the weight reaches a stable value. The constant weight in moisture content analysis can be calculated using the following formula:

Description:
a = Weight of the empty dish (grams)
b = Weight of the sample + dish (before oven) (grams)
c = Weight of the sample + dish (after oven) (grams)

Phenol Content Test for Mackerel Scad

Phenol content is analyzed using the titration method. First, 2 grams of mackerel scad sample are weighed and dissolved in 100 mL of distilled water in a measuring flask. Next, 5 mL of the sample is placed into an Erlenmeyer flask, and then 5 mL of 0.1 N $KBrO_3$ solution, 0.2 g of KBr, and 3 mL of 3 N HCl solution are added. The solution is mixed and allowed to stand for 30 minutes, after which 5 mL of KI solution is added. Then, 8 drops of starch indicator are added, and the solution is titrated with 0.1 N thiosulfate solution until the blue color disappears. The percentage of phenol content can be calculated using the following formula:

$$\%\text{Phenol} \; \frac{(b-a) \times (BMf/6) \times 1000}{0{,}1 \times \text{sample weight}} \; x\ 100$$

Description:
b = Volume of Blanko
a = Volume of sample
BMf = Molecular weight of phenol
6 = Number of bromine atoms used

Organoleptic Test for Smoked Mackerel Scad

1. Taste Test
   - Evaluated by tasting the mackerel scad sample that has been soaked in liquid smoke.
   - Conducted by 15 panelists.
2. Smell Test
   - Smelled from a distance of 1⁄2 cm from the nose to assess the odor of the mackerel scad sample that has been soaked in liquid smoke.
   - Conducted by 15 panelists.
3. Texture Test
   - The texture of the mackerel scad sample soaked in liquid smoke is assessed by touch.
   - Evaluated by 15 panelists

Data Analysis

The obtained data are analyzed using statistical hypothesis testing. Statistical analysis for moisture content, phenol content, and organoleptic tests is performed by applying a completely randomized design using ANOVA testing.

RESULT AND DISCUSSION

Analysis of Liquid Smoke Components Using GC-MS

GC-MS analysis of the liquid smoke from the pyrolysis of coconut shells revealed five peaks. The GC chromatogram for the liquid smoke is shown in Table 1, while the five spectra of the compounds in the GC chromatogram of the liquid smoke from coconut shells can be seen in Figure 1.

Table 1. Characteristics of Grade II Liquid Smoke

Peak	tR (minutes)	Area %	IUPAC Name	Molecular Formula
1	1.514	5.68	Hydnocarpic acid	$C_{16}H_{28}O_2$
2	1.617	11.46	Diethyl ether	$(C_2H_5)_2O$
3	1.797	53.24	Acetic acid	CH_3COOH
4	3.402	21.15	Furfuraldehyde	$C_5H_4O_2$
5	8.973	8.48	Phenol	C_6H_6O

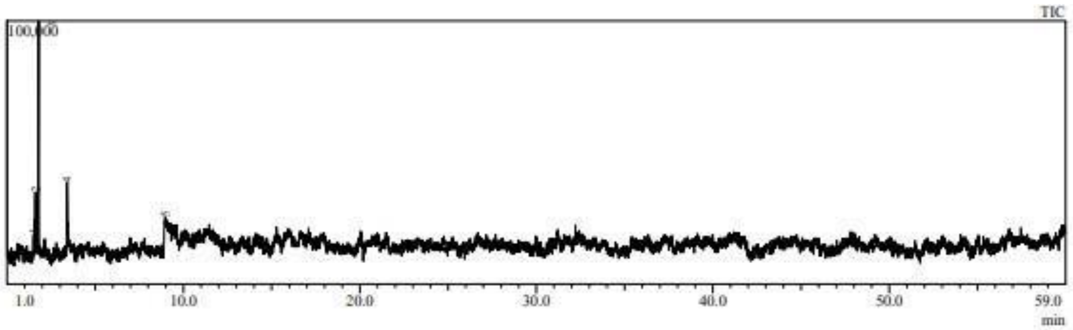
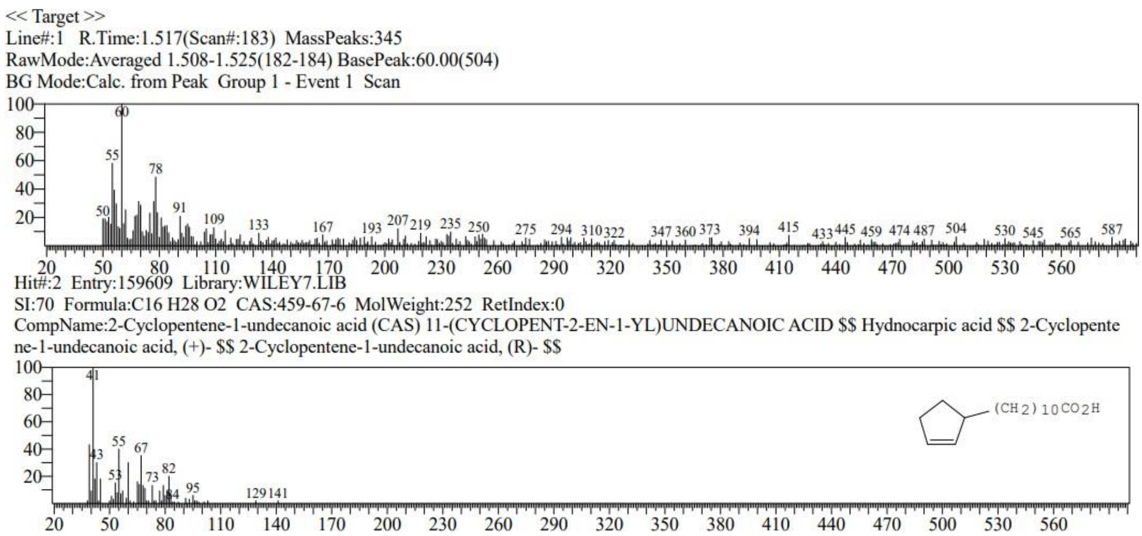
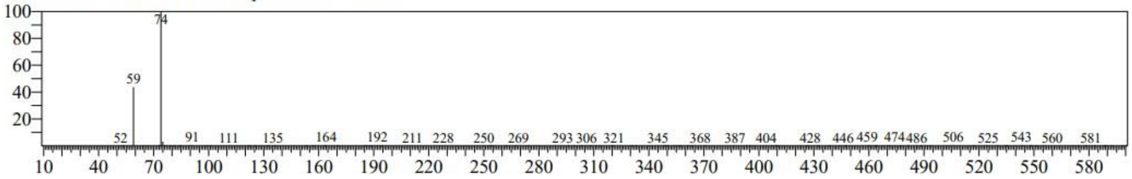


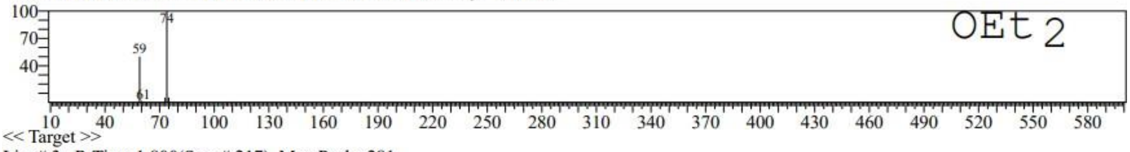
Figure 1. GC Chromatogram of Grade 2 Coconut Shell Liquid Smoke



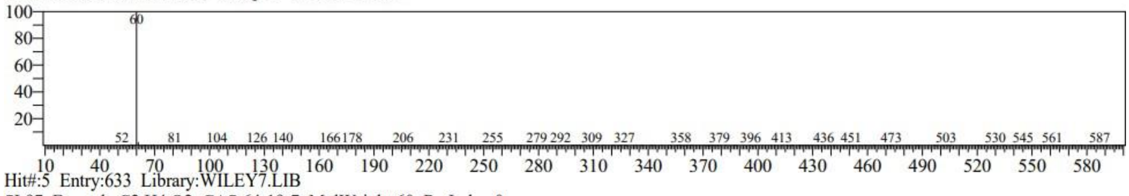
<< Target >>
Line#:2 R.Time:1.617(Scan#:195) MassPeaks:228
RawMode:Averaged 1.608-1.625(194-196) BasePeak:74.00(10399)
BG Mode:Calc. from Peak Group 1 - Event 1 Scan



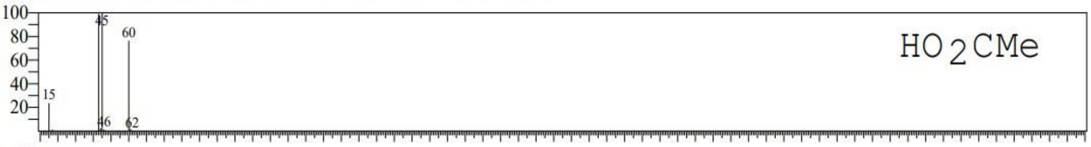
Hit#:2 Entry:1833 Library:WILEY7.LIB
SI:94 Formula:C4 H10 O CAS:60-29-7 MolWeight:74 RetIndex:0
CompName:Ethane, 1,1'-oxybis- (CAS) Ethyl ether $$ Diethyl ether $$ Ether $$ Pronarcol $$ Ethoxyethane $$ Solvent ether $$ Diethyl oxide $$ Sulfuric ether $$ Anesthesia ether $$ Anesthetic ether $$ Anaesthetic ether $$ 3-Oxapentane $$ 1,1'-Oxybisethane $$ (C2H5)2O $$ Aether $$ Diaethyl aether $$ Dwuetylowy eter $$ Etere etilico $$ Ether ethylique $$ Ether, ethyl $$ Ethyl ether, tech. $$ Ethyl oxide $$ Oxyde d'ethyle $$ Rcra waste number U117 $$ UN 1155 $$ ETHENE,-ETHYLOXY $$ Diethyl ether $$



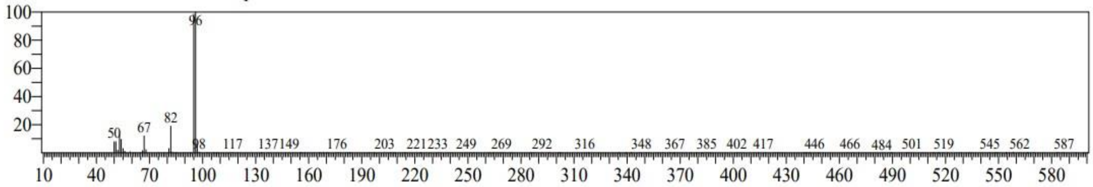
<< Target >>
Line#:3 R.Time:1.800(Scan#:217) MassPeaks:281
RawMode:Averaged 1.792-1.808(216-218) BasePeak:60.00(63309)
BG Mode:Calc. from Peak Group 1 - Event 1 Scan



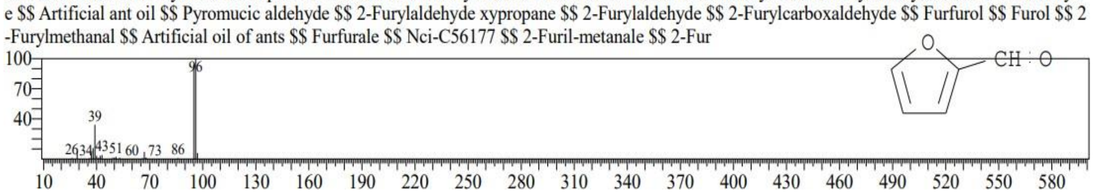
Hit#:5 Entry:633 Library:WILEY7.LIB
SI:97 Formula:C2 H4 O2 CAS:64-19-7 MolWeight:60 RetIndex:0
CompName:Acetic acid (CAS) Ethylic acid $$ Vinegar acid $$ Ethanoic acid $$ Glacial acetic acid $$ Methanecarboxylic acid $$ CH3COOH $$ component of Aci-Jel $$ Acetasol $$ Acide acetique $$ Acido acetico $$ Azijnzuur $$ Essigsaeure $$ Octowy kwas $$ Acetic acid, glacial $$ Kyselina octova $$ UN 2789 $$ UN 2790 $$ Ethanoic acid monomer $$ Aci-Jel $$

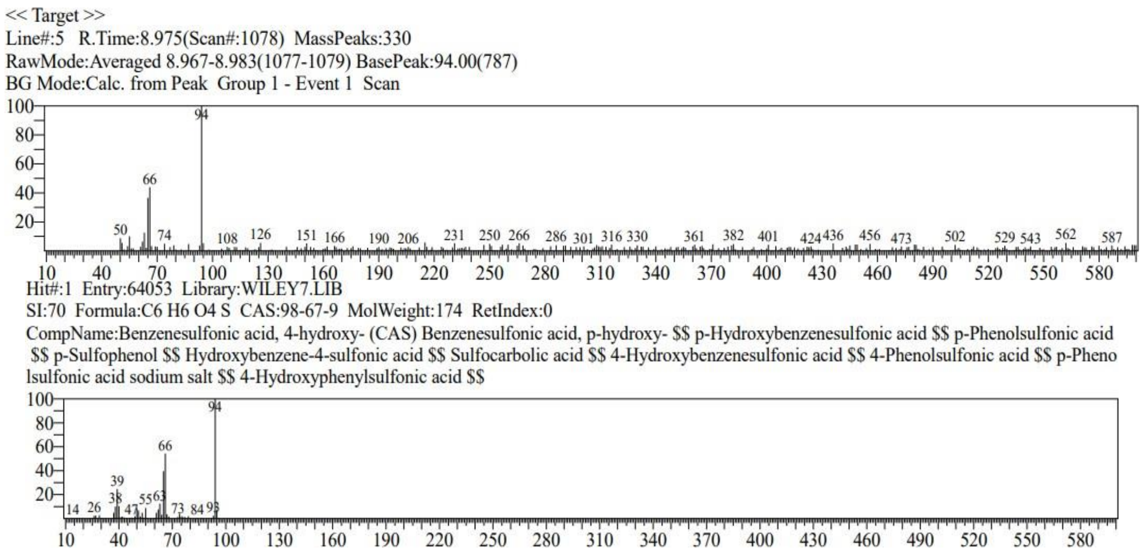


<< Target >>
Line#:4 R.Time:3.400(Scan#:409) MassPeaks:277
RawMode:Averaged 3.392-3.408(408-410) BasePeak:96.00(7990)
BG Mode:Calc. from Peak Group 1 - Event 1 Scan



Hit#:4 Entry:5252 Library:WILEY7.LIB
SI:88 Formula:C5 H4 O2 CAS:98-01-1 MolWeight:96 RetIndex:0
CompName:2-Furancarboxaldehyde (CAS) Furfural $$ 2-Furaldehyde $$ Fural $$ Furole $$ Furale $$ Furfurole $$ 2-Furfural $$ Furaldehyde $$ Furancarbonal $$ 2-Formylfuran $$ .alpha.-Furole $$ Furfuraldehyde $$ 2-Furancarbonal $$ 2-Furanaldehyde $$ Furfurylaldehyde $$ 2-Furfuraldehyde $$ Artificial ant oil $$ Pyromucic aldehyde $$ 2-Furylaldehyde xypropane $$ 2-Furylaldehyde $$ 2-Furylcarboxaldehyde $$ Furfurol $$ Furol $$ 2-Furylmethanal $$ Artificial oil of ants $$ Furfurale $$ Nci-C56177 $$ 2-Furil-metanale $$ 2-Fur





Based on Table 1, it can be concluded that the liquid smoke contains five compounds. These five compounds were analyzed based on retention time and area percentage. The compound with the largest area percentage is Acetic Acid, which has an area percentage of 53.24% and a retention time of 1.797.

Water Content Test on Smoked Flying Fish

Water content is a crucial element in food products as it affects the texture, appearance, and flavor of the food. In liquid smoke products, water content plays a critical role as it influences physical properties, chemical changes, and outcome (Makhsud, 2016).

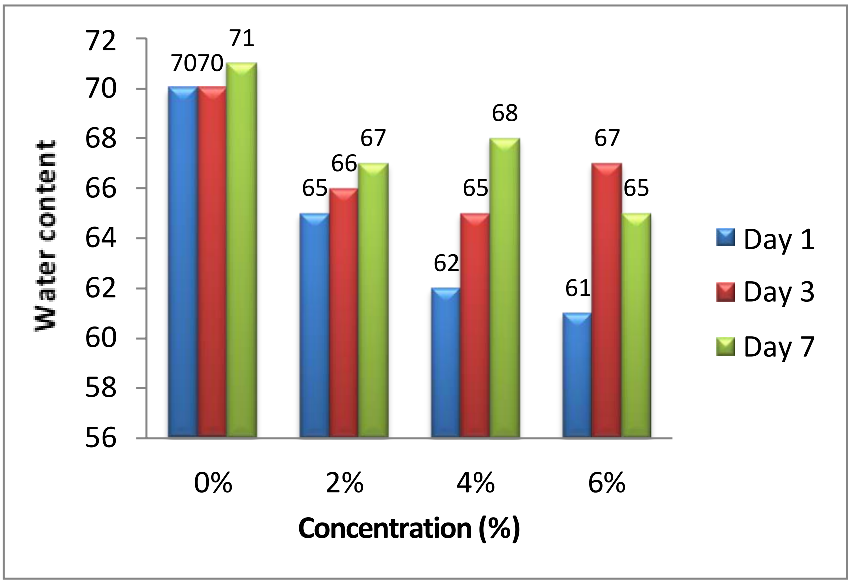


Figure 2. Water Content in Smoked Flying Fish

Based on the data obtained from the graphs, the water content in smoked flying fish increases with the duration of storage from day 1 to day 7. The average water content in flying fish increases with the concentration of liquid smoke from the control up to concentrations of 2, 4, and 6%. According to the research findings, the average water content of flying fish ranges from 61-71%. On day 3 and day 7, the smoked flying fish product is no longer suitable for consumption because its water content exceeds the limit set by SNI 2725:2013, which specifies a maximum water content of 60-65% for smoked fish.

High moisture content is caused by a short soaking time and minimal concentration variation, which results in inconsistent water evaporation during drying, leading to persistently high moisture levels. The reduction in moisture content is influenced by air temperature and the surrounding environmental humidity.

Phenol Content Test on Smoked Flying Fish

Phenol content analysis is conducted to determine the amount of phenol in the product and whether it falls within the normal range. If the phenol content exceeds normal limits, the quality of the produced product may be reduced. However, if the phenol content remains within the normal range, it can help extend the product's shelf life (Hutomo et al., 2015).

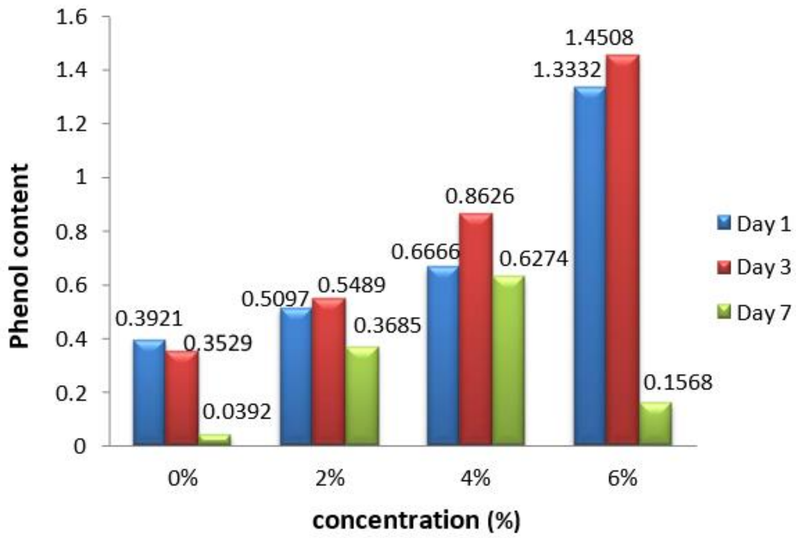


Figure 3. Phenol Content on Smoked Flying Fish

According to Girard (1992), the safe limit for phenol content in smoked products ranges from 0.06 mg/kg to 5000 mg/kg, or 6 ppm to 5000 ppm (0.0006% - 0.5%). Therefore, the phenol content in flying fish processed with liquid smoke from coconut shells, ranging from 0.0392% to 1.4508%, exceeds the established safe limits. The high phenol content on Day 3 indicates that phenol absorbed into the fish tissue may require time to redistribute throughout the fish. Consequently, the measured phenol content increases after several days.

For use as a preservative, liquid smoke concentrations of 0.2% and 4% are more effective than those above 6% due to the fact that phenol levels exceeding the safe limit cannot be used for preserving smoked fish. High phenol content can cause toxicity to humans, produce an overly strong taste and aroma, violate food safety regulations, and leave harmful residues that may endanger consumer health.

Organoleptic Test of Smoked Flying Fish

The organoleptic test is an evaluation method that uses human senses (sensory) to assess the quality or freshness of fish. This method is commonly used to determine the product's quality. In this test, panelists provide personal feedback on their preferences or dislikes regarding the product. The bar chart showing the percentage of organoleptic evaluation for flying fish with added liquid smoke during the storage period can be observed in Figure 4.

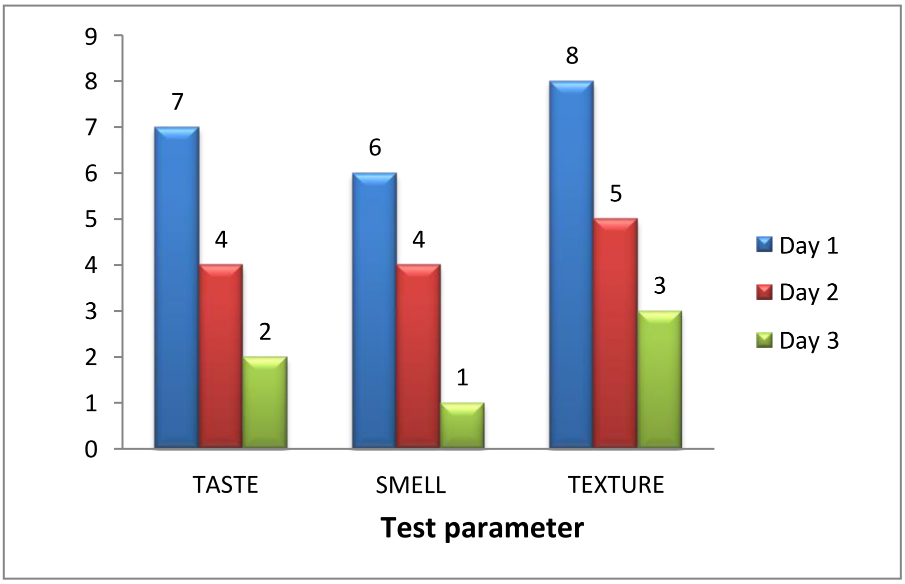


Figure 4. Organoleptic Test on Smoked Flying Fish

Values 1-9 represent the preference parameter with the following:

1 = strongly dislike	6 = somewhat like
2 = very dislike	7 = like
3 = dislike	8 = very like
4 = somewhat dislike	9 = strongly like
5 = neutral	

Taste

Taste is an organoleptic characteristic related to the sense of taste and is the result of a combination of compounds formed during the processing. As one of the organoleptic aspects, taste serves as a primary indicator for evaluating the level of acceptance of a food product. Products with taste that is not suitable or does not meet standards are usually rejected by consumers (Muchtar & Hastian, 2023)

"The average sensory values (taste) are as follows: on day 1, the interval value is 7 (like); on day 4, the interval value is 4 (somewhat dislike); and on day 7, the interval value is 2 (strongly dislike). The sensory value of the sample decreased in taste. This is due to bacterial growth on the preserved flying fish, leading to a less favorable taste for the panelists. During long-term storage, taste can change and may be rejected by consumers due to fluctuations in food components during reactions, as well as changes in physical, chemical, and organoleptic properties.

Smell

Aroma is the smell of food products perceived through the sense of smell. This aroma can attract panelists' interest in the product and help them assess their preference or dislike for it. The characteristic odor in flying fish is caused by the breakdown of proteins during the smoking process.

The storage duration of smoked flying fish affects the organoleptic value in terms of smell. Based on responses from 15 panelists, the average smell value of smoked flying fish is as follows: on day 1, the interval value is 6 (somewhat like); on day 3, the interval value drops to 4 (somewhat dislike); and on day 7, the interval value becomes 2 (strongly dislike). This decrease in smell value may be due to the release of undesirable compounds carried along with the smoke.

The organoleptic value in terms of smell indicates that panelists no longer liked the smell on days 3 and 7. Conversely, on day 1, the product still showed good results with a noticeable smoky aroma. However, the smoke in the product imparts a distinctive odor that is not found in foods made with flavor enhancers.

Texture

Texture is the sensation perceived through the mouth or touch with fingers, referring to the sensation of pressure and consistency of the material. (Mawar et al., 2023). According to (Mardiah et al., 2022), the texture of fresh fish should not be soft or slimy. According to the quality standards for fresh fish set by SNI (2729:2013), the texture of fresh fish must be firm, compact, and highly elastic. The highest average organoleptic value for texture, which is 8 (very like), was obtained on day 1 of storage, while the lowest value, which is 3 (dislike), was recorded for the product stored on day 7.

Based on the research results, panelists liked the flying fish on day 1, but on days 3 and 7, the flying fish was no longer favored by the panelists. The issue of texture deterioration indicates that as the concentration increases, the texture of the flying fish sample becomes firmer.

CONCLUSION

In conclusion, it can be summarized that liquid smoke grade 2 from coconut shell contains five types of compounds, which have been analyzed using GC-MS. The compound with the largest area is acetic acid, which accounts for 53.24% with a retention time of 1.797.

The addition of liquid smoke to flying fish at different concentrations significantly impacts several aspects of the quality of the smoked fish, including the smell, taste, and texture of the final product. Changes in each of these parameters reflect differences in the intensity and characteristics of the smoking process applied.

Declaration by Authors

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