Comparison of Raw Materials for Making Liquid Smoke with Pyrolysis Method as an Alternative to Formalin and Borax in Food

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Abstract. The manufacture of liquid smoke from the comparison of raw materials for teak and pine wood powder waste is carried out by the pyrolysis method, purified by distillation and filtration which will be applied as a preservative in food. The pyrolysis process was carried out using a reactor with a capacity of 2 kg at 450 °C for 4 h. The comparison of teak and pine wood waste used is (1:0); (0:1); and (1:1). The results of the pyrolysis vapor condensation were then purified by the distillation method at various distillation temperatures of 100, 101–125, and 126–150 °C. Mixed wood liquid smoke has the best quality at a distillation temperature of 126–150 °C with a pH of 2.9, a density of 1.029 g/mL, and an almost white beige color and can preserve food for up to 4 days with a maximum moisture content of 88.12% and TVB on day 4 was 24,512 mgN%.

Keywords: Distillation · Liquid Smoke · Pine · Pyrolysi · Teak · Total Volatile Base

1 Introduction

Pine and teak are rigid wood species that grow naturally in Indonesia, especially in Aceh. This tree wood is widely used because it has a high chemical composition of lignin, hemicellulose, and cellulose. Currently, there are many industries that have been established to produce wood that can be used as building construction materials, furniture, paper-making materials and others. The large number of wood production causes the waste generated from the wood industry to also be large. Waste from the wood production process can be in the form of lumps, pieces, and also sawdust. One technology that can be applied to the utilization of wood waste is to process it into liquid smoke.

Liquid smoke is a product of the pyrolysis process that has been condensed and has undergone a phase change from gas to liquid. The combustion process can be carried out

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Table 1. Liquid Smoke Quality Standards in Japan (Japan Liquid Smoke Association)

No	Parameter	Wood vinegar	Wood vinegar distillate
1	pH value	1.5–3.7	1.5–3.7
2	Specific gravity	>1.005	>1.001
3	Organic acid content	1–18%	1–18%
4	Color	yellow, brown	colorless, brown
5	Transparency	transparent	transparent
6	Insoluble level	there isn't any	there isn't any

(Source: Dewi et al., 2018)

directly or indirectly from raw materials containing cellulose, lignin, and hemicellulose which can be used as natural preservatives. Pyrolysis is a process of decomposition or breakdown of the chemical structure of organic matter (biomass) into a gas phase using a high heating process with limited oxygen levels [1]. The process of making liquid smoke can use raw materials such as nyamplung shell [2], oil palm shell [3], coconut shell, oil palm shell and sawdust [4], teak wood, and mahogany wood [5], coffee skin shell [6] and others. Each biomass has different characteristics and compositions depending on its type and shape [1].

The liquid smoke produced from the pyrolysis process has a brownish yellow color and a distinctive smoke aroma. The quality of liquid smoke is also determined by the purity of the compounds contained therein, especially phenols and organic and carbonyl acids. Therefore, a purification process needs to be carried out to separate the two compounds so that high quality liquid smoke is produced [7]. Distillation of liquid smoke is carried out to separate the active substances in liquid smoke in this case phenol, organic acids and also carbonyl so that liquid smoke is obtained which has high preservation properties and is protected from carcinogenic compounds such as PAHs and Tar.

Liquid smoke has several ingredients including; Phenolic compounds, these compounds act as antioxidants so that they can extend the shelf life of the product. Carbonyl compounds, these compounds in smoke have a role in the coloring and taste of the product. The carbonyl compound content of coconut shell is 11.3%. Acidic compounds, these compounds have a role as antibacterial, give the product flavor and affect the pH and shelf life of food.

Currently, the use of preservatives such as formalin and borax has been widely applied to fast food such as tofu, meatballs, noodles, and barbecue spices so that the food is more durable. Excessive use of chemical preservatives can harm human health. Therefore, liquid smoke can be used as a solution to extend the shelf life of food so that it lasts longer and does not have a negative impact on human health. Compounds that are antibacterial in liquid smoke are the presence of acid and phenol compounds. The acid compounds inhibiting bacterial growth are greater than the phenol compounds, but if the two compounds are combined, the quality of the liquid smoke will increase. Liquid Smoke Quality Standards in Japan (Japan Liquid Smoke Association) can be seen in Table 1.

2 Methodology

The research was conducted using a set of pyrolysis equipment, distillation apparatus, and glassware as supports. The materials used are teak and pine wood powder waste, and chemicals such as saturated H3PO4, 2% boric acid, Tashiro indicator, 0.02N HCl, 7% TCA, saturated K2CO3 (1:1), and vaseline.

2.1 Trial Procedure

2.1.1 Psyrolysis Process

The pyrolysis process of teak and pine wood powder was carried out in a ratio (teak:pine) of 1:0; 0:1; and 1:1. The sawdust that has been dried for 1 day is weighed as much as 2 kg and then put into the pyrolysis reactor at a temperature of 400oC for 4 h. Then the resulting smoke is condensed and liquid smoke is obtained which is still dark in color due to the presence of tar compounds. The liquid smoke was allowed to stand for 24 h to separate the tar compound. Then the liqui smoke is filtered and analyzed.

2.1.2 Distillation

The liquid smoke resulting from pyrolysis is further purified by a distillation process with temperature variations100 °C, 101–125 °C, 126–150 °C for 3 h, then the distillate was accommodated for further processing.

2.1.3 Filtration

The resulting liquid smoke distillate is filtered using activated carbon obtained from the by-product of pyrolysis and then activated with a saturated H3PO4 solution. The purpose of filtration is to obtain liquid smoke with a clear color and a less pungent odor. Then analyzed.

2.2 Analysis Stage

2.2.1 Yield

The yield can be obtained from the ratio of the mass of liquid smoke produced after the pyrolysis process to the mass of the raw material multiplied by 100%. The calculation of yield can be seen in the following formula.

$$\frac{\text{Weight of liquid smoke obtained (output)}}{\text{Weight of raw materials (input)}} \times 100\% \tag{1}$$

2.2.2 pH

In the pH test after the pyrolysis process, the pH paper was dipped into the liquid smoke sample, then allowed to stand for a while. Then read the pH results obtained. In the post-distillation test, the pH meter is immersed in a sample of liquid smoke until a constant reading is obtained, and the readings are recorded on the display.

2.2.3 Density

The pycnometer was weighed using an analytical balance. Furthermore, the pycnometer is filled with liquid smoke until there are no air bubbles. Then weighed again. Calculation of the density of liquid smoke:

$$\rho = \frac{(\text{pycnometer} + \text{liquid smoke}) - (\text{empty pycnometer})}{\text{pycnometer volume}} \quad (2)$$

2.2.4 Moisture Content

The empty cup was dried in an oven for 2 h at a temperature of 1050C until a constant weight was obtained. The crucible that had been baked was cooled in a desiccator for 30 min until it reached room temperature and weighed (A g). The sample of liquid smoke is weighed as much as 2 g in a cup (B g). The cup which has been filled with the sample is dried in an oven at a temperature of 1050C to a constant weight. The sample was transferred using a clamp into a desiccator for ± 30 min, then weighed (Cg). Calculation of water content of liquid smoke:

$$\text{Water content} = \frac{B - C}{B - A} \times 100\% \quad (3)$$

2.2.5 TVB Test (Total Volatile Base)

The procedure for testing the TVB-N value can be carried out in accordance with the procedure in SNI-01–4495-1998. Prepare 2 g of pounded sample and add 75 ml of 7% TCA. Then filtered and analyzed for TVB levels. Get a Conway cup ready. Then add 1 ml of boric acid to the inside of the cup, and the filtrate to the outside of the cup. Cover the Conway cup and add 1 ml of K2CO3 to the outside of the cup. Close the Conway cup tightly by rubbing Vaseline on the lid. The blank was done with this procedure but the filtrate was replaced with 5% TCA. Samples in the dish were incubated for 2 h at 25 °C. Then the samples and blanks were analyzed by titration using 0.02 N HCL. The following is the formula for calculating the TVB value.

$$\text{MgN\%} = \frac{(\text{Vs} - \text{Vb}) \times \text{N.HCL} \times 14,007 \times 100}{\text{Weight of sample (gr)}} \quad (4)$$

2.2.6 Organoleptic Test

Organoleptic tests were carried out on 20 panelists by utilizing the human senses to analyze the quality and safety of a product including smell, color, and appearance. Panelists will judge based on a predetermined scale and information.

3 Result and Discussion

Research on making liquid smoke from teak and pine wood sawdust by pyrolysis as a natural preservative by varying the ratio of raw materials, distillation temperature, and

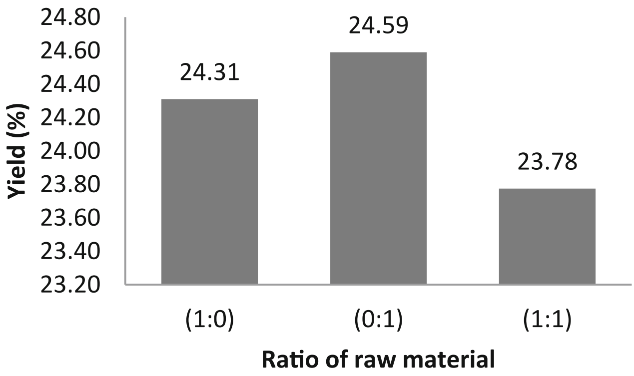


Fig. 1. Effect of Raw Material Comparison on Liquid Smoke Yield

also preservation time, it can be seen the yield value, pH value, density value, color of liquid smoke, value of water content of tofu, value of TVB (total volatile base) of tofu, and organoleptic test.

3.1 The Effect of Comparison of Types of Raw Materials on the Quality of Liquid Smoke

3.1.1 Yield

The results showed that the highest percentage yield was obtained from raw materials with a ratio (0:1) or pine wood of 24.59%. Meanwhile, the lowest percentage of liquid smoke was obtained from the ratio (1:1) or mixed wood, which was 23.78%. Overall the difference in the percentage yield of each ratio is not too different. The different yield percentages are caused by the large number of wood components such as lignin and cellulose from different raw materials [5]. In addition, the liquid smoke produced is grade 3 liquid smoke which still contains water content mixed in the smoke product because it has not gone through a further purification process so that the resulting yield is more [9] (Fig. 1).

3.1.2 pH

The results showed that the pH value of pyrolysis liquid smoke from various ratios of raw materials had met the Japanese Quality Liquid Smoke Standards ranging from 1.5 to 3.7. The resulting pH indicates that the liquid smoke product is acidic. The pyrolysis process at temperatures above 200 °C is an exothermic reaction, which is a reaction that produces heat. At this stage the decomposition process increases rapidly, the acidity of the liquid smoke product is largely influenced by the wood components decomposed during the pyrolysis process such as hemicellulose and cellulose. At a temperature of 200–250 °C hemicellulose decomposition occurs and at a temperature of 280–350 °C cellulose decomposition occurs [10] which produces organic acid compounds such as acetic acid (Fig. 2).

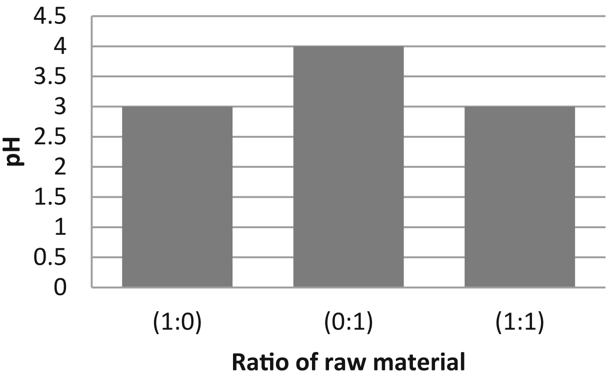


Fig. 2. Graph of the Effect of Comparison of Raw Materials on the pH of Liquid Smoke

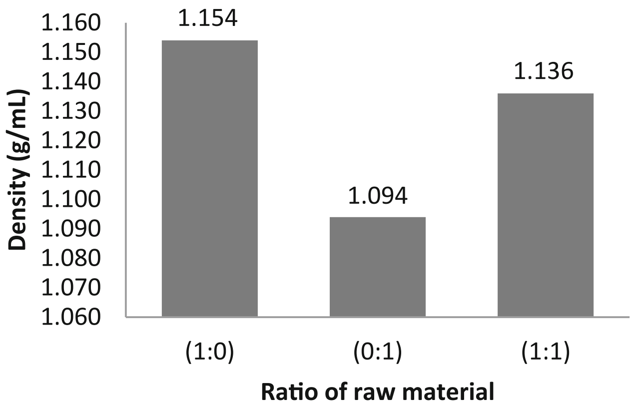


Fig. 3. Graph of the effect of the comparison of raw materials on the density of liquid smoke

3.1.3 Density

The graph above shows the density value of the pyrolysis liquid smoke product from various raw material ratios. Density shows the ratio of the weight of a sample per unit volume. The results showed that the density of liquid smoke produced after the pyrolysis process was in accordance with the Japanese Quality Liquid Smoke Standard, which was $>$1.005 [8]. The highest density was obtained from the ratio (1:1) or mixed wood of 1.136 g/mL. Then followed by density at ratio (1:0) or teak wood of 1.154 g/mL and ratio (0:1) or pine wood of 1.094 g/mL. The high density value indicates that liquid smoke contains a lot of compounds such as acids, phenols, carbonyls, and by-products in the form of tar compounds (Fig. 3).

Table 2. Pyrolysis liquid smoke color

Raw material ratio	Color
(1:0)	Reddish dark brown
(0:1)	Light brown
(1:1)	Dark dark brown

3.1.4 Color

The color of liquid smoke which is a product of the pyrolysis process from variations in the ratio of these raw materials is in accordance with the Japanese Quality Liquid Smoke Standard, which is yellow to brownish in color (Table 2).

Comparison of raw material ratios shows the effect on the color of liquid smoke. The darkest liquid smoke is produced by liquid smoke in a ratio (1:1) or mixed wood which is dark brown in color. While the lightest liquid smoke is obtained from the ratio (0:1) or light brown pine wood. In addition to carbonyl compounds that act as colorants in liquid smoke, the dark color of liquid smoke is caused by the presence of tar compounds contained in the liquid smoke. Tar is a compound that is basically black in color, is toxic, and has a high molecular weight. Therefore, to obtain liquid smoke that is free from tar content, it is necessary to carry out a further distillation process in order to produce liquid smoke with a clearer color so that it can be applied to food products.

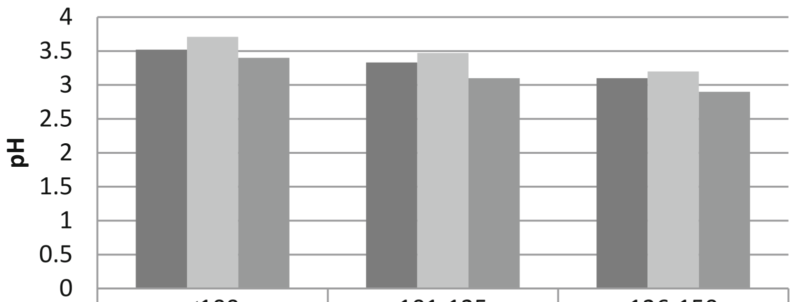
3.2 Effect of Distillation Temperature on Liquid Smoke Quality

3.2.1 pH

Based on the graph above, it can be seen that the pH value of the liquid smoke produced in the distillation process is in accordance with the Japanese Quality Liquid Smoke Standard, which ranges from 1.5 to 3.7 [8]. Overall, the pH value of the resulting liquid smoke is not much different. The lowest pH value of liquid smoke is found at a distillation temperature of 126–150 °C, namely the ratio of raw materials (1:1) or mixed wood of 2.9. Meanwhile, the highest pH was found at a distillation temperature of 100 °C, namely the ratio (0:1) or pine wood of 3.71. The higher the distillation temperature, the lower the pH value. The low pH value is due to the presence of organic acids in the liquid smoke which evaporates at its boiling point. The higher the levels of organic acid compounds in liquid smoke, the lower the pH value. High quality liquid smoke has a low pH value. The lower the pH value, the better the quality of the liquid smoke produced. This affects the organoleptic properties and shelf life of smoked products. At a low pH, bacteria or microbes as a nuisance in the preservation process will be difficult to live and develop so that food products will last a long time (Fig. 4).

3.2.2 Density

The Fig. 5 the effect of distillation temperature on the density of the resulting liquid smoke. The density of liquid smoke from distillation is lower than the density of liquid



	≤100	101-125	126-150
(1:0)	3.52	3.33	3.1
(0:1)	3.71	3.47	3.2
(1:1)	3.4	3.1	2.9

Fig. 4. Graph of the Effect of Distillation Temperature on the pH of Liquid Smoke

smoke resulting from pyrolysis because it has undergone a purification process (distillation) by separating the tar content in the toxic liquid smoke. Based on the graph, the density of liquid smoke produced is in accordance with the Japanese Quality Liquid Smoke Standard, which is >1.005 [8] except for the liquid smoke density ratio (0:1) or pine wood at a distillation temperature of 100 °C. This is because it contains a lot of water content so that the density is close to the density of water. The higher the distillation temperature, the greater the density of the liquid smoke produced. The highest density of liquid smoke was obtained from the ratio (1:1) or mixed wood at a distillation temperature of 126–150 °C of 1.029 g/mL. Meanwhile, the lowest density of liquid smoke was obtained from the ratio (0:1) or pine wood at a distillation temperature of 100 °C of 0.998 g/mL. From the data, it can be seen that the mixing of raw materials will also increase the density of liquid smoke, it can be seen that the density of pine wood will increase when mixed with teak wood.

3.2.3 Color

Increasing the distillation temperature has an effect on the color of the liquid smoke produced. The higher the distillation temperature, the lighter and nicer the color of the liquid smoke produced. The distillation process can remove impurities such as tar. Tar has high boiling point, which is above 200 °C, so with variations in the distillation temperature, tar compounds can be separated and will be left in the form of black residue. Carbonyl compounds that play a role in the formation of liquid smoke color will decrease with increasing distillation temperature. This is because carbonyl compounds have low boiling points. At a distillation temperature of 100 °C the color of the liquid smoke is slightly darker than at a temperature of 101–125 °C and a temperature of 126–150 °C. This is because the carbonyl compound has a boiling point of 100 °C so it will evaporate at a temperature of 100 °C. After the distillation process, a filtration process using activated carbon is carried out. The following liquid smoke is filtered with activated carbon. The filtering process with activated carbon has an influence on the color and aroma of liquid

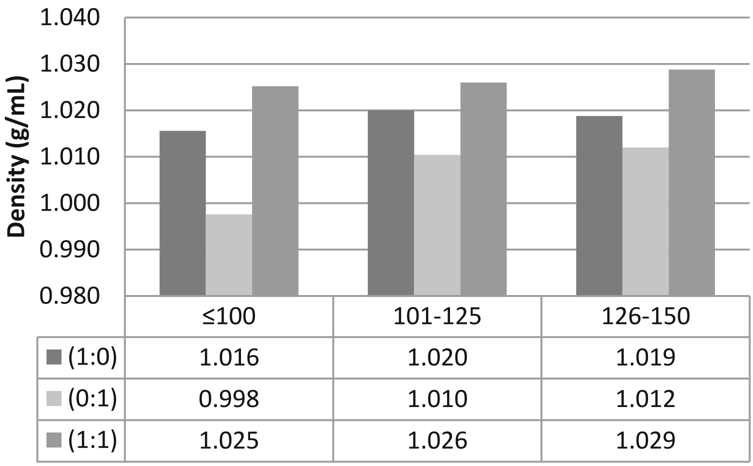


Fig. 5. Graph of Effect of Distillation Temperature on Liquid Smoke Density

Table 3. Color of Distilled Liquid Smoke

Raw material ratio	**Color**
(1:0)	Yellow gold
	dark brown
	light brown
(0:1)	golden yellow
	chocolate
	light brown
(1:1)	light cream
	light cream
	white cream

smoke, where the color of the liquid smoke produced becomes clearer and the aroma of liquid smoke is not too strong (Table 3 and Figs. 6 and 7).

3.3 Effect of Preservation Time Using Liquid Smoke on Tofu Product Quality

3.3.1 Water Content (%)

The picture above shows the water content of tofu increases with the length of storage time. The higher the distillation temperature, the lower the water content of the tofu. Based on the quality requirements of tofu [11], the maximum water content of tofu is 92%. The maximum water content that has met the tofu quality standard is on the 4th day of preservation with the highest water content of 88.12%. The highest water content was found on the 6th day of 95.1% and had exceeded the water content limit set by SNI.

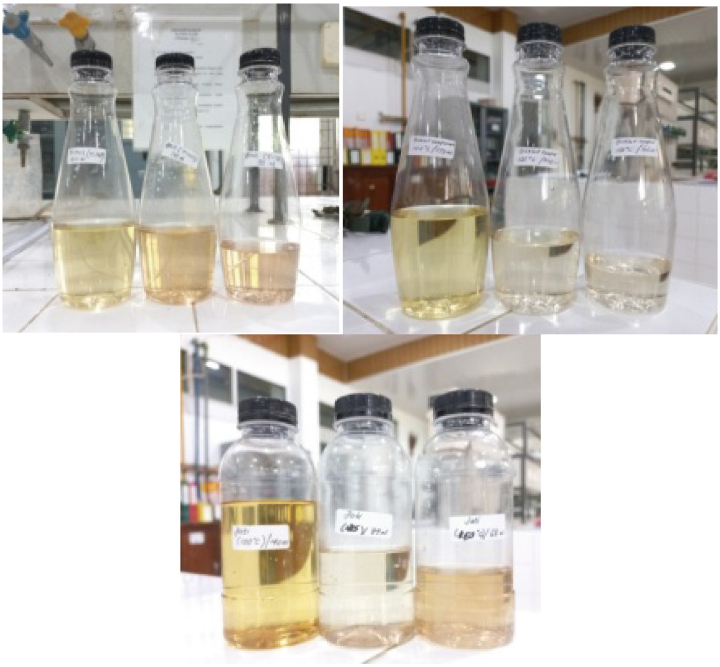


Fig. 6. Liquid Smoke Filtration with Activated Carbon

Food products with low water content and preserved as soon as liquid can prevent food products from being damaged due to microorganism activity (Fig. 8).

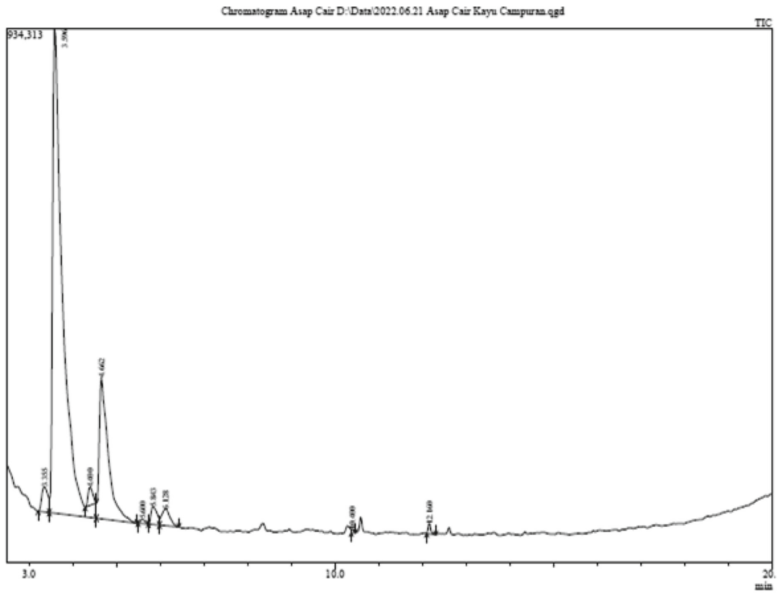
3.3.2 TVB Value (Total Volatile Base)

The value of TVB (Total Volatile Base) is used as an indicator to see the quality of food ingredients that have been preserved with liquid smoke are still in the category of suitable for consumption or not.

Testing the TVB value was carried out on tofu samples given liquid smoke mixed with distillation temperature126–1500C 2%. If the TVB value of a food product has exceeded the limit (30–35 mgN%), then the food is considered unfit to eat [3]. The TVB value on the 4th day was still included in the category of fit for consumption with a TVB value of 24,512 mgN%. However, the longer the curing time, the higher the TVB value. The TVB value on the 5th day has crossed the limit for consumption with a TVB value of 36,418 mgN%. This is because the longer preservation time causes the protein to dissolve in water so that the protein content decreases which causes the TVB value to increase (Fig. 9).

3.3.3 Organoleptic Test

Based on the data in Table 4, it can be seen that the organoleptic test data for color, odor, and appearance of tofu that had been preserved with mixed wood liquid smoke at



Peak Report TIC

Peak#	R.Time	Area	Area%	Height	Height%	A/H	Name
1	3.355	435961	2.34	44002	3.47	9.91	2-Cyclopenten-1-one, 3-methyl- (CAS) 3-M
2	3.596	13595055	73.02	847851	66.88	16.03	Phenol $$ Benzenol
3	4.400	241945	1.30	31969	2.52	7.57	dl-Erythro-1-phenyl-1,2-propanediol
4	4.662	3515923	18.88	248397	19.59	14.15	Phenol, 3-methyl-
5	5.600	55763	0.30	7762	0.61	7.18	Phenol, 2,5-dimethyl-
6	5.843	308743	1.66	31749	2.50	9.72	Phenol, 3,4-dimethyl-
7	6.128	370057	1.99	29860	2.36	12.39	2-Methoxy-4-methylphenol
8	10.400	18529	0.10	7369	0.58	2.51	Azulene, 1,2,3,5,6,7,8,8a-octahydro-1,4-dir
9	12.160	77109	0.41	18733	1.48	4.12	1(2H)-Naphthalenone, octahydro-4a,8a-dim
		18619085	100.00	1267692	100.00		

Fig. 7. Test the Content of Liquid Smoke Compounds with GCMS

a distillation temperature of 126–150 °C 2%. The organoleptic results showed that on the odor parameter, the panelists on average began to give a dislike response on the 5th day. This is due to microbial contamination of the tofu protein, causing an unpleasant or abnormal odor in the tofu.

The organoleptic results on color parameters showed that on average the panelists did not give a dislike response to the color of tofu until the 6th day. The addition of liquid smoke did not result in a significant color change in the tofu. This is because the use of liquid smoke is only used when soaking the tofu. The color of the tofu is still in accordance with SNI 3142–2018, which is normal or white.

The organoleptic results on the appearance parameters showed that on average the panelists gave a dislike response on the 5th day due to the appearance of mucus in the tofu. The slimy tofu shows the declining quality of tofu. The appearance of mucus on the surface of tofu is probably caused by contamination from the mucus-forming bacteria group [12].

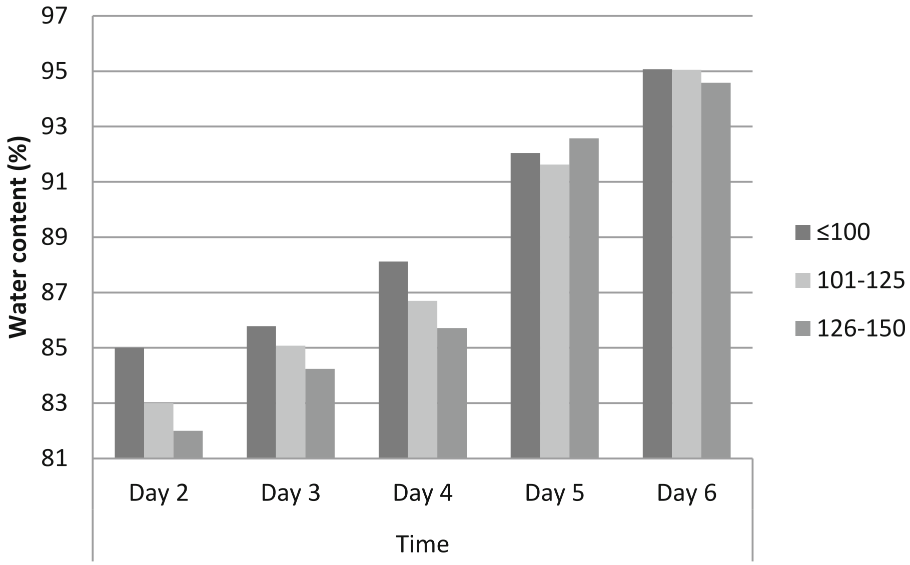


Fig. 8. Graph of Effect of Preservation Time on Tofu Water Content

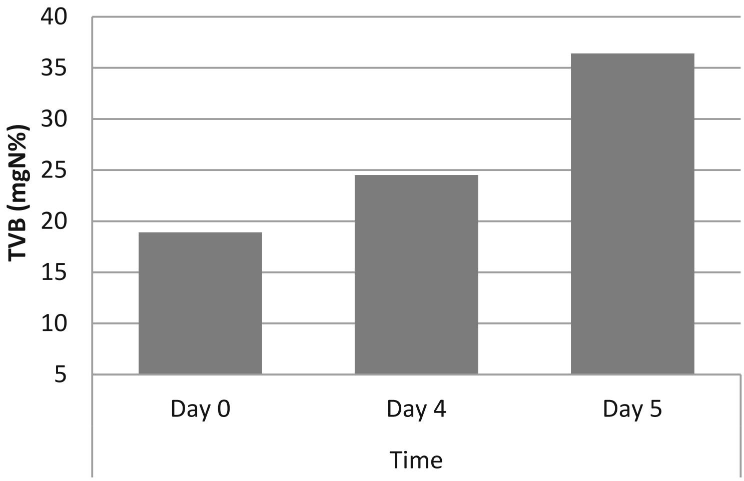


Fig. 9. Effect of Preservation Time on Tofu TVB (Total Volatile Base) Value

Table 4. Organoleptic test

Time	Sample Code	Panelist	Parameters		
			Smell	Color	Texture
Day 2	T100	1–20	2	5	5
	T125	1–20	4	5	5
	T150	1–20	4	5	5
Day 3	T100	1–20	2	5	4
	T125	1–20	4	5	5
	T150	1–20	4	5	5
Day 4	T100	1–20	2	5	3
	T125	1–20	3	5	4
	T150	1–20	4	4	4
Day 5	T100	1–20	1	3	1
	T125	1–20	1	3	1
	T150	1–20	2	3	3
Day 6	T100	1–20	1	3	1
	T125	1–20	1	3	1
	T150	1–20	1	3	1

4 Conclusions

From the results of this study it can be concluded that the effect of the comparison of types of raw materials on the quality of liquid smoke does not have a major effect on the quality of the resulting liquid smoke. The three liquid smoke produced by the ratio of types of raw materials have met the Japanese Liquid Smoke Quality Standards. Then. The higher the distillation temperature, the better the quality of the liquid smoke produced, such as pH, density and color. The liquid smoke produced is grade 1 liquid smoke. And the liquid smoke produced is able to preserve food (tofu) up to the 4th day, so it can be an alternative to natural food preservatives.

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