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Research Paper

Optimization of Liquid Smoke Products Made from Rubberwood with Pyrolysis Method

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Abstract
A large amount of rubberwood waste in Indonesia is caused by abundant rubberwood production. However, the utilization of rubberwood waste has not been optimized in Indonesia. One of the efforts to utilize rubberwood waste is to process it into liquid smoke. Liquid smoke is the dispersion of smoke vapor in water. The raw material used in this research is rubberwood. This study aimed to determine the quality of liquid smoke after purification by knowing the volume, density, pH, viscosity, yield, and phenol content using rubberwood as raw material and the pyrolysis method. The pyrolysis process is purified by distillation based on differences in boiling points. This research is divided into two stages of distillation, namely low grade purified liquid smoke and high grade purified liquid smoke. The analysis results will follow the quality standards of Japanese liquid smoke. The results show that the best liquid smoke from this research is liquid smoke with a pyrolysis time of 3 hours because it meets Japanese liquid smoke quality standards.

Keywords
Liquid Smoke, Distillation, Rubberwood, Pyrolysis

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1. INTRODUCTION

China currently occupies the position of the largest rubber producer globally because it produces 545 thousand tons of rubber annually with a land area of 475,000 hectares. Meanwhile, Indonesia is the fifth largest rubber producer in the world because it accounts for 21% of natural rubber production (Abdullah et al., 2020). Total yearly production reaches 2,175,000 metric tons and has a rubber plantation area of 3.5 million hectares. Indonesia's most significant rubber production is located on the islands of Sumatra and Java. Based on data from the Directorate General of Estate Crops Indonesia (2021), for the last five years, the most significant rubber production in Indonesia is in South Sumatra Province. In 2021, rubber production in South Sumatra will reach 870,966 tons. Many rubberwood trees are no longer productive due to large rubberwood production in South Sumatra. Usually, the rubberwood tree will be rejuvenated by cutting it down to replace it with a new, young rubber tree. The rubberwood waste that has been cut down can be reused as a different product, for example, liquid smoke.

Liquid smoke is a compound that evaporates simultaneously from the reactor through pyrolysis techniques or thermal decomposition and condenses in the cooling system (Simon et al., 2005). The condensation of the smoke will be generated through the chimney of the pyrolysis reactor, which produces liquid smoke. The liquid smoke contains several compounds formed by the pyrolysis of wood constituents, such as cellulose, hemicellulose, and lignin (Faisal and Gani, 2018). The benefit of liquid smoke for the industry is that it can provide a specific taste and aroma because it has antimicrobial and antioxidant properties and is used as a latex coagulant. In the wood industry, wood smeared with liquid smoke resists termite attacks.

The liquid smoke component contains phenol, acid, and carbonyl groups (Xin et al., 2021). These three groups act as antioxidants and antimicrobials, provide color effects, and give smoked flavors to food products (Rizal et al., 2020). Liquid smoke phenolic compounds have antibacterial and antioxidant properties and exhibit antimicrobial activity that is effective in vitro against various organisms such as bacteria, yeast, and molds (Pisoschi et al., 2018). The higher concentration of phenolic substances in the liquid smoke may have provided for the expanded inhibition against fungal growth (Oramahi et al., 2020). Usually, the liquid smoke obtained still contains high tar and dangerous compounds such as benzopyrene. A purification or distillation process is carried out on the liquid smoke to get a good quality of liquid smoke. The distillation of liquid smoke aims to remove unwanted and dangerous compounds by adjusting the boiling temperature (Irawansyah et al., 2022). This study aims that the rubberwood liquid smoke will meet the Japanese standardization requirements for liquid smoke.

2. EXPERIMENTAL SECTION

2.1 Materials

Previous research conducted by Afrah et al. (2020) used a pyrolysis reactor-type device. This tool has been modified by converting the condenser into a radiator to get more advantage. The radiator was chosen because it has a more economical price. The working principle is almost the same as the condenser, which is that the process is carried out by flowing steam into a room containing a pipe. The cooling medium used in the radiator is water. This research uses the same tool, namely the pyrolysis reactor.

The raw materials used in the previous research were wet rubberwood and dry rubberwood. Rubberwood contained 31.33%-33.49% (w/w) of lignin (Srinivasakannan and Bakar, 2004) and 8.54% yield of monomeric phenolics was obtained after pyrolysis (Mante et al., 2013). The analysis results stated that dry rubberwood had better quality than wet rubberwood. Therefore, dry rubberwood is used as raw material in the pyrolysis process. Dry rubberwood is obtained from the Ogan Ilir area of South Sumatra. The dry rubberwood obtained will be dried again to reduce the water content in the rubberwood. This drying is done in the sun for approximately one day. The source of fuel in this pyrolysis process is firewood.

2.2 Methods

Figure 1(A) shows a flow chart of liquid smoke production starting by searching for rubberwood from the Ogan Ilir area of South Sumatra. This rubberwood is used as raw material for the pyrolysis process to produce liquid smoke products, where the rubberwood is reduced in size before being put into the pyrolysis reactor. The dried rubberwood will be burned in the reactor using the pyrolysis method at a predetermined time.

The reactor was designed with a separate fuel section from the raw material in the reactor tube as shown in Figure 2. The smoke produced in the pyrolysis process flows towards the condenser and a condensation process will occur, producing liquid smoke. The liquid smoke still contains a lot of water and other compounds. A liquid smoke purification process is carried out using the distillation method to remove unwanted compounds. The distillation can also remove tar and benzopyrene compounds. The distillation will be carried out in 2 stages, namely low grade purified liquid smoke and high grade purified liquid smoke. The temperature range used for low grade purified liquid smoke was ≤100°C, while for high grade purified liquid smoke, it was ≥100°C.

Product characterization was needed to see the result and determine the obtained quality. The Trace 1300 gas chromatograph and ISQ Quadrupole mass spectrometer detector was used on characterization to get the phenol concentration. The pH parameter of the product was characterized using a pH meter, while the viscosity was tested using an Oswald viscometer. The pycnometer was used to investigate the density of the product.

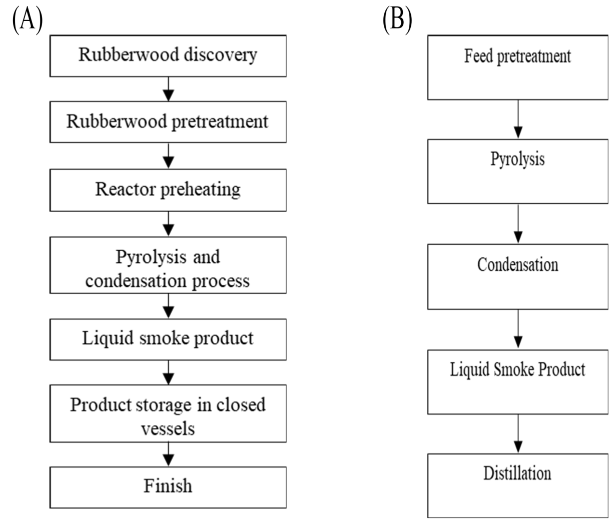

Figure 1. (A) Flowchart of Liquid Smoke Before Distillation, (B) Flowchart of Liquid Smoke

3. RESULTS AND DISCUSSION

3.1 Liquid Smoke Production

The simple liquid smoke production process generally uses a series of tools include, combustion chamber, pyrolysis reactor and condenser. The condenser in this study was replaced with a radiator. Previous research mentions that the glass condenser can be used as a cooling medium (Ali and Al Fiqri, 2020). The use of the glass condenser is not optimal because the dimensions of the reactor and the glass condenser are too small, so the material capacity of the raw materials that can enter was limited and cause the research time to run longer. In previous studies, the pyrolysis method was not carried out so that raw materials could be burned directly in the open using a condenser of copper coils in a spiral shape. The direct burning process caused a lot of harm and considered less effective.

Previous research carried out other modifications, namely using a radiator as a heat exchanger. This is proven to solve the problem in previous studies where the time required for each test was shorter time and increased the amount of liquid smoke produced (Afrah et al., 2020). Therefore, this study chose to use radiators as a cooling device that acts as a heat exchanger. Radiators have the same working principle as condensers and work based on process heat transfer between the hot and cold liquid.

Figure 3 shows a series of liquid smoke production equipment, which includes a combustion chamber, pyrolysis reactor, and radiator. According to its function, the combustion chamber was the place where the heat source combustion process occurs and can affect the results of combustion temperature. The combustion temperature in the combustion chamber must

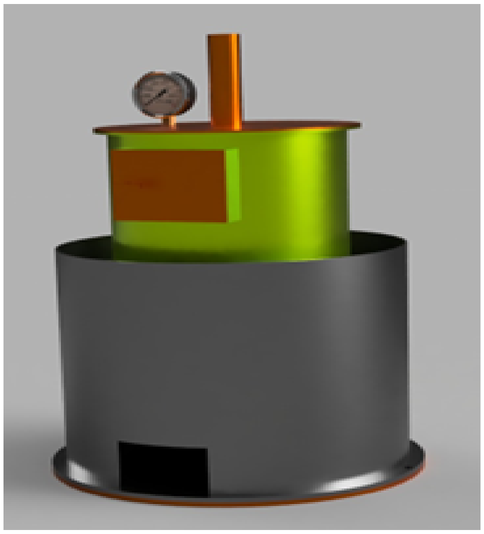

Figure 2. Pyrolysis Reactor Design

Figure 3. Series of Liquid Smoke Production Equipment Pyrolysis Method

be maintained so that the existing heat is not lost and wasted so that the combustion of the pyrolysis process can be more optimum (Nussbaumer, 2003).

The pyrolysis reactor is the primary equipment for the liquid smoke production process. The pyrolysis reactor functions as a place for the decomposition reactions of compounds to occur constituent of rubber wood into several organic compounds and will produce hot gas. Meanwhile, the radiator serves as a place of heat exchange in this as a cooler.

This research uses as much as 5 kg of dry rubber wood as raw materials with variations in pyrolysis time of 1, 2, 3, 4, and 5 hours. Components contained in rubber wood include cellulose, hemicellulose, lignin, pentose, silica, ash content, and water level. The components contained in rubber wood will be decomposed into organic compounds through the pyrolysis process and can produce liquid smoke.

The process of producing liquid smoke begins by burning the fuel in the combustion chamber. The fuel that was used in this process was rubber wood. The decomposition that occured in the combustion chamber causes the temperature in the pyrolysis reactor to increase and smoke to be produced. At the time in the reactor, there was a pyrolysis process that broke down the compounds that converted rubber wood into organic compounds at temperatures around 300-650°C (Shariff et al., 2016). The smoke released in the reactor enters the radiator and condensed to produce liquid smoke. Process the cooling that occurs in the reactor is based on the principle of heat exchange through the cooling medium in the form of air and water is sprayed on the outside of the radiator (Huang et al., 2022).

The smoke produced from the pyrolysis process is liquid smoke tar and charcoal. The liquid smoke produced still contains a lot of impurities and dangerous substances, so it must be physically filtered. Filtering is done by removing impurities using a filter cloth. This pyrolysis process was held for 5 hours according to the research variables. The liquid smoke resulting from the pyrolysis of rubber wood must be through a purification process to remove unwanted compounds. Distillation was carried out using a temperature of ≤100°C for low purified and a temperature ≥100°C for high purified liquid smoke. The distillation system was shown in Figure 4.

Figure 4. Series of Distillation Process Tools

The distillation process at a temperature 100°C for low purified liquid smoke produces liquid smoke products with the lowest quality because it has the lowest phenol content. Although it has low quality of liquid smoke, low purified liquid smoke has a higher volume since it has a higher amount of water. The water can reduce the density and quality of liquid smoke (Triastuti et al., 2019). In comparison, the distillation process at a temperature of 100°C for high purified liquid smoke produces a liquid smoke product that has a high quality compared to the other liquid smoke fraction. However, high purified liquid smoke has a lower distillate volume because it has a high content of phenols and organic acids, while the small amount of water was contained in this type of product. Phenol and organic acid function as an antimicrobial agent in liquid smoke, and their role will increase if the two compounds exist together.

3.2 Liquid Smoke Quality Analysis

The liquid smoke obtained from the pyrolysis of rubber wood must go through the following steps of quality testing. The feasibility of liquid smoke products is required for comparison as the standard of the quality of the liquid smoke. In this study, the standard used was the Japanese liquid smoke standard shown in Table 1 below this:

Table 1. Liquid Smoke Distillation Quality Standards in Japan

Parameter	Standard
pH	1.5 – 3.7
Specific Gravity (g/cm^3)	>1.001
Color	Yellow, Brown
Transparency	Transparent
Total Acid (%)	1-18

(Source: Sari et al. (2019))

Table 1 shows the characteristics needed to analyze the quality of liquid smoke from distillation that can be categorized as a liquid smoke product. The quality standard of liquid smoke used is based on Japanese quality standards. This is done because Indonesian national standards are not yet available, so that the production of liquid smoke to be commercialized must meet predetermined specifications.

After analyzing the quality of the liquid smoke product, validation between the standard and experimental molar concentrations was needed. The results were used to determine the experimental results from the phenol unpurified liquid smoke analysis, as shown in Table 2. The liquid smoke was characterized to see if the results were within the range of standard specifications, which indicated the excellent quality of the product if within the range and bad quality if it was outside the range.

Table 2 shows the results of the analysis of phenol unpurified liquid smoke and the molar concentration of phenol at the specified pyrolysis time. Table 2 shows the best results of pyrolysis at 4 hours with a yield phenol of 46.98 mg/L. These results were than detected using GC-Trace 1300 dan MS-ISQ Quaropole and produced the components contained in rubber wood liquid smoke. The analysis was carried out using the percent composition of rubber wood liquid smoke. Acids, organics, phenols, and carbonyls are compounds that are formed after the pyrolysis process is carried out. The GC-MS results for rubber wood liquid smoke can be seen in Figure 5.

Table 2. Phenol Unpurified Liquid Smoke Analysis

Time (hour)	Molar Concentration (mg/L)	Phenol density (kmol/m^3)
1	23.45	0.000249
2	40.62	0.000432
3	43.80	0.000465
4	46.98	0.000499
5	21.28	0.000226

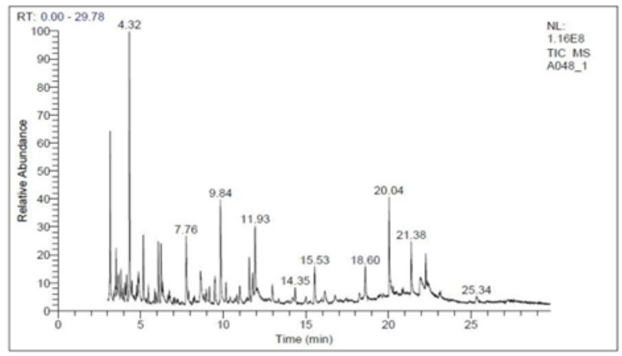

Figure 5. Chromatogram of GC-MS Rubber Wood Liquid Smoke

Table 3 shows the result of the GC-MS analysis of rubber wood liquid smoke with phenolic components and their various derivatives. Based on Table 3 above, the phenol yield in rubber wood liquid smoke shows a figure of 4.21%. This is in line with previous research conducted by Mante et al. (2013) which stated that the yield of simple phenol from the pyrolysis process was 4.52%, while the content for monomeric phenolics was 8.54%. Then, to get better results, the distillation process is carried out. The distillation process is carried out to determine if the liquid smoke meets the Japanese standard.

Distillation is carried out to separate liquid smoke from impurities and harmful substances. Distillation in this study was carried out in two stages, namely, low level pure liquid smoke and high level pure liquid smoke. The difference in distillation temperature can affect the chemical composition of the liquid smoke, so the difference can be determined by the quality of each liquid smoke produced.

Figure 6 shows that there is a difference in color from the results of low grade purified liquid smoke and high grade purified liquid smoke, where high grade purified liquid smoke tends to be light yellow, while low grade purified liquid smoke is brown in color. This is due to the presence of tar compounds in low grade purified liquid smoke products. The product distillate is used to determine the feasibility of liquid smoke products including density, pH, viscosity, yield, and phenol.

Density is one of the physical properties of testing the stability of liquid smoke which is determined by the ratio between

Table 3. The Result of Compound Rubber Wood Liquid Smoke

Components	Area (%)
3-Cyclopentane-1-acetaldehyde,2-oxo	15.67
2-Cyclopenten-1-one,2-hydroxy-3-methyl	8.82
Phenol,2,6-dimethoxy	7.95
1-Hydroxy-2-butanone	7.46
Phenol,2-Methoxy	6.55
Furo[3,4-b]furan-2,6(3H,4H)-dione,4-ethyldihydro-3-methlene-,[3aR-(3aa,4a,6aa)]	5.86
Phenol,4-methoxy-3-(methoxymethyl)	4.32
Phenol	4.21
Phenol,4-ethyl-2-methoxy	3.81
2-Propanone,1-(acetyloxy)-	3.73
5,9-Dodecadien-2-one,6,10-dimethyl-,(E,E)-	3.67
R-1-Cyano-2-methylpyrrolidine	3.58
Creosol	3.55
2-Cyclopenten-1-one,2-methyl	3.40
Butanoic acid	3.38
Cyclopentanone	3.12
Furan-2,5-diethoxytetrahydro	2.88
Cyclopropane,1-methylene-2(4,4-diethoxybutyl)-3-O-Benzyl-d-glucose	2.81
2-Furaldehyde diethyl acetal	2.73
Trimethoxyamphetaminee,2,3,5	2.52

weight and sample volume (Sari et al., 2021). The amount of density is not related to the quality of the liquid smoke but shows the number of components in the liquid smoke (Gea et al., 2020). Density also shows the density of the liquid molecular bonds in it (Jayanudin et al., 2012). Liquid smoke density analysis is presented in the form of a graph below:

Figure 7 shows that the density of liquid smoke in this study ranged from 0.96741-1.04992 g/mL. This is related to the level of acid contained in liquid smoke. The smaller the density value indicates that the liquid smoke contains less water. Figure 8 also shows that the longer the pyrolysis time, the decreased density of liquid smoke. This is because the products that are mostly formed are organic compounds such as acids, organics, phenols, and carbonyls (Handayani and Sa'diyah, 2022).

Based on Figure 7 overall, the density results obtained after distillation analysis met the Japanese quality standard, >1.001 g/mL. At the pyrolysis time of 1 hour at a high grade purified liquid smoke temperature, the liquid smoke contains a lot of water so all components are categorized into low grade purified liquid smoke.

The next analysis is the pH value. The pH value indicates the number of decomposition processes of wood components to produce organic acids in liquid smoke. The pH value corresponds to the acid being titrated. The higher the total titrated acid, the lower the pH of the liquid smoke and the higher the phenol value (Mbougueng et al., 2020).

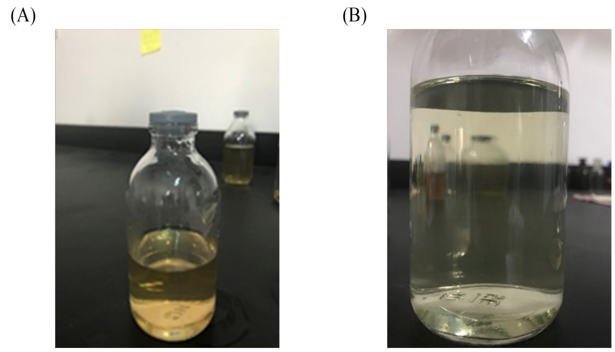

Figure 6. (A) Low Grade Purified Liquid Smoke; (B) High Grade Purified Liquid Smoke

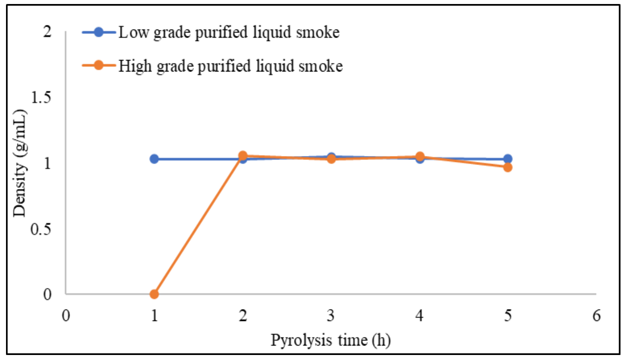

Figure 7. Liquid Smoke Density Quality Based on Pyrolysis Time

Figure 8 shows that the pH value of rubber wood liquid smoke ranged from 1.95 to 2.69. The lower the resulting pH value, the higher the quality of the liquid smoke produced. This has an influence on the durability and shelf life of the product as well as the organoleptic properties of liquid smoke. The obtained pH value on the product was occured because the rubber wood has hemicellulose, cellulose, and lignin components so that the pyrolysis process produces acid (Sakulkit et al., 2020).

Differences in pH values can be caused by differences in distillation and pyrolysis temperatures. The pH of liquid smoke at a temperature of 100-125°C is acidic this is because the phenol content in liquid smoke affects the pH value of liquid smoke. The high levels of phenol will cause the pH value of liquid smoke to be lower (Arumsari and Sa'diyah, 2021). Based on Table 2 regarding the pH of the rubber wood liquid smoke distillate, it has met the Japanese liquid smoke standard, namely, >1.5–3.7.

In addition to density and pH, another quality test for liquid smoke is viscosity. Viscosity is a measure of the viscosity of a fluid. Viscosity is an important parameter in the stability of liquid smoke storage. The small value of viscosity is due to the bonds between molecules of compounds in the fluid will be far apart (Permanasari, 2020). The viscosity of liquid

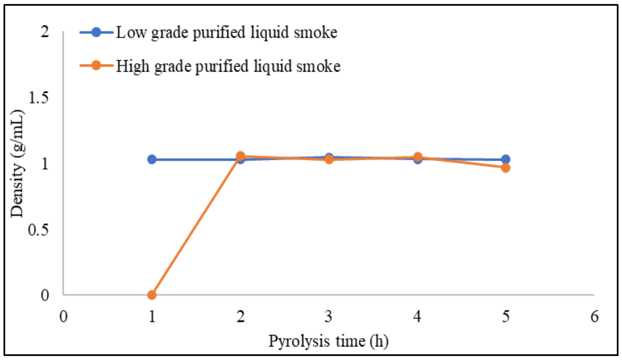

Figure 8. Liquid Smoke pH Quality Based on Pyrolysis Time

smoke varies greatly depending on the raw materials used and operating conditions during the process of formation of liquid smoke.

The viscosity of liquid smoke product was affected by temperature and time of pyrolysis process. The viscosity of liquid smoke will decrease rapidly if it occurs at room temperature and will change with the length of storage (Mohan et al., 2006). In this study, the viscosity test of liquid smoke was carried out using an Ostwald viscometer with an operating temperature of around 20–40°C.

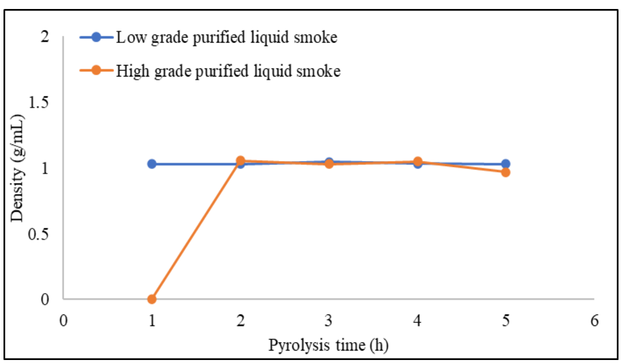

Figure 9. Liquid Smoke Viscosity Quality Based on Pyrolysis Time

Viscosity will decrease if the water content in the liquid smoke increases (Diebold, 1999). The smaller the viscosity, the lower the tar content in the liquid smoke (Adiningsih and Priatni, 2021). In Figure 9 shows liquid smoke from various time variables. Viscosity values that fluctuate between 0.9159-1.3083 Cst. Figure 9 shows that the theory corresponds to the experiments in this study, namely that increasing pH will result in a decrease in viscosity and can increase the stability of liquid smoke.

Yield is the result of the division between the amount of liquid smoke produced and the amount of raw materials. The yield results were expressed in percent (%). The factors that affect the yield of liquid smoke are operating temperature, heating rate, presence of oxygen, water content and material particle size and pressure (Gao et al., 2016). The water content affects the yield where the higher the water content, the higher the yield formed. However, this is negatively correlated with quality. The high water content reduces the concentration of the active ingredients of liquid smoke (Arnim and Marlida, 2012). Another factor is the type of raw material. Dry rubber wood produces higher yields than other types of wood.

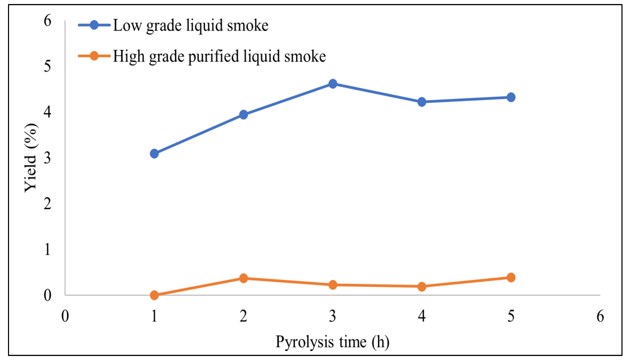

Figure 10. Liquid Smoke Yield Quality Based on Pyrolysis Time

The liquid smoke yield will increase with the longer pyrolysis time until the conditions that produce non-condensable gas increase so that the increasing pyrolysis time can reduce the liquid smoke yield (Handayani and Sa'diyah, 2022). It is the same as in Figure 10 at 3 hours the yield of liquid smoke increased until the gas condition was difficult to condense, the yield of liquid smoke decreased, namely, at the pyrolysis time of 4 and 5 hours. The size of the yield value in Figure 10 shows that the pyrolysis time and reactor temperature can affect the resulting yield.

One of the dominant liquid smoke compositions was phenol. Phenol is a compound that functions as an antioxidant and can extend the shelf life of products and give flavor to foodstuffs (Costa et al., 2021). Phenol is the result of the decomposition of lignin at high temperatures, namely 300-450°C (Lu and Gu, 2022). Kraft lignin can be volatilized with maximum decompostition during pyrolisis process with operation condition range from 200°C and 500°C (Mante et al., 2013). The more lignin contained in the wood, the more phenol produced in the product. This phenol total concentration in liquid smoke was related to the pH and total acid, so high phenol in liquid smoke will result in a lower pH and higher total acid in the product (Gracia and Ismail, 2022).

Figure 11 showed that the liquid smoke that produced the most phenol was found at a pyrolysis time of 4 hours. This is in accordance with the theory which states that the lower the pH in liquid smoke, the higher the phenol. At the time of pyrolysis 1 hour the content contained in the liquid smoke is mostly water. This is in line with previous research conducted by Maga (2018) which states that liquid smoke contains up to 92% water in addition to components dispersed in liquid smoke, phenol, carbonyl, acid, furan, and polycyclic aromatic hydrocarbons (PAH).

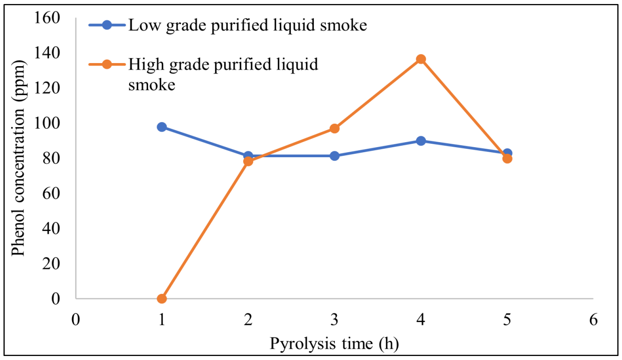

Figure 11. Liquid Smoked Phenol Quality Based on Pyrolysis Time

Therefore, the quantity of liquid smoke in low grade purified liquid smoke is higher so that no sample can be distilled on high grade purified liquid smoke samples. In this study it can be concluded that the liquid smoke product produced is in accordance with the standards of the Japanese liquid smoke association. This shows that liquid smoke is feasible to be commercialized. Based on the analysis of the characteristics of liquid smoke, the best liquid smoke product is liquid smoke with a pyrolysis time of 4 hours with a pH of 2.695, a density of 1.04992 g/mL, a viscosity of 0.9159 Cst, a yield of 0.1889%, and a total phenol of 136.32 ppm.

4. CONCLUSION

In this study, it can be concluded that the liquid smoke product produced is in accordance with the standards of the liquid smoke association in Japan. This shows that the tools used to produce liquid smoke from wet rubberwood waste and dry rubberwood can work well. The difference in distillation in low grade purified and high grade purified is clearly seen in the analysis of pH, yield, and phenol content. In low grade purified and high grade purified distillation, liquid smoke which has the lowest pH is 2.39 and 1.94 (with a pyrolysis time of 2 hours). The density analysis for low grade purified and high grade purified did not change, namely 1.02951 (with a pyrolysis time of 3 hours).

For viscosity analysis, it is according to the theory, which is that if the pH continues to increase, the viscosity will decrease. The liquid smoke yield analysis that meets the theory is liquid smoke with a pyrolysis time of 3 hours and distilled at low grade purified and liquid smoke with a pyrolysis time of 5 hours and distilled at high grade purified, namely 4.6122 and 0.3869. For phenol analysis, where the higher the pyrolysis temperature, the phenol content will increase. The data that fulfills the following theory is liquid smoke with a pyrolysis time of 4 hours. According to the results of the analysis of liquid smoke that has been distilled, it is concluded that most of the liquid smoke products from this study have met the quality standards of Japanese liquid smoke. However, suppose the liquid smoke is compared and searched for which one is the best, according to the results of the analysis. In that case, it is liquid smoke with a pyrolysis time of 3 hours because it meets the quality standard criteria of each parameter.

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