

## Enhanced Quality of Liquid Smoke Produced from Candlenut Shell by Technology Biorefinery for Food Ingredients

Sulhatun<sup>1,\*</sup>, Faisal<sup>2</sup>, Satria Nanda<sup>2</sup>, Syarifah Akmal<sup>3</sup>, and Tubagus Rayyan Fitra Sinuhaji<sup>4</sup>

<sup>1</sup>Department of Chemical Engineering, Faculty of Engineering, Universitas Malikussaleh  
Jl. Malikussaleh, Lhokseumawe, Aceh

<sup>2</sup>Department of Mechanical Engineering, Faculty of Engineering, Universitas Malikussaleh  
Jl. Malikussaleh, Lhokseumawe, Aceh

<sup>3</sup>Department of Chemical Engineering, Faculty of Engineering, Diponegoro University  
Jl. Prof. Soedarto SH., Semarang, Central Java

<sup>\*</sup> Corresponding author: [sulhatun@unimal.ac.id](mailto:sulhatun@unimal.ac.id)

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### Abstract

*This study investigates the production of liquid smoke from candlenut shells using a pyrolysis reactor with a capacity of 300 kg, integrated with a gas-contact condenser. Pyrolysis was conducted for 1–6 hours at temperatures ranging from 350 to 400 °C to generate liquid smoke. The research primarily focuses on product purification through distillation to improve quality in terms of color, odor, and chemical composition, while eliminating carcinogenic compounds such as tar residues. Variations in distillation temperature and time were evaluated to determine their effect on liquid smoke quality. The optimal pyrolysis conditions were achieved at temperatures between 350 and 450 °C, with a processing time of 3.5 hours, resulting in a total phenol area content of 56.76%. Distillation at 200 °C yielded the highest distillate yield of 73.42%, corresponding to a phenol content of 73.42% and an overall yield of 38.74% relative to the total pyrolysis products. Distillation of liquid smoke increased phenol levels by up to 17.12%. At a lower distillation temperature of 175 °C, the product yield reached 36.49% of the total pyrolysis output. These results demonstrate that distillation conditions have a significant influence on the quality and yield of liquid smoke.*

**Keywords:** Biorefinery; Candlenut; Food Ingredient; Liquid smoke; Pyrolysis.

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### INTRODUCTION

Indonesia has significant natural resource potential. A variety of crops can be found in this country. Candlenut, coconut, and oil palm are among the commodities that are experiencing rapid growth in Indonesia. Regional potential is a map of an area that can be developed and can benefit the community by supporting the national economy. The leading commodities found in abundance in Aceh are agriculture and plantations, consisting of oil palm, coffee, rubber, coconut, cocoa, sugar cane, areca nut, candlenut, nutmeg, patchouli, cinnamon, cloves, lemongrass, pepper, sago, tobacco, and sugar palm. Agricultural and plantation production data are presented in the graph below (Figure 1) (Agussabti et al., 2022; Dewi et al., 2022).

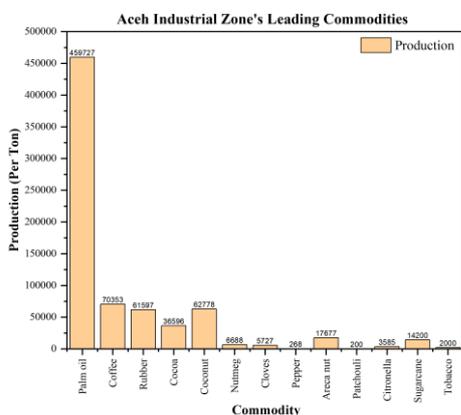


Figure 1. Aceh Industrial Zone's leading commodities

Candlenut is one of the potential local natural resources in Aceh owned by almost all districts in Aceh province (Syimir Fizal et al., 2022). In 2012, Aceh was once the largest candlenut-producing region in Indonesia, with a candlenut plantation area accounting for 35.63% of Aceh's total area, approximately 5,573,200 hectares. Aceh Province was once the largest candlenut-producing province in Indonesia, with the candlenut area accounting for 35.631% of Aceh's total area, spanning 55,372 Hectares.

The area of candlenut plantations in all districts of Aceh Province is shown in Table 1 below.

Table 1. Plantation crop area data (hectares)

Plantation Crops	Planted Area (Hectares)					
	2012	2013	2014	2015	2016	2017
Betel Nut	41065	38053	38220	38721	39244	39844
Candlenut	20271	19858	18523	18908	16874	16840
Nutmeg	21031	21104	21580	22043	23438	23994

Source: (Badan Pusat Statistik, 2018)

The most significant potential of candlenut lies in the fruit, which consists of a shell and a seed. The seed contains the kernel and skin. This kernel can be processed into oil for food. The seed processing process produces waste, namely the candlenut shell,

which has not been optimally utilized (Shaah et al., 2021).

Candlenut shells are a solid biomass waste characterized by their high content of hemicellulose, cellulose, and lignin, which can be used as a raw material for the production of liquid smoke (wood finer) (Balat, 2011). Liquid smoke produced by pyrolysis through direct or indirect combustion yields a range of compounds, including phenols, carbonyls, acids, furans, alcohols, and various polyaromatic hydrocarbon compounds (Guillén et al., 2001). Furthermore, the distillation process produces purer liquid smoke, free from toxic compounds, which can be used in the food and pharmaceutical industries (Šimko, 2005).

Sulhatun (2018, 2019) stated that liquid smoke produced from the biomass pyrolysis process undergoes thermal degradation of the raw material at a temperature of 350-450 °C, reaching 75-80% (Sulhatun et al., 2018, 2019). Biomass undergoes three main stages of the decomposition process: the initial stage, also known as hydrolysis; the primary decomposition stage; and the repolymerization stage, which occur in series and parallel, and are influenced by several process parameters (Varma et al., 2018). Liquid smoke offers various benefits, both as a raw material and as a support for the chemical industry, including raw materials for medicines, antiseptics, natural preservatives, and flavors (Shaah et al., 2021). The raw material used to produce liquid smoke is candlenut shells, which are abundant in Indonesia, particularly in Aceh province. Therefore, in welcoming the industrialization era, a government program is crucial for developing technological process expertise, creating new jobs, and stimulating the growth of other chemical industries that use locally sourced raw materials. Candlenut Shell Liquid Smoke is a product produced from the pyrolysis of candlenut shell biomass, which has the potential to be used as a raw material for the food industry due to its phenol-rich organic compounds (Faisal, 2018). This product, made from candlenut shell biomass, combines modern technology to produce a natural preservative, flavor, and raw material for the pharmaceutical industry. The potential for implementing candlenut-based biorefinery technology in various innovative products to encourage the growth of regional industries, thereby supporting the national economy (Figure 2).

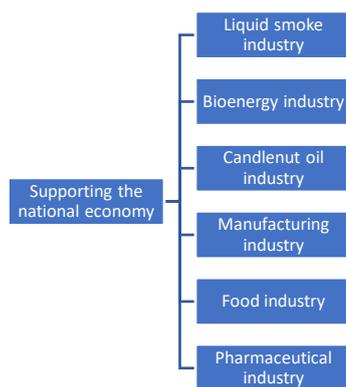


Figure 2. Supporting the national economy in Aceh's region

The application of biorefinery technology is the ideal technology for developing food products, as it utilizes natural sources of ingredients and a production process that supports the concept of a green economy. Biorefinery technology, which can be developed for the production of food additives as natural preservatives and flavors based on natural ingredients, produces natural flavors that do not contain synthetic chemicals (Tsegaye et al., 2021). Byproducts produce 75% Biochar, which can be used as an alternative energy source. The aspects of the innovation tool developed are a Biomass Pyrolysis Tool with a double condenser system, a liquid smoke purification distillation tool, and a combustion furnace with an innovative and efficient fuel energy source (Rex et al., 2023).

To overcome the increase in waste production due to limited final disposal sites (TPA), efforts are being made to develop science and technology to process several candlenut by-products into something useful (Shaah et al., 2021).

Candlenut shells have the same components as other biomass: cellulose, hemicellulose, and lignin. The composition of the shell and wood is nearly identical. Therefore, in this study, the pyrolysis process used candlenut shells as raw material, and their purification was carried out using simple pyrolysis and distillation to produce liquid smoke. The distillation process conditions (temperature and time) influenced the increase in liquid smoke production, along with increasing the capacity of the equipment used (Demirbas, 2009).

The increasing use of food additives, such as preservatives and flavorings, necessitates innovative thinking and ideas to explore and develop natural food sources using environmentally friendly technology and sustainable production processes (Zang et al., 2023).

The process involves the pyrolysis, which is the decomposition, oxidation, polymerization, and condensation reactions of these compounds. In the pyrolysis process, liquid smoke, tar, and non-condensable gases are produced. Liquid smoke,

resulting from the decomposition of organic compounds contained in candlenut shells during the pyrolysis process, is a byproduct of the activated charcoal industry (Anggraini et al., 2024; S. Zhang et al., 2020). It has high economic value when used in various chemical industries, including the rubber, agriculture, livestock, plantation, and food industries. Smoke is defined as a suspension of solid and liquid particles in a gas medium (Rizal et al., 2020). Liquid smoke is a mixture of wood smoke dispersion solutions from water made by condensing liquid smoke from pyrolysis. The results of liquid smoke pyrolysis depend on the raw materials and pyrolysis temperature. Pyrolysis produces coconut shell liquid smoke with a content of 4.13% phenolic compounds, 11.3% carbonyl, and 10.2% acid (Budaraga et al., 2016).

Pyrolysis technology is a thermochemical method for converting biomass materials into energy and various other chemical products, including liquid organic oil, organic dust, and pyrolysis gas. This process depends on the level of heat given and the duration of heating (Varma et al., 2018). Biomass pyrolysis reactions can be classified into three categories: Gradual heating, rapid heating, and very rapid heating, based on the results of the biomass pyrolysis process, including the production of liquid oil and biomass residue (Khuenkaeo & Tippayawong, 2020).

Meanwhile, biomass pyrolysis is produced through thermal degradation of raw materials at high temperatures without oxygen, producing liquid products of around 30% to 70% by weight of a large number of compounds, mainly in the form of oxides that can be easily separated into two fractions. The fraction is insoluble in water and is a concentrated tar with a specific gravity greater than water. The organic fraction is called bio-oil (Balat, 2011).

The pyrolysis parameters considered include the selection of raw material type (biomass type, particle size, initial biomass handling), reaction conditions (pyrolysis temperature, pressure, particle heating rate, and contact time), reactor configuration used, the process carried out, and various other variables such as the addition of catalysts and steam cooling mechanisms (steam condensation) (Garcia-Perez et al., 2008; Y. Zhang et al., 2018).

Several studies on the production of liquid smoke or bio-oil using the pyrolysis method have been carried out, for example Wei-Hinsen Chen et al (2016) surveyed the characteristics of the results of pyrolysis of coconut fiber and pellets in an environment containing N<sub>2</sub> and CO<sub>2</sub> to determine the impact pattern of biomass and gas carriers of the upper product at temperatures of 400°C, 450°C and 500°C. The use of raw materials such as palm seeds, coconut shells, and plum seeds, as well as grass, sawdust, straw, wheat, and pyrolysis waste. Pyrolysis produces oil, charcoal, and gas.

The primary objective of this research is to investigate the impact of distillation time and temperature on the quality of distillation products, and to compare the

products before and after distillation in terms of increasing candlenut shell liquid smoke products, which are utilized as food products through the application and development of Biorefinery technology. Biorefinery innovation technology is employed to process Candlenut Shell Biomass into liquid smoke products as food additives, utilizing local natural ingredients.

## MATERIAL AND METHOD

This research was conducted in stages, including the preparation of the feedstock, biomass pyrolysis, and distillation using a simple distillation equipment design to determine the optimal conditions for the distillation process and produce liquid smoke, as well as the product analysis stage.

The design of Pyrolysis/distillation equipment is made with stainless Steel. A suite of tools used for a simple distillation process is made as follows (Figures 3 and 4):

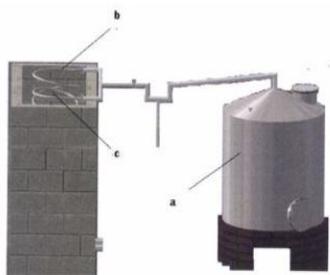


Figure 3. Design and construction of technological innovation: pyrolysis equipment with a double condenser unit system



Figure 4. Design and construction of innovative distillation equipment technology to improve the quality of liquid smoke

### Preparation of raw materials

Candlenut shells, a raw material, were obtained in an amount of approximately 1000 kg. Before starting the drying process, the material was pyrolyzed, and the tube was directly inserted without destruction, as the size of the shells was adequate.

### Phase of Pyrolysis Process

The tool uses a batch reactor equipped with a spiral-shaped double condenser cooler. The cooling water temperature is maintained at around 28-35 ° C. by regulating the flow rate of incoming and outgoing

condensation water. The pyrolysis process temperature ranges from 300 to 450°C, and the production time is 6 hours. Next, at this stage, the dried candlenut shells are weighed, with three burnings of 300 kg each, and inserted into the reactor from below so that the fire catches the flammable pyrolysis sample. The pyrolysis tool is run for 6 hours to produce a grade 3 liquid smoke product, which is then condensed into a grade 3 liquid smoke and collected in the tank.

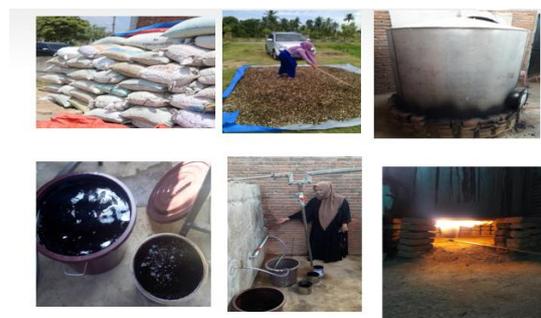


Figure 5. Liquid smoke production process using the pyrolysis method

### Distillation phase

Liquid smoke, ranging from 0 to 35 liters, is included in the distillation reactor and then heated to a variable temperature. In this research, the fixed variables used a 35-liter capacity of raw materials for liquid smoke. While the independent variable, time distillation, varied (15, 30, 45, 60, 75, 80, 95, and 110 minutes), and the temperature of distillation was maintained at 150 °C, 175 °C, and 200 °C. At the same time, bound variables include the degree of acidity (pH), analysis of the phenol compound, and the percentage of liquid smoke generated.

Distillation is a separation process based on the different boiling points of the components to be separated, solution concentration, and purification of the liquid element. In this distillation process, two liquids are produced: the distillate products and the byproducts, called residue (Duanmu et al., 2024; Faisal, 2018). Phenol, acid and carbonyl compound is present in liquid smoke that each has a different boiling points *i.e.* discount Phenol has a boiling point of 162-285 °C, acid has a boiling point of 118-176 °C, and carbonyl having a boiling point 21-97 °C, Distillation conducted on liquid smoke of pecan shell as much as 35 liters using temperature variations of 150°C, 175°C, and 200 °C. (Bahadur et al., 1995) Distillation is aimed at improving the quality of liquid smoke in terms of color, smell, and composition of the product, primarily to increase phenol levels and reduce the loss of Polycyclic Aromatic Hydrocarbon (PAH) compounds, such as benzopyrene, which are toxic and carcinogenic.

### Analysis phase

The product testing stage is carried out by conducting GCMS analysis to see the composition of the product

produced before and after distillation and conducting product analysis on the physicochemical properties of the product including the degree of acidity (pH), analysis of phenol compounds, % liquid smoke content produced from the distillation process which will be applied as a Food Additive and the composition of the distillation product (Guillén et al., 2000).

Analysis phase conducted by measuring the degree of acidity, density measurements, and phenol levels found in liquid smoke using a GC-MS.

## RESULTS AND DISCUSSION

In this research, pyrolysis was performed on samples in batches using a pyrolysis reactor equipped with a cooling spiral.

### Pyrolysis Result



Figure 6. The product of biorefinery technology is a combination of pyrolysis and distillation processes, which produce food additive products.

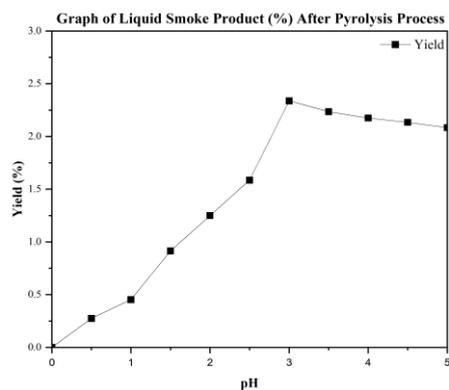


Figure 7. Percentage graph of liquid smoke product from the pyrolysis of candlenut shell

Based on Figure 7, it can be seen that the pyrolysis process at a temperature of 350-450°C (10-hour time process) results in a pyrolysis percentage of 2.33% of the total number of products, with pyrolysis occurring at the highest pH of 3. This indicates that the product Liquid smoke produced still has a high acid content (Budaraga et al., 2016).

### The influence of time and temperature on the percentage of products of pyrolysis liquid smoke

The results obtained demonstrate a relationship between pyrolysis time and the percentage of liquid smoke product resulting from the show time pyrolysis effect on the rate of products produced by pyrolysis. As the pyrolysis time increases, the acquisition of liquid smoke increases (Mohd Hasan et al., 2019). The graph shows a drastic increase in the acquisition of liquid smoke from 2.5 to 3 hours, followed by a decrease during the interval from 5 hours to 5.5 hours. This is due to the decomposition of biomass being more perfect because the distribution of heat in the reactor is more even. This causes the contact heat and material to increase the rate of decomposition of biomass as much as possible, producing liquid smoke (Mohd Hasan et al., 2019; Zheng et al., 2012).

In the process of pyrolysis Biomass of candlenut shell occurred several stages of the process decomposition that is at the early stages of the process of evaporation of water followed by decomposition of biomass material such as Hemicellulose, lignin and cellulose that produce gas - light gases CO and CO<sub>2</sub> and water discharge CO<sub>2</sub> decomposition highest range of time 0.5 hours to 1.5 hours on a pyrolysis process at a temperature of 350-400°C. Subsequently, the decomposition of hemicellulose, cellulose, and lignin occurs at temperatures ranging from 250°C to 250°C. The products being formed at the moment are tar (Bennadji et al., 2013; Wang et al., 2019). The decomposition of biomass progresses with increasing pyrolysis temperature and time, resulting in a residue that is primarily charcoal. Pyrolysis at temperatures of 400°C produces compounds of high organoleptic quality, and at higher temperatures, the process repeats. The pyrolysis temperature affects the distribution and properties of the product. In general, the results of the acquisition of Bio Oil show that the concentration achieved an operating temperature of up to 550°C, with conditions that decrease the temperature. At temperatures higher than 600°C, bio-oil and char will decompose into gas due to the numerous reactions that occur during secondary formation. Temperature pyrolysis has a significant influence on the distribution of the product. Generally, the bio-oil yield of each concentration at a temperature of between 400 °C and 550 °C, and then decreased after the heating process. At temperatures higher than 600°C, bio-oil and char products in the form of gas are converted to predominantly secondary termination reactions (Ali et al., 2016; Patra et al., 2017). Fraction of polar compounds, Aliphatic and aromatic in the bio-oil is formed in line with the rise in temperature of 300°C - 500°C to a temperature of 600-800 °C, the temperature usually reaches 700 °C to increase the formation of bio-oil carbon in the compound polycyclic aromatic hydrocarbons (PAHs), pyrene and phenanthrene, also decarboxylation and dehydration reaction formation, acquisition composition variations gases (CO, CO<sub>2</sub>, CH and so on) (Keiluweit et al., 2012; Matamba et al., 2020).

### Analysis of liquid smoke results from pyrolysis

The chemical composition of the liquid smoke shell resulting from pecan pyrolysis. Analysis was by GC-MS before distillation, as shown in Figure 8 below:

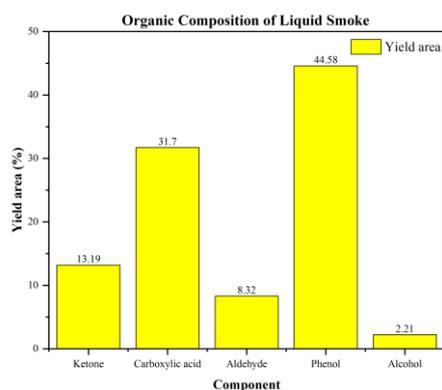


Figure 8. The GC-MS analysis of liquid smoke after the pyrolysis process

Based on the GC-MS analysis of liquid smoke obtained from the pyrolysis of pecan shells, 11 main components were identified. These include acetic acid (4.79%), cyclopentanone (5.13%), specific dioxolan derivative (4-methyl-1,3-dioxolan-2-one) (3.42%), 2-methyl-2-cyclopenten-1-one (4.81%), furfural (2-furancarboxaldehyde) (11.91%), [dimethyl amino derivative] (3.70%), valeranone (9.51%), and phenolic derivatives. The dominant phenolic compounds are 2-methoxyphenol (guaiacol) and 2-methoxy-4-methylphenol (creosol), with concentrations of 42.87% and 13.89%, respectively. Consequently, the total content of these phenolic compounds is 56.76%. The pyrolysis liquid smoke analysis results indicate the presence of polyaromatic hydrocarbons. Hydrocarbons are compounds. Polyaromatic components are carcinogenic, making liquid smoke particularly hazardous. Therefore, it is necessary to separate them through the distillation process to eliminate carcinogenic components (Ding et al., 2007; Guillén et al., 2000; Oramahi et al., 2024).

### Distillation Result

To improve the quality of liquid smoke that has economic value and can be used as a natural preservative, the distillation process of pyrolysis liquid smoke is necessary. Distillation aims to eliminate Polycyclic Aromatic Hydrocarbons (PAH) as carcinogenic benzopyrene (Guillén et al., 2000; Jira, 2004; Singh et al., 2023).

### Distillation influence of time and temperature on the percentage of liquid smoke products

From Figure 9 on the graph, it can be seen that distillation is carried out at a temperature of 150°C, resulting in a distillate with a percentage of only around 24.77%, which is the lowest percentage of product. At a temperature of 175°C, the yield

reached 36.49%. At a temperature of 200°C, the number of products continues to increase to 38.74%, which corresponds to the highest distillation yield. The big difference in the results of the distillate obtained at 200°C is due to the composition of chemical compounds contained in the condensed liquid smoke distillate, which is already perfect (Niu et al., 2016; Zhao et al., 2019).

The product density and pH values generated at each temperature are presented in Table 2. Based on the analysis of liquid smoke GC-MS distillation at various temperatures in Figure 9, the results obtained from the chemical composition of liquid smoke can be seen in Table 2 below:

Table 2. Analysis of the degree of acidity (pH) and analysis of the smoke density of the liquid after distillation

Temperature (°C)	Density (kg/m <sup>3</sup> )	pH
150	0.980813	4.54825
175	0.977181	3.56234
200	0.977912	4.2256

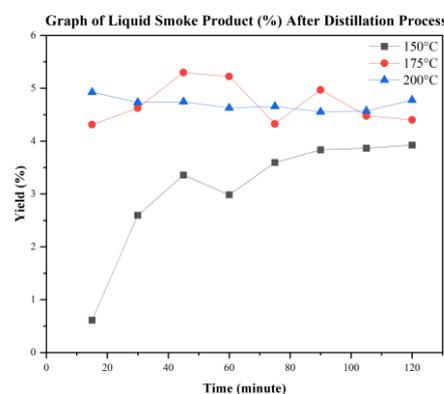


Figure 9. Percentage graphics products liquid smoke against time and temperature distillation

### Liquid smoke GC-MS analysis results after distillation at different temperature variations

The chemical composition of liquid smoke from distillation at temperatures of 150 °C, 175 °C, and 200 °C is shown in Figures 10 and 11.

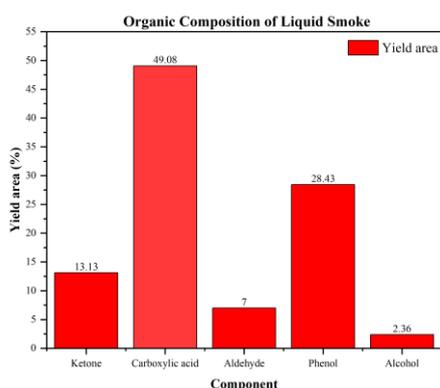


Figure 10. The GC-MS analysis of liquid smoke after the first distillation

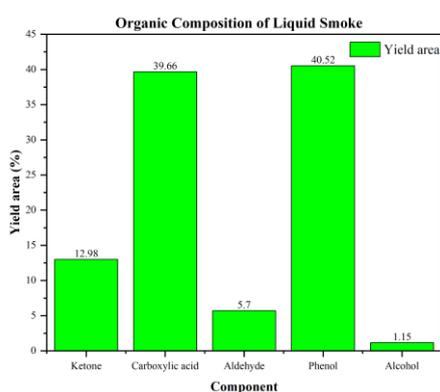


Figure 11. The GC-MS analysis of liquid smoke after the second distillation

The analysis results in Figures 9 and 10 show that the lowest temperature distillation at 150 °C yields 10 components, comprising three ketones and aldehydes, two phenolic compounds, and one 5-methyl-4-methylene compound. In the liquid smoke results, pyrolysis achieved a total phenol content area of up to 53.52% and continued to increase at a temperature of 175 °C, reaching 73.42%, before declining at a temperature of 200 °C to 64.99% (Alizadeh et al., 2013; Knowles et al., 1975).

Variations in distillation temperature significantly alter the chemical composition and phenolic content of liquid smoke. The highest phenolic content was observed at a distillation temperature of 175°C. At 150°C, ten chemical components were identified: cyanobutadienitrile (2.67%), cyclohexanone (3.34%), [cyclopentene derivative] (8.09%), cyclopentenone (8.91%), furfural (14.27%), 2-acetylfuran (3.09%), guaiacol (2-methoxyphenol) (41.04%), and creosol (2-methoxy-4-methylphenol) (12.48%) (Montazeri et al., 2013; Oramahi et al., 2024).

Meanwhile, at 175°C, the composition shifted to nine main compounds, including furfuryl alcohol (1.75%), cyclopentene (4.57%), furfural (7.85%), 2-

acetylfuran (2.03%), guaiacol (55.45%), creosol (17.97%), [unidentified phenol derivative] (4.63%), as well as organic acids: acetic acid (4.11%) and propionic acid (1.64%).

At the maximum distillation temperature of 200°C, the variety of chemical compounds decreased significantly, resulting in only six main components. These compounds include 2-methyl-2-cyclopenten-1-one (6.60%), cyclopentenone (5.01%), furfural (8.31%), guaiacol (2-methoxyphenol) (60.80%), creosol (2-methoxy-4-methylphenol) (4.19%), and acetic acid (6.96%). Temperature variations evidently alter the composition of the distillation products. For instance, compounds such as 2-acetylfuran, which were present at lower temperatures, were not detected at 200°C. Similarly, the compound identified as [5-methyl-4-methylene derivative], which appeared at 175°C, was eliminated at 200°C (Harvey et al., 2015; Pham et al., 2018).

## CONCLUSION

From the research, manufacturing liquid smoke from candlenut shell can be concluded as follows:

- 1) Percentage of maximum product of liquid smoke produced from candlenut shell is at a temperature ranging from 350-450 °C and pyrolysis time of 3.5 hours, with a phenol content of 57.3% of the total amount of liquid smoke produced
- 2) The percentage of the maximum product of liquid smoke produced from the distillation of liquid smoke from a candlenut shell is at a temperature of 200 °C, and the phenol content is 73.42% with the highest yield of 38.74% of the total number of pyrolysis products.
- 3) Distillation is done on a liquid smoke product, which can result in pyrolysis, increasing the levels of phenol to achieve an increase of 16.12%. If carried out at a temperature of 175 °C, the percentage of product yield amounted to 36.49 % of the total number of pyrolysis products.

## CONFLICT OF INTEREST

No conflict of interest in this research.

## ACKNOWLEDGEMENTS

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