

## Production of Food – Grade Liquid Smoke from Candlenut Shell through a Consecutive Pyrolysis – Distillation Process

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### Abstract

*Concerning the increasing demand for food-grade liquid smoke, this research focuses on liquid smoke purification through distillation to improve quality, in terms of color, odor, and chemical composition, while eliminating carcinogenic compounds such as tar residues. For that reason, this study aims to investigate the production of liquid smoke from candlenut shells using a 300 kg capacity pyrolizer. Pyrolysis was conducted at 350 – 400°C for 1 – 6 hours to produce liquid smoke. Distillation temperature and time were varied to examine their effect on liquid smoke quality. Based on the phenol content, an optimal pyrolysis condition was achieved at temperatures between 350 and 450°C for 3.5 hours from which the highest phenol content value (56.76% w/w) was obtained. In addition, distillation at 200°C for 40 minutes resulted in the highest distillate yield of 38.74% w/w, which corresponds to a phenol content of 73.42% w/w. Furthermore, heavy metal contamination tests revealed that mercury, arsenic, cadmium, and lead levels were well below their maximum tolerable limit for food additives. These results demonstrate that the distillation condition significantly influences the quality and yield of liquid smoke.*

**Keywords:** Candlenut; distillation; food ingredient; liquid smoke; pyrolysis.

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### INTRODUCTION

As a tropical country, Indonesia is rich in variety of both native and non-native crops with wide potential

applications. Candlenut, coconut, and oil palm are among the leading crop commodities in Indonesia. Aceh Province is a good example, where agriculture

and plantations, including oil palm, coffee, rubber, coconut, cocoa, sugarcane, areca nut, candlenut, nutmeg, patchouli, cinnamon, cloves, lemongrass, pepper, sago, tobacco, and sugar palm dominate the crop-based business. Aceh's leading industrial crop commodities production data are presented in Figure 1 (Agussabti et al., 2022; Dewi et al., 2022).

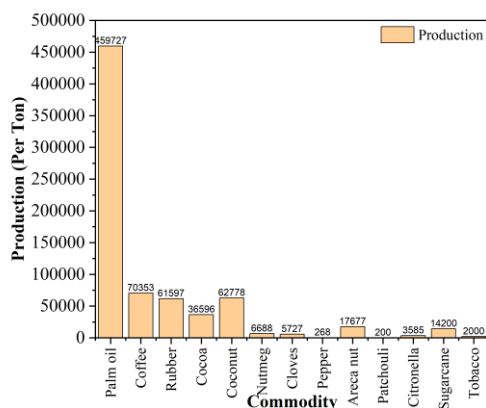


Figure 1. Aceh Industrial Zone's leading commodities

Candlenut is one of the important crops in Aceh that is planted by people in all districts in Aceh province (Syimir Fizal et al., 2022). In 2012, Aceh Province was once the largest candlenut-producing province in Indonesia, with the candlenut plantation area spanning 55,372 hectares, which accounts for about 35.631% of Aceh's total area. The area of candlenut plantations in all districts of Aceh Province is shown in Table 1.

Table 1. Plantation crop area data (hectares)

Crops	Plantation Area (Hectares)					
	2012	2013	2014	2015	2016	2017
Betel Nut	41,065	38,053	38,220	38,721	39,244	39,844
Candlenut	20,271	19,858	18,523	18,908	16,874	16,840
Nutmeg	21,031	21,104	21,580	22,043	23,438	23,994

Source: (Badan Pusat Statistik, 2018)

The most important potential of candlenut lies in the fruit, which comprises shell, pulp and seed. Candlenut seed kernel can be processed into an edible oil, leaving the candlenut shell as the by-product. However, this residue is underutilized and has yet to be optimally utilized (Shaah et al., 2021). Similar to most plant biomass residues, candlenut shell is rich in hemicellulose, cellulose, and lignin content. Therefore, its vast availability and low cost make this biomass residue become a promising raw material for the production of liquid smoke (wood finer) (Balat, 2011). Liquid smoke is a mixture of water-soluble wood smoke dispersion solutions, produced by condensing smoke from pyrolysis. Smoke is defined as a suspension of solid and liquid particles in a gas medium (Rizal et al., 2020). Liquid smoke can be produced by pyrolysis through direct or indirect combustion that yields a wide range of organic compounds, namely phenols, carbonyls, acids, furans,

alcohols, and various polyaromatic hydrocarbon compounds (Guillén et al., 2001). Furthermore, a distillation process can result in a purer and toxic compounds-free liquid smoke, which can be used in the food and pharmaceutical industries (Šimko, 2005). Liquid smoke offers various benefits, both as a raw material and as a support for the chemical industries, such as medicines, antiseptics, natural preservatives, and flavors (Shaah et al., 2021). However, it's important to consider that heavy metals like cadmium, mercury, arsenic, and lead levels in the liquid smoke must be within safe limits. The permissible levels for cadmium, mercury, arsenic and lead are 2 mg/kg, 1 mg/kg, 3 mg/kg and 0.001 mg/kg., respectively.

Pyrolysis is a thermochemical method for converting biomass materials into various chemical products, including liquid organic oil, organic dust, and pyrolysis gas. Based on the heating rate applied, biomass pyrolysis can be classified into three categories: gradual heating, rapid heating, and very rapid heating (Khuenkao & Tippayawong, 2020). This biomass decomposition process comprises three main stages: the initial stage, also known as devolatilization stage, primary decomposition stage, and repolymerization stage. These stages occur in series and parallel, and are influenced by several process parameters (Varma et al., 2018). For food-grade liquid smoke, the controlled decomposition of lignin is the most critical reaction. Lignin serves as the primary precursor of phenolic compounds, such as guaiacol and creosol, which confer the essential antioxidant properties and smoky aroma required for food additives. The general reaction pathways for the three main biomass components can be described as follows:

1. Lignin decomposition: Lignin  $\rightarrow$  Phenols + Guaiacol + Syringol + Vanillin + Bio-oil + Gases.
2. Hemicellulose decomposition: Hemicellulose  $\rightarrow$  Acetic acid + Furfural + Hydroxyacetone + Gases + Bio-oil.
3. Cellulose decomposition: Cellulose  $\rightarrow$  Levoglucosan + Hydroxyacetone + Acetic acid + Formaldehyde + Gases + Bio-oil + Bio-char.

The process parameters were strictly maintained to optimize these reactions. By maintaining the pyrolysis temperature between 350 – 450°C, secondary reactions that lead to the formation of carcinogenic polycyclic aromatic hydrocarbons (PAHs) can be prevented. Previous research proved that during pyrolysis, biomass undergoes thermal degradation in the absence of oxygen at a temperature of 350 – 450°C to produce liquid smoke with the yield obtained ranging between 30 and 70% w/w, composed of numerous compounds, mainly oxides, which can be easily separated into two fractions. (Sulhatun et al., 2018, 2019). The fraction is insoluble in water and is a concentrated tar with a specific gravity higher than that of water (Balat, 2011). In addition to producing liquid smoke, pyrolysis of biomass will result in biochar, tar, and non-

condensable gases as by-products that can be used as an alternative energy source. Liquid smoke obtained from pyrolysis depends on the raw materials and pyrolysis temperature. Pyrolysis of coconut shell produces coconut shell liquid smoke with a content of 4.13% phenolic compounds, 11.3% carbonyl, and 10.2% acid (Budaraga et al., 2016).

To overcome the steady increase in biomass residue production from candlenut processing and the continuous reduction of final disposal sites, an innovative process is developed to convert this residue into other valuable products (Shaah et al., 2021). In this study, candlenut shell was used as the raw material for the preparation of liquid smoke through a pyrolysis process, followed by a distillation process to produce food-grade liquid smoke by improving the quality of liquid smoke in terms of color, smell, and its composition, primarily to increase phenol level and reduce the toxic and carcinogenic polycyclic aromatic hydrocarbons (PAHs) levels. Temperature and duration of the distillation process influence the increase in liquid smoke quality, along with an increase in pyrolysis yield (Demirbas, 2009). Process and equipment innovations include a biomass pyrolysis equipment equipped with a double-condenser system, a distillation equipment for liquid smoke purification, and a combustion furnace employing an efficient fuel as the energy source for both the pyrolysis reactor and the distillation column (Rex et al., 2023). 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 operation, and other parameters, such as the use of catalysts and vapor condensation mechanisms (Garcia-Perez et al., 2008; Zhang et al., 2018).

The primary objective of this research is to investigate the effect of distillation temperature and time on the quality of liquid smoke, and to assess the yield and quality of liquid smoke derived from biorefinery technology of candlenut shell before and after distillation. This biorefinery innovation technology is employed to process candlenut shell biomass into liquid smoke as an alternative food additive.

## MATERIAL AND METHOD

### Materials

Candlenut shell (approximately 1000 kg) as the raw material was obtained from a candlenut processing industry in Aceh province, Indonesia. Prior to pyrolysis, the candlenut shell was washed with fresh water to remove dirt and further dried in an oven at 105°C for 24 hours to reduce the moisture.

### Equipment

This research was conducted in stages, including the preparation of the feedstock, biomass pyrolysis, liquid

smoke distillation, and the product analysis stage. The pyrolysis and distillation equipment were made of stainless steel with their physical appearance are presented in Figures 2 and 3.

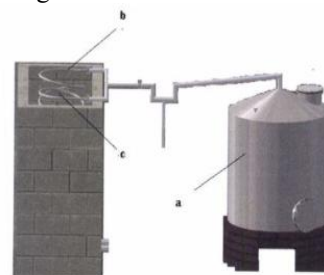


Figure 2. The designed and fabricated pyrolysis equipment used for liquid smoke preparation study



Figure 3. The designed and fabricated pyrolysis equipment used for liquid smoke purification study

### Liquid smoke production and purification

Liquid smoke production was carried out utilizing a batch pyrolysis reactor equipped with an own-build spiral-shaped double condenser (Figure 4). A carefully weighed candlenut shell was introduced into the pyrolysis reactor and the heat was immediately supplied to the pyrolysis reactor to allow the pyrolysis reaction to occur. The pyrolysis process temperatures were varied between 300 to 450°C, while the process duration was set at 6 hours. Flow rates of the incoming and outgoing cooling water were adjusted to maintain its temperature to be around 28 – 35°C. This process resulted in a grade 3 liquid smoke product, which is then collected in the tank.



Figure 4. Liquid smoke production process using the pyrolysis method: (a) Candlenut shells collected as raw material; (b) The initial drying and preparation of the candlenut shells; (c) The batch pyrolysis reactor unit; (d) The collected grade 3 liquid smoke product; (e) Collection of the liquid smoke distillate from the

double condenser system; and (f) The combustion furnace supplying heat to the pyrolysis reactor.

For the purification process, a determined volume of liquid smoke, ranging from 0 to 35 liters, was introduced into the distillation equipment and subsequently heated according to the temperature studied. Two independent variables were studied for the distillation process, namely the distillation time and temperature. Distillation time was studied at 15, 30, 45, 60, 75, 80, 95, and 110 minutes, whereas the distillation temperatures were investigated at 150°C, 175°C, and 200°C. Distillation temperature was controlled by adjusting the gas flow rate into the furnace. The fixed variables, including acidity (pH), phenol content, and yield (%) of liquid smoke generated.

### Analytical procedure

Physical and chemical analysis of the liquid smoke before and after distillation were conducted by measuring the density, acidity, yield (%) of liquid smoke and phenol levels (GC-MS) (Guillén et al., 2000).

### RESULTS AND DISCUSSION

Liquid smoke has been successfully prepared from candlenut shell through a combined pyrolysis and distillation process. The results are discussed below.

#### Influence of pyrolysis time and temperature on liquid smoke yield

The results obtained demonstrate that the increase in pyrolysis time results in an improved liquid smoke yield (Hasan et al., 2019). Liquid smoke yield increased sharply from 2.5 to 3 hours, but it decreased from 5 to 5.5 hours. This is because at a longer pyrolysis time, biomass undergoes a longer contact with the heat that promotes its decomposition, producing liquid smoke (Hasan et al., 2019; Zheng et al., 2012).

Similar to pyrolysis of other biomass, the pyrolysis of candlenut shell also occurs through several complex thermochemical stages that are water evaporation followed by decomposition of biomass material such as hemicellulose, lignin and cellulose that produce liquid smoke, gas - light gases CO and CO<sub>2</sub>. During pyrolysis, water evaporation mainly happens in the first 1.5 hours that corresponds to pyrolysis temperatures between 150°C – 250°C. Subsequently, the decomposition of hemicellulose, cellulose, and lignin occurs at temperatures ranging from 350 – 450°C (Bennadji et al., 2013; Wang et al., 2019). The decomposition of biomass proceeds with increasing pyrolysis temperature and time, resulting in the formation of more charcoal residue. Pyrolysis at temperatures of 350 – 450°C produces compounds of high organoleptic quality. The pyrolysis temperature affects the distribution, composition and properties of the product. Generally, the bio-oil yield is very high at pyrolysis temperatures between 400°C and 550°C, and then decreases at higher pyrolysis temperatures. At

temperatures above 600°C, bio-oil and char will further decompose into gas through numerous side reactions during secondary pyrolysis (Ali et al., 2016; Patra et al., 2017).

The formation of polar, aliphatic and aromatic compounds in bio-oil increases with the rise in temperature, which ranges from 300°C to 800°C. However, the formation of polycyclic aromatic hydrocarbons (PAHs), pyrene and phenanthrene, as well as decarboxylation and dehydration reaction formation, usually more likely to occurs at temperatures 700°C of higher. In addition to liquid formation, a higher pyrolysis temperature also yields a variety of gases, such as CO, CO<sub>2</sub>, CH<sub>4</sub>, etc. (Keiluweit et al., 2012; Matamba et al., 2020).

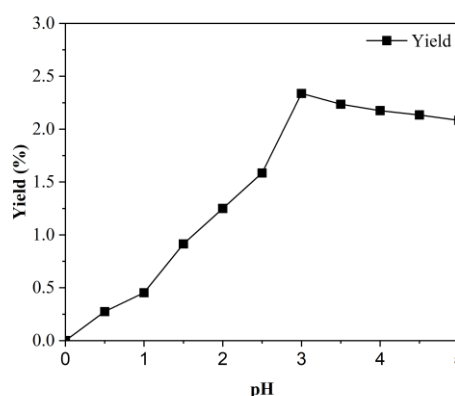


Figure 6. Correlation between pH and liquid smoke yield (%)

Based on Figure 6, the pyrolysis of candlenut shell at 350 – 450°C (10-hour process) results in the highest liquid smoke yield of 2.33% w/w, which corresponds with a pH value of 3. The result indicates that the liquid smoke produced still has a high acid content (Budaraga et al., 2016).

#### Physicochemical properties of raw liquid smoke

The chemical composition of the liquid smoke resulting from candlenut shell pyrolysis analyzed using GC-MS before distillation is displayed in Figure 7.

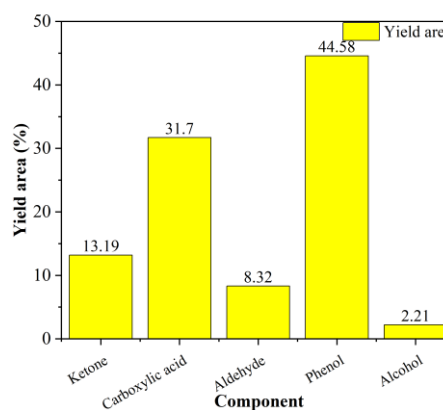


Figure 7. Chemical composition of liquid smoke

Based on the GC-MS analysis of liquid smoke obtained from the pyrolysis of candlenut shells; 5 main group of components were identified. These include phenol (44.58%), carboxylic acid (31.70%), ketone (13.19%), aldehyde (8.32%), and alcohol (2.21%). The dominant phenolic compounds identified in the liquid smoke are 2-methoxyphenol (guaiacol) and 2-methoxy-4-methylphenol (creosol), with concentrations of 40.69% and 3.89%, respectively. Consequently, the total content of these phenolic compounds is 44.58%. In most biomass pyrolysis, the resulting liquid smoke indicates the presence of polyaromatic hydrocarbons that are carcinogenic, making the liquid smoke becomes hazardous. Therefore, it is necessary to separate them through the distillation process to eliminate or totally remove the carcinogenic components (Ding et al., 2007; Guillén et al., 2000; Oramahi et al., 2024).

### Influence of distillation time and temperature on liquid smoke yield

Figure 8 demonstrates that liquid smoke distillation carried out at 150°C, resulting in a liquid smoke distillate with a product yield of only 24.77% w/w, which is the lowest compared to other distillation temperatures applied. At distillation temperatures of 175°C and 200°C, the liquid smoke yields reached were 36.49% w/w and 38.74% w/w, respectively. A higher distillation temperature promotes the vaporization of more volatile compounds, leading to a higher product yield (Niu et al., 2016; Zhao et al., 2019).

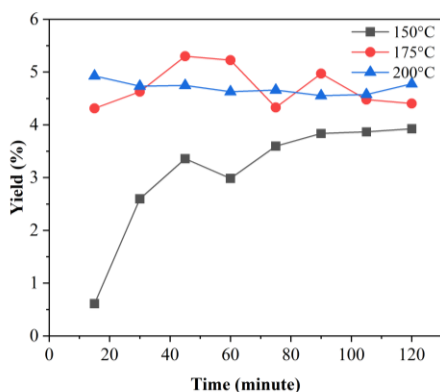


Figure 8. Effect of distillation time and temperature on liquid smoke yield

The density and pH values of liquid smoke generated at each distillation temperature are presented in Table 2. As seen in Table 2, a higher distillation temperature results in liquid smoke with a lower density. However, the difference in the density of the liquid smoke obtained is not significant. Similar observations were also found for the pH of the liquid smoke attained. A similar pH identifies the similarity of the components

present in liquid smoke and their composition.

Table 2. Acidity (pH) and density of liquid smoke after distillation

Temperature (°C)	Density (kg/m <sup>3</sup> )	pH
150	0.9808	4.55
175	0.9772	4.56
200	0.9779	4.23

### Physicochemical properties of distilled liquid smoke

The chemical composition of liquid smoke from distillation at temperatures of 150°C, 175°C, and 200 °C are depicted in Figures 9 and 10.

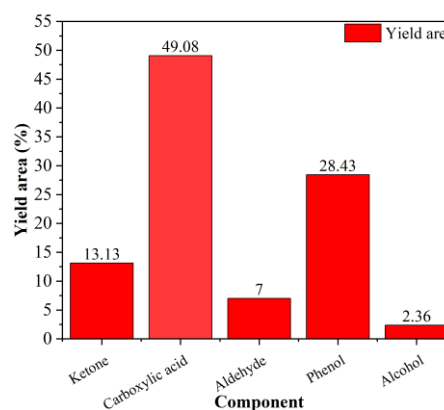


Figure 9. Chemical composition of liquid smoke after first distillation

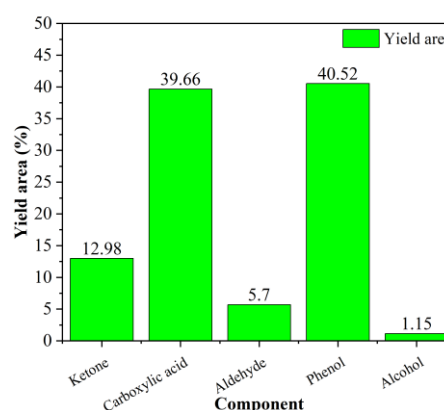


Figure 2. Chemical composition of liquid smoke after second distillation

Figure 9 displays that liquid smoke obtained from one step distillation of candlenut shells comprises 5 main components. These include carboxylic acid (49.08% w/w), phenol (28.43% w/w), ketone (13.13% w/w), aldehyde (7% w/w), and alcohol (2.36% w/w). Meanwhile, Figure 10 shows that liquid smoke obtained from two steps distillation of candlenut shells contains 5 main components. These include carboxylic acid (39.66% w/w), phenol (40.52% w/w), ketone (12.98% w/w), aldehyde (5.7% w/w), and alcohol (1.15% w/w). It is obvious that ketone and

aldehyde content decline slightly as the distillation step increase for ones and twice. Alcohol content declines to only nearly 50% from its original content as the number of liquid smoke distillation rises from 1 to 2. Similarly, carboxylic acid content also decreases sharply from 49.08% w/w to 49.08% w/w. As expected, phenol content of liquid smoke increases from 28.43% w/w to 40.52% w/w as the number of liquid smoke distillation rises from 1 to 2.

The analysis results in Table 3 shows that the lowest distillation temperature (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% w/w and continued to increase at a temperature of 175 °C, reaching 73.42% w/w, before declining at a temperature of 200 °C to 64.99% w/w (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% w/w), cyclohexanone (3.34% w/w), [cyclopentene derivative] (8.09% w/w), cyclopentenone (8.91% w/w), furfural (14.27% w/w), 2-acetylfuran (3.09% w/w), guaiacol (2-methoxyphenol) (41.04% w/w), and creosol (2-methoxy-4-methylphenol) (12.48% w/w) (Montazeri et al., 2013; Oramahi et al., 2024). Meanwhile, distillation of candlenut shell liquid smoke at 175°C resulted in the product with nine main compounds, including furfuryl alcohol (1.75% w/w), cyclopentene (4.57% w/w), furfural (7.85% w/w), 2-acetylfuran (2.03% w/w), guaiacol (55.45% w/w), creosol (17.97%), [unidentified phenol derivative] (4.63% w/w), as well as organic acids: acetic acid (4.11% w/w) and propionic acid (1.64% w/w). At the highest distillation temperature of 200°C, the number of chemical compounds decreased significantly, leaving only six main compounds. These compounds include 2-methyl-2-cyclopenten-1-one (6.60% w/w), cyclopentenone (5.01% w/w), furfural (8.31% w/w), guaiacol (2-methoxyphenol) (60.80% w/w), creosol (2-methoxy-4-methylphenol) (4.19%), and acetic acid (6.96% w/w). Evidently, temperature variations 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 identified at 175°C, was diminished at 200°C (Harvey et al., 2015; Pham et al., 2018). The chemical compositions of candlenut shell liquid smoke obtained from distillation at 150°C, 175°C, and 200°C are summarized in Table 3.

Table 3. Chemical composition of liquid smoke distilled at various temperatures.

No.	Component	Area (%)		
		150°C	175°C	200°C
1.	Cyanobutadiena (pentadienitrile)	2.67	-	-
2.	Alcohol			
	Furan methanol	-	1.75	-
3.	Ketone and aldehyde compounds			
	Cyclohexanone	3.34	4.57	6.60
	2 Cyclopenten-1-one 2-methyl	8.09	-	-
	Cyclopentenone	8.91	-	5.01
4.	Furan and piran furfural compounds			
	2 Furan			
	carboxilaladehide	14.27	7.85	8.31
	2 Acetyl 1 fural	3.09	2.03	-
5.	Phenol compound and derivatives			
	2-Methoxyphenol (guaiacol)	41.04	55.45	60.8
	2 Methoxy-4-methyl phenol	12.48	17.97	4.19
6.	5 Methyl 4 methylene	3.60	4.63	-
7.	Acid compounds			
	Acetic acid	-	4.11	6.96
	Propanoic acid	-	1.64	-
8.	1,2 Ethanediol	2.51	-	-
	Total	100	100	100

## CONCLUSION

The production of liquid smoke from candlenut shell via pyrolysis process at 300 – 450°C effectively results in a high-value product rich in phenolic compounds. Chemical analysis confirms that the resulting liquid smoke, both before and after distillation at temperatures of 150 – 200°C, did not contain carcinogenic compounds commonly found in other types of biomass, specifically benzo[a]pyrene. The distillation process serves as an essential purification step, physically separating unwanted residues from liquid smoke, significantly enhancing its quality without altering its inherent safety profile. Furthermore, mercury, arsenic, cadmium, and lead levels in the resulting liquid smoke were well below the maximum tolerable limits for food additives. Consequently, candlenut shell liquid smoke meets the stringent safety and quality standards required for food flavoring and pharmaceutical applications.

## CONFLICT OF INTEREST

No conflict of interest in this research.

## ACKNOWLEDGEMENTS

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