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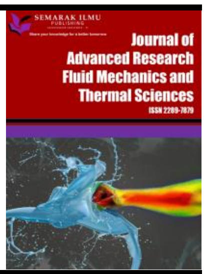

Characterization of Distillation and Redistillation Product of Coconut Shell Liquid Smoke at Various Temperatures

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ABSTRACT

This study investigates the impact of distillation temperature on phenol content in coconut shell liquid smoke, a product derived from pyrolysis and subsequent condensation processes. Liquid smoke, renowned for its versatile applications including preservation, antioxidation, antiseptic, and antibacterial properties, necessitates distillation for both quality enhancement and safety. The research comprises a two-stage distillation, featuring 11 temperature variations (100-150°C) in the first stage and 9 variations (75-155°C) in the second stage. Characterization involves pH assessment and Gas Chromatography-Mass Spectrometry (GC-MS) analysis to identify chemical components. The pH range in the initial distillation stage spans 1.18-1.30, and the second stage ranges from 1.11-1.39. GC-MS analysis of raw coconut shell liquid smoke reveals 25 components, including phenol, furan, ketone, acid, alcohol, benzenediol and derivatives, alkyl aryl ether, deoxycytidine, and silicone grease. GC-MS evaluation is conducted on products of the first stage at 125 and 135°C, and the second stage products at 145 and 155°C. In the first distillation stage, liquid smoke contains phenol and assorted compounds. Conversely, the second stage yields purer phenol compounds. The findings suggest potential applications of the redistillation product as raw material in food preservation, antiseptics, and insecticides.

1. Introduction

Coconuts, a hallmark of Indonesia's abundant resources, are deeply ingrained in the nation's economy and culture. The ubiquity of coconut trees across diverse regions underscores their role as a vital natural asset. Indonesia's Central Bureau of Statistics (BPS) has recorded an expansive coconut plantation area encompassing approximately 3.4 million hectares, yielding an impressive annual output exceeding 2.8 million tons of coconuts [1,2]. Notably, the Yogyakarta region contributes significantly to this landscape, contributing around 46,000 tons annually. However, coconuts offer more than just quantity; their multifaceted utility spans every part, including the often-underestimated coconut shell. This versatile shell finds purpose across diverse domains, from

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traditional handicrafts to serving as a potent source of solid fuel, particularly in the form of charcoal. Remarkably, the coconut shell's composition, encompassing cellulose (36.13%), hemicellulose (20.36%), and lignin (32.33%), renders it a prime candidate for biomass charcoal production [3,4]. The pyrolysis process used in this endeavor yields not only charcoal but also gas or smoke, carrying residual compounds of significance, including phenol [4]. In recent times, the emergence of liquid smoke industries highlights the potential of this product in various applications, even though it remains in a developmental stage. Liquid smoke holds great promise due to its chemical composition, which imparts attributes such as preservative, antioxidant, antiseptic, and antibacterial capabilities [5].

Coconut shell liquid smoke boasts a complex composition comprising main components such as acetic acid, carbonyl, benzene, phenol, and their derivatives. Predominating this composition are phenol and its derivatives stemming from the pyrolysis-induced decomposition of lignin [6-8]. However, the combustion process at elevated temperatures introduces harmful compounds, necessitating quality enhancement measures. Distillation stands out as a valuable technique to eliminate these undesirable constituents, operating within a temperature range of approximately 100 to 200°C [9]. Apart from its purification role, distillation enhances the visual clarity of the liquid smoke, offering a versatile solution to compound separation based on boiling point discrepancies [10,11]. This process's accessibility and effectiveness extend to laboratory environments, where its implementation requires straightforward equipment, aligning well with its cost-effective and adaptable nature.

This study delves into a two-stage distillation process aiming to elevate the quality of coconut shell liquid smoke. The initial phase targets the removal of harmful compounds, while a subsequent redistillation stage seeks to isolate pure phenol compounds. Phenol's diverse attributes, encompassing antioxidative, antibacterial, and antifungal properties, extend its potential utility to food preservation [9,12-16]. Thus, the study's primary objective is to establish optimal conditions that yield enhanced quantities of coconut shell liquid smoke, simultaneously maximizing phenol content. Through this exploration, the research contributes valuable insights to harnessing the potential of coconut shell liquid smoke across various applications.

2. Methodology

2.1 Apparatus and Materials

This study used a comprehensive array of distillation apparatus, encompassing essential tools such as an electric oven, thermometer, condenser, still head, distilling flask, receiving flask, and temperature controller. The primary material under investigation comprised liquid raw materials derived from the conventional pyrolysis process applied to coconut shells, conducted within the temperature spectrum of 250-350°C. These raw materials were sourced from the charcoal industry located in Sleman, situated within the Special Region of Yogyakarta, Indonesia.

2.2 Methods

The methodology used in this research centered around the distillation process, comprising two distinct stages aimed at characterizing the resultant liquid smoke from coconut shells across varying temperature conditions. The primary objective encompassed the elimination of detrimental compounds present in the liquid smoke while concurrently isolating pure phenol compounds. The initial stage of distillation involved subjecting the coconut shell liquid smoke to a range of temperatures to ascertain the most optimal distillation temperature. This first stage encompassed

11 temperature variations: 100, 105, 110, 115, 120, 125, 130, 135, 140, 145, and 150°C. Subsequently, the second stage of distillation was conducted using a sample derived from the outcome of the first stage, specifically utilizing the distillate volume with the highest yield. This second stage comprised 9 temperature variations: 75, 85, 95, 105, 115, 125, 135, 145, and 155°C. The experimental setup involved placing a 100 mL sample within a distillation flask, followed by heating the flask within an electric oven. A temperature controller was implemented to ensure precise control of the distillation temperature. The distillation process was conducted over a duration of 1.5 hours, during which distillate volumes were recorded at 10-minute intervals. To maintain consistent cooling conditions, the cooling water temperature was maintained at 17°C. The schematic representation of the distillation apparatus is provided in Figure 1, while Figure 2 depicts the assembly of the distillation tools utilized in the study.

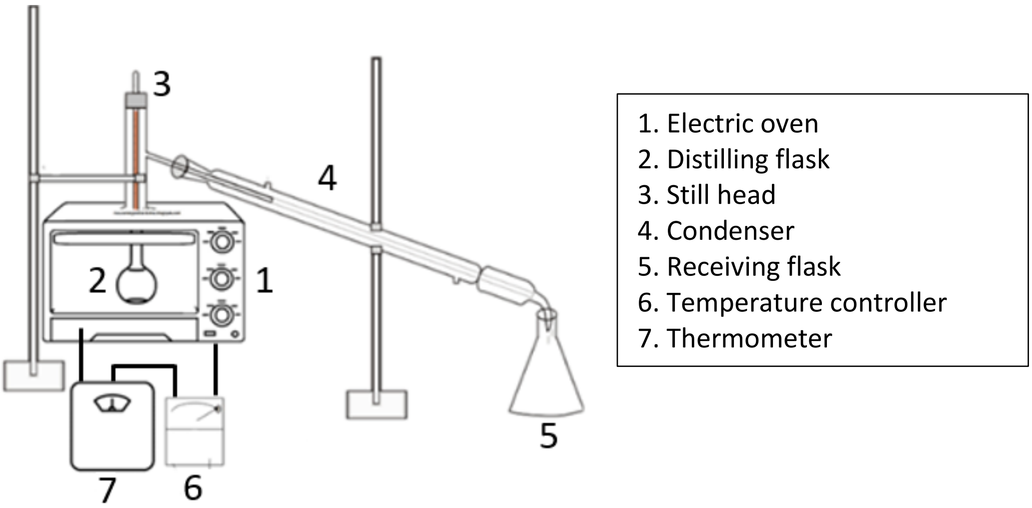

Fig. 1. Schematic diagram of distillation tool

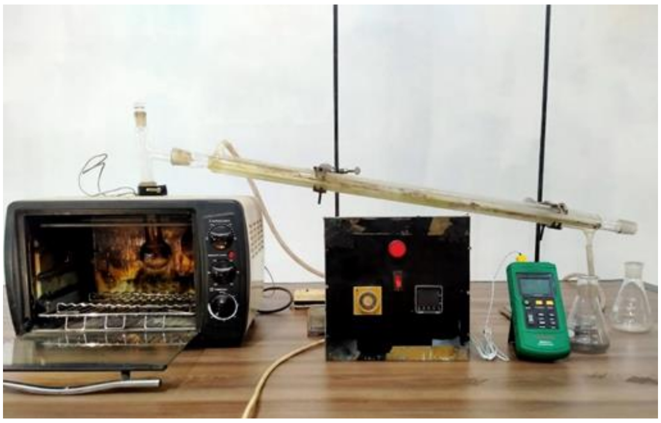

Fig. 2. Pictorial view of distillation tools

The pH assessment was performed to quantify the acidity level within the distillation product. The measurement of pH values was executed using a digital pH meter, specifically the ATC 2011 model. To ensure accuracy, the pH meter was calibrated utilizing buffer solutions characterized by pH values of 4 and 7. For compound composition analysis, Gas Chromatography-Mass Spectrometry (GC-MS) was used. The specific GC-MS model utilized was the Agilent Technologies 7890B Shimadzu GC-2010 Plus. The analysis encompassed both the raw material sample and the distillation product, with the aim of discerning alterations in the composition of liquid smoke compounds after the distillation process. This analysis facilitated the identification and quantification of distinct compound content variations brought about by the distillation procedure.

3. Results

3.1 Coconut Shell Liquid Smoke Distillation Product

The distillation process serves as a critical mechanism for purifying liquid smoke, effectively segregating compounds to eliminate undesirable and hazardous components. This separation is achieved through exploiting the varying boiling points of the constituents present in the liquid smoke [17]. A comprehensive evaluation of the coconut shell liquid smoke via GC-MS analysis revealed the presence of several undesirable compounds, including furans, ketones, acids, alcohols, benzenediol and its derivatives, as well as alkyl, aryl, and ethers. To obtain purer phenolic compounds and eliminate harmful substances, a two-stage distillation approach was adopted, ensuring the safety of the liquid smoke for various applications. The process's efficacy was substantiated by the discernible enhancement in liquid smoke quality, indicated by the transformation in color from a deep brown to a vivid yellow, signifying the separation of tar compounds [18]. Moreover, the resulting distillate displayed a distinctive aromatic profile attributed to the aromatic compounds present within. The distillation temperature emerged as a pivotal factor influencing both the process and the attributes of the distillate. During the first distillation stage, conducted within the 100-150°C temperature range with 5°C increments, a notable pattern emerged, as shown in Figure 3(a). Notably, a significant increase in distillation volume was observed between 100-135°C, followed by a decline post-135°C due to escalated evaporation rates beyond the condenser's capacity [19]. The optimal temperature for this stage was determined as 135°C, as it yielded the highest distillate volume. Transitioning to the second distillation stage, where temperatures ranged from 75 to 155°C with 10°C differences, similar trends emerged, as shown in Figure 3(b). Temperatures below 105°C failed to yield distillate due to insufficient evaporation of volatile compounds. Conversely, temperatures within the range of 115-145°C exhibited a gradual and stable rise in distillate volume, while 155°C resulted in a decrease due to excessive evaporation leading to liquid smoke escaping the condenser [20]. The second distillation stage's optimal temperature was identified as 145°C, as it produced the highest distillate volume. These findings collectively underscore the pivotal role of distillation temperature in influencing both the process's efficiency and the quality of the obtained distillate. The research contributes to a deeper understanding of the intricacies of the distillation process for liquid smoke purification, enhancing its potential for safe and diverse applications.

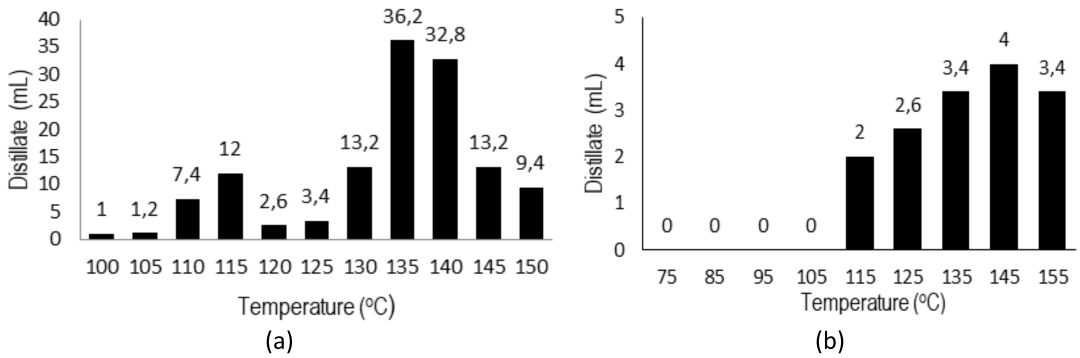

Fig. 3. (a) The first stage of distillation product (b) The second stage of distillation product

The analysis of distillation product volume data presented in Table 1 yielded a key insight: the peak yield within the first distillation stage was attained at a temperature of 135°C, signifying the optimal condition for this phase. Consequently, the liquid smoke sample selected for the subsequent second distillation stage derived from the product of the first stage, obtained at the specified temperature of 135°C. Moreover, the subsequent investigation of the second distillation stage pinpointed its peak yield at a temperature of 145°C, affirming the optimal operational temperature for this phase as well. This dual optimization not only enhances the efficiency of the distillation process but also offers a methodical approach to obtaining higher yields in both stages while ensuring the highest quality of the resultant liquid smoke distillate.

Table 1
Distillation yield of the first and second distillation stages

1st Stage	Temp.(°C)	100	105	110	115	120	125	130	135	140	145	150
Distillation	Yield (%)	1.0	1.2	7.4	12.0	2.6	3.4	13.2	36.2	32.8	13.2	9.4
2nd Stage	Temp.(°C)	75	85	95	105	115	125	135	145	155		
Distillation	Yield (%)	n/a	n/a	n/a	n/a	2.0	2.6	3.4	4.0	3.4		

3.2 pH Value of Coconut Shell Liquid Smoke Distillation Product

The pH value represents a crucial parameter used to gauge the quality of the distillation product, offering valuable insights into the chemical decomposition processes within coconut shells that yield organic acids present in liquid smoke [21]. Previous research endeavours have shed light on this aspect; Anggraini and Yuniningsih [22] reported a pH value of 1.41 for liquid smoke derived from coconut shells, while Sari *et al.,* [15] observed a pH value of 2.4 for coconut shell liquid smoke subjected to redistillation using the vacuum method at 81°C. Further research by Saloko *et al.,* [23] yielded a pH value of 2.54 for the redistillation of coconut shell liquid smoke at 98±2°C. The pH values obtained from the distillation product are outlined in Table 2. The initial-stage distillation product exhibited pH values ranging from 1.18 to 1.30. At distillation temperatures of 100 and 105°C, the limited volume of distillate precluded pH value determination due to electrode immersion constraints. In contrast, the second-stage distillation product exhibited pH values spanning from 1.11 to 1.39. Notably, distillation temperatures between 75 and 105°C yielded no distillate during the second stage, rendering pH value data available solely within the temperature range of 115-155°C. Interestingly, despite differences in distillation stages, pH values remained largely consistent. This homogeneity stems from the similar compound composition present in the distillation outcomes of coconut shell liquid smoke. The inherent acidic nature of organic acids within liquid smoke accounts for the lower pH values, whereas the dominant phenol compound also contributes to the acid classification of liquid smoke pH [24]. Phenols, characterized by the release of H+ ions from hydroxyl groups, further accentuate the acidity. The observed low pH values bear significance, indicating high-quality liquid smoke distillation products with extended shelf life [22]. Moreover, these low pH values hold potential as food preservatives, augmenting the utility of coconut shell liquid smoke within diverse applications [25,26].

Table 2
The pH value of the distillation result

1st Stage	Temp.(°C)	100	105	110	115	120	125	130	135	140	145	150
Distillation	pH	n/a	n/a	1.30	1.25	1.27	1.28	1.18	1.23	1.18	1.22	1.22
2nd Stage	Temp.(°C)	75	85	95	105	115	125	135	145	155		
Distillation	pH	n/a	n/a	n/a	n/a	1.34	1.39	1.11	1.11	1.23		

3.3 Characterization of Coconut Shell Liquid Smoke Distillation Product Using GC-MS

Gas Chromatography-Mass Spectrometry (GC-MS) played a pivotal role in dissecting the compounds present in the distillation process of coconut shell liquid smoke. Identification of these compounds was achieved by cross-referencing their mass spectra with data from the GC-MS system's spectral database. The analysis of coconut shell liquid smoke raw materials yielded insightful results, as illustrated in Figure 4 and Table 3. The raw material analysis revealed the presence of 25 distinct chemical components, including phenol (21.66%), furans (5.23%), ketones (2.60%), acids (54.41%), alcohols (2.21%), benzenediol, and its derivatives (8.73%), alkyl aryl ether (4.06%), deoxycytidine (0.46%), and silicone grease (0.63%). These findings underlined the necessity for the distillation process, as the raw liquid smoke retained harmful chemical compounds that required separation. Sari *et al.,*'s [15] study corroborated this need, showing the GC-MS analysis of redistilled liquid smoke derived from coconut shells using the vacuum method, encompassing carbonyl (0.62%), acids, and derivatives (0.07%), furan and pyran derivatives (0.11%), phenol (2.71%), alcohols (35.57%), and methyl ethyl ether (18.32%).

The subsequent GC-MS analysis of coconut shell liquid smoke distillation outcomes is illustrated in Figure 5 and Table 4. The initial distillation stage at 125°C revealed five predominant chemical compounds: ethyl iso-allocholate (65.27%), phenol (CAS) izal (25.20%), phenol 2-methoxy-(CAS) guaiacol (4.41%), phenol 4-methoxy-3 methyl (CAS) 4-(2-benzo[1,3]dioxol-4-yl-vinyl) (2.43%), and 8-methoxy-12a-methyl 1, 2, 3, 3a, 3b, 4, 5, 6, 7, 8, 9, 10, 10b, 11, 12, 12a-hexadecahydro-benz (2.70%). This stage notably yielded alcohol, phenols, and their derivatives as the predominant compounds. Distillation at 135°C resulted in phenol (CAS) izal (71.30%), phenol 2-methoxy-(CAS) guaiacol (12.33%), N-(benzylidene)-2,2-dimethyl-1-(4-methyl) phenylcyclopropylamine (5.57%), indole-2-acetic acid alpha-(1-acetyl-3-ethylidene-4-piperidyl)-alpha-(hydroxymethyl)-3-(methoxymethyl)-methyl ester (CAS) (5.46%), and 2-N-butoxy-3,3,5,5-tetrachloro-2-methyl-6 trichloromethyl-4-pyridone (5.35%). The predominant compounds here were phenol and its derivatives. GC-MS analysis of the coconut shell liquid smoke redistillation result is shown in Figure 6 and Table 5. Further analysis of the second distillation stage at 145°C demonstrated two primary chemical compounds: phenol (CAS) izal (92.18%) and phenol 2-methoxy- (CAS) guaiacol (7.82%). The 155°C distillation produced comparable results with phenol (CAS) izal (87.17%) and phenol 2-methoxy- (CAS) guaiacol (12.83%). Notably, both products from the second distillation stage contained solely phenol compounds and derivatives, underscoring the increased purity achieved through redistillation.

This elevated purity, particularly in phenol compounds, contributes to the safety and versatility of coconut shell liquid smoke across various applications. Phenol compounds have demonstrated efficacy in inhibiting the growth of Staphylococcus aureus, E. coli, and Candida sp bacteria, making them valuable for food preservation and potential wound-healing applications [27-29]. Furthermore, these compounds hold promise as natural pesticides in agriculture, showcasing potential for plant pest control [30,31]. The systematic GC-MS analysis and resultant insights into compound compositions present a valuable foundation for optimizing the quality and applications of coconut shell liquid smoke.

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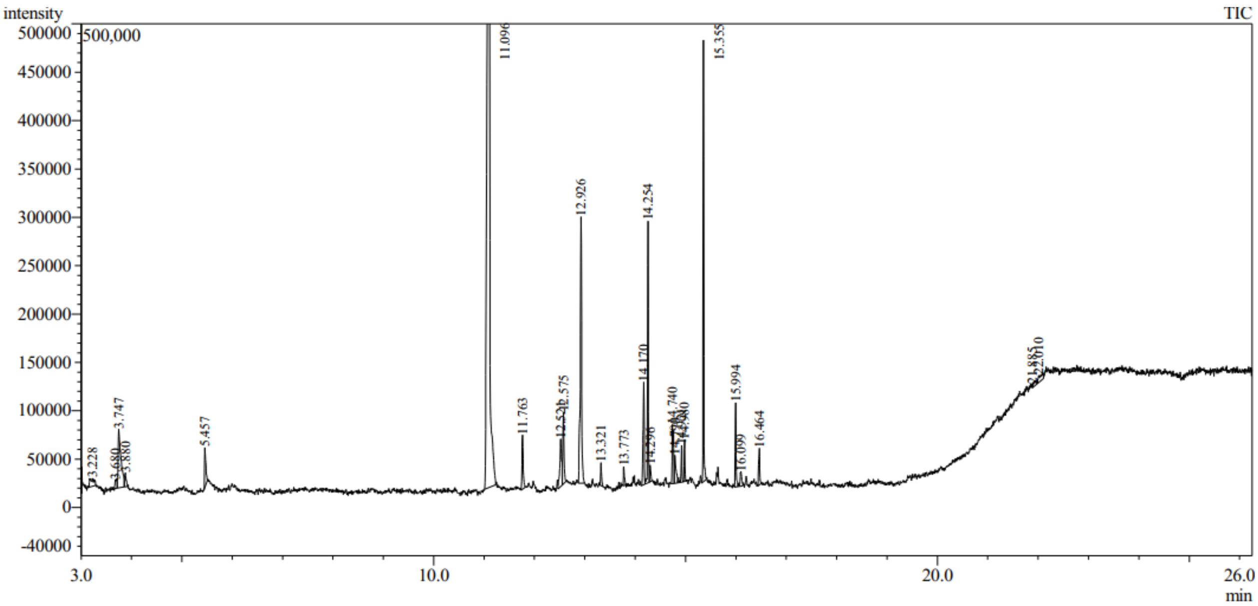
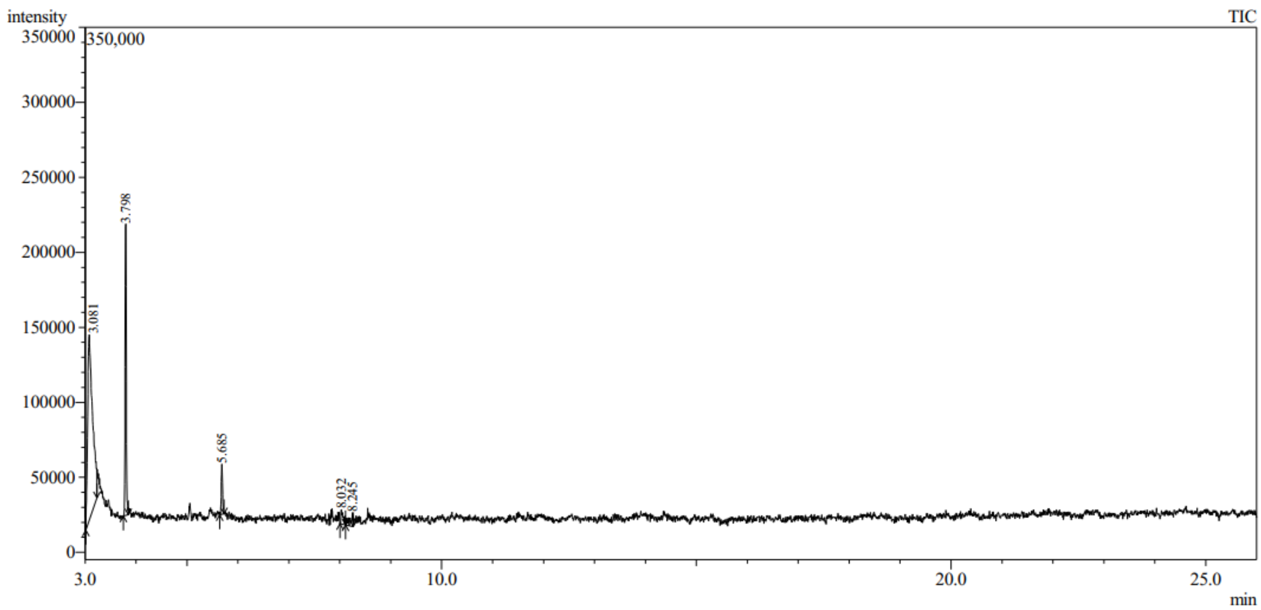


Fig. 4. GC-MS spectra of raw materials

Table 3
GC-MS analysis of coconut shell liquid smoke raw materials

Peak	% Area	Compound	Molecular Weight (grams/mole)
1	0.81	Heptanoic Acid (CAS)	130
2	0.45	2-Cyclopenten-1-One (CAS) Cyclopentenone	82
3	4.00	2-Furancarboxaldehyde (CAS) Furfural	96
4	0.49	Butanoic Acid, 2-Propenyl Ester (CAS) Allyl n-Butanoate	128
5	1.23	2(3H)-Furanone, dihydro- (CAS) Butyrolactone	86
6	53.11	Benzenesulfonic Acid, 4-Hydroxy- (CAS) Benzenesulfonic Acid, p-hydroxy	174
7	1.63	2-Cyclopenten-1-One, 2-Hydroxy-3-Methyl- (CAS) Corylon	112
8	2.12	Heptane (CAS) N-Heptane	100
9	1.95	Phenol, 2-Methyl- (CAS) o-Cresol	108
10	8.60	Phenol, 2-Methoxy- (CAS) Guaiacol	124
11	0.52	3-Ethyl-2-Hydroxy-2-Cyclopenten-1-One	126
12	0.47	1,5-Heptan-4-Ol, 3,3,6-Trimethyl- (Artemisiaalcohol)	154
13	2.69	2-Methoxy-4-Methylphenol	138
14	4.89	1,2-Benzenediol (Cas) Pyrocatechol	110
15	0.46	Cytidine, 2'-Deoxy- (CAS) Deoxycytidine	227
16	2.05	3-Methoxy-Pyrocatechol	140
17	1.02	1,4-Benzenediol (CAS) Hydroquinone	110
18	0.78	Phenol, 4-Ethyl-2-Methoxy- (CAS) p-Ethylguaiacol	152
19	0.77	4 Methyl Catechol	124
20	7.64	Phenol, 2,6-Dimethoxy- (CAS) 2,6-Dimethoxyphenol	154
21	1.74	2,5-Dimethoxybenzyl Alcohol	168
22	0.62	1,6-Anhydro-Beta-D-Glucopyranose (Levoglucosan)	162
23	0.97	1,2,3-Trimethoxy-5-Methyl-(CAS) Toluene	182
24	0.35	Benzene, 1,4-Bis (Trimethylsilyl)	222
25	0.63	Silicone grease	-



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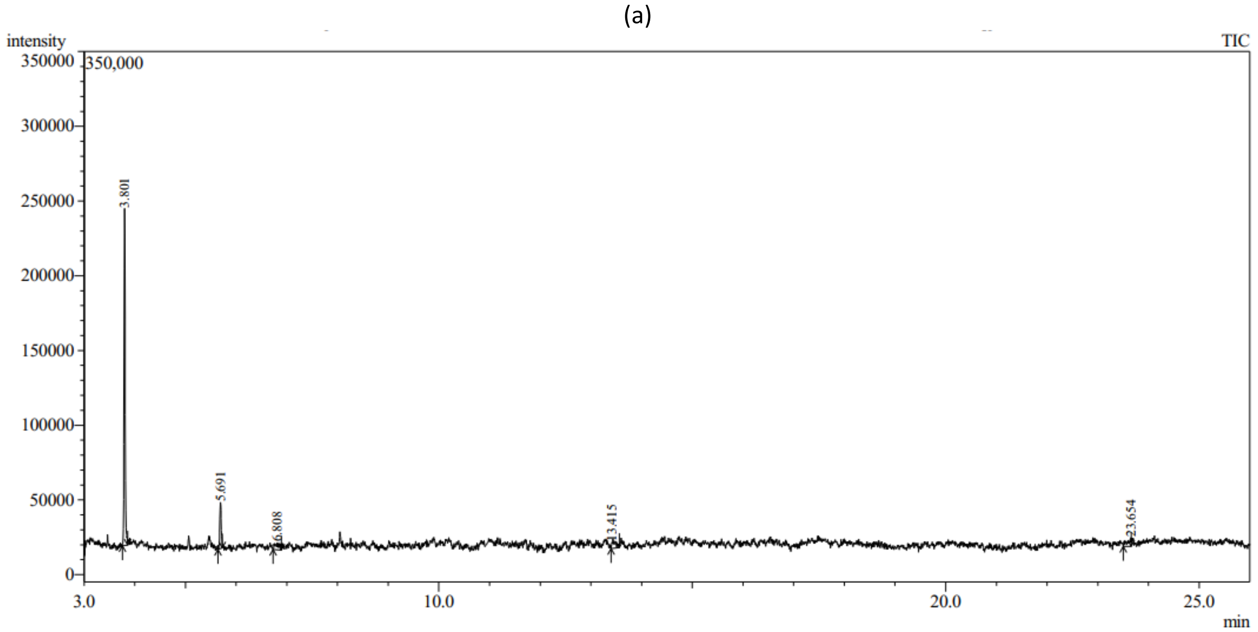
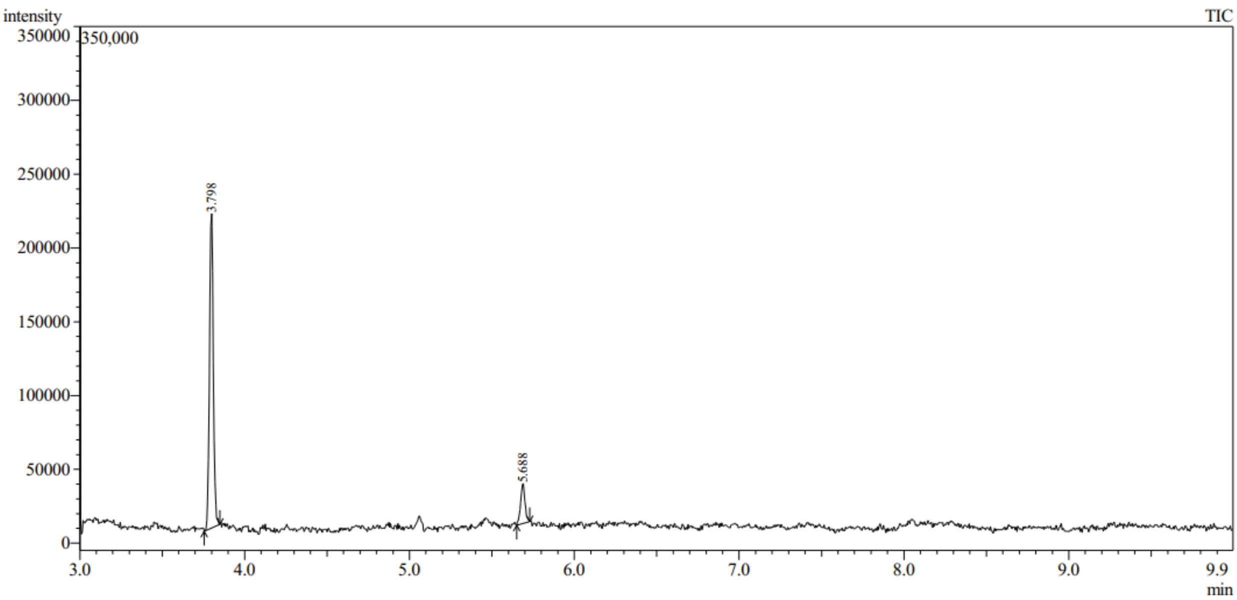


(b)

Fig. 5. GC-MS spectra of first distillation stage product (a) at 125°C (b) at 135°C

Table 4
GC-MS analysis of first distillation stage product

Temp. (°C)	Peak	% Area	Compound	Molecular Weight (grams/mole)
125	1	65.27	Ethyl Iso-Allocholate	436
	2	25.20	Phenol (CAS) Izal	94
	3	4.41	Phenol, 2-methoxy- (CAS) Guaiacol	124
	4	2.43	Phenol, 4-methoxy- 3 methyl- (CAS) 4-(2-Benzo[1,3]dioxol-4-yl-vinyl)	138
	5	2.70	8-Methoxy-12a-Methyl-1,2,3,3a,3b,4,5,6,7,8,9,10,10b,11,12,12a-Hexadecahydro-Benz	320
135	1	71.30	Phenol (CAS) Izal	94
	2	12.33	Phenol, 2-methoxy- (CAS) Guaiacol	124
	3	5.57	N-(Benzylidene)-2,2-dimethyl-1-(4-methyl) Phenylcyclopropylamine	263
	4	5.46	Indole-2-acetic acid, alpha-(1-acetyl-3-ethylidene-4-piperidyl)- alpha-(hydroxymethyl)-3-(methoxymethyl)-, methyl ester (CAS)	414
	5	5.35	2-N-Butoxy-3,3,5,5-Tetrachloro-2-Methyl-6-Trichloromethyl-4-Pyridone	435



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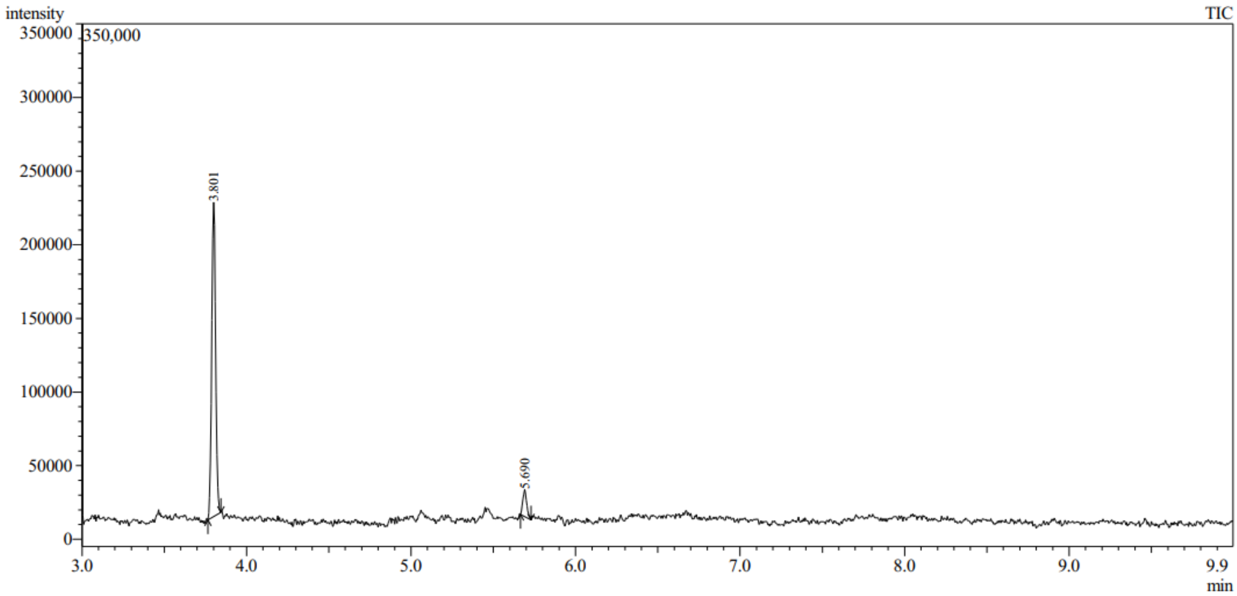

(b)

Fig. 6. GC-MS spectra of second distillation stage product (a) at 145°C (b) at 155°C

Table 5
GC-MS analysis of second distillation stage product

Temp. (°C)	Peak	% Area	Compound	Molecular Weight (grams/mole)
145	1	92.18	Phenol (CAS) Izal	94
	2	7.82	Phenol, 2-methoxy- (CAS) Guaiacol	124
155	1	87.17	Phenol (CAS) Izal	94
	2	12.83	Phenol, 2-methoxy- (CAS) Guaiacol	124

The comparison of phenol compound content, expressed as percent area (% area), across different stages of the distillation process reveals intriguing insights, as depicted in Figure 7. Initial examination demonstrates that the phenol compound constitutes 21.66% of the raw material in coconut shell liquid smoke. However, after the first distillation stage at temperatures of 125 and 135°C, this percentage increases to 32.04% and an impressive 83.63%, respectively. Remarkably, the second distillation stage, conducted at both 145 and 155°C, results in a 100% phenol compound content. These findings signify a marked augmentation in the phenol compound content within the distillation product in comparison to the initial raw material prior to distillation.

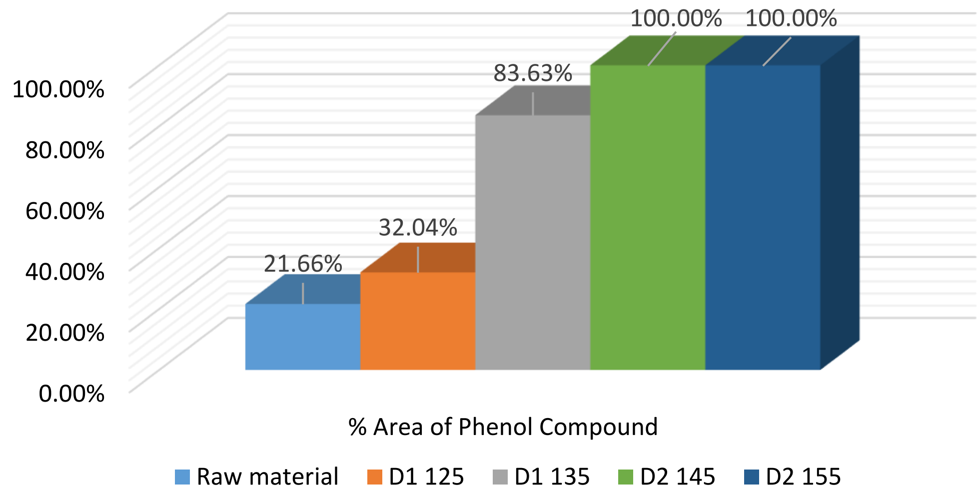

Fig. 7. Percent area of phenol compound content

Furthermore, the % area of phenol compound content in the second stage distillation outperforms that of the first distillation stage. This discrepancy underscores the potency of the redistillation process in enhancing the phenol compound concentration, a crucial aspect of the final product. Notably, this process yields even more remarkable results than the single distillation product combined with filtration, as observed in the research conducted by Putranto *et al.,* [16]. Their distillation-filtration method achieved a phenol content of 89.62%, while the distillation-redistillation method implemented in this study achieved an exceptional 100% phenol content. This comparison underscores the superiority of the distillation-redistillation approach in generating an enriched phenol compound content. In conclusion, the distillation-redistillation method proves to be a superior strategy in enhancing the phenol content of the final product, surpassing both the single distillation product and the distillation-filtration approach.

4. Conclusions

This study systematically investigated the influence of distillation and redistillation temperatures on the phenol compound content in coconut shell liquid smoke. The key findings and implications are as follows:

i. The distillation and subsequent redistillation processes play a pivotal role in enhancing the purity of the phenol compounds present in coconut shell liquid smoke, indicating the effectiveness of this purification method.
ii. The pH values observed during the distillation stages provide valuable insights into the chemical changes occurring within the liquid smoke. The recorded pH ranges of 1.18-1.30 and 1.11-1.39 in the first and second distillation stages, respectively, further validate the quality enhancement achieved through the distillation process.
iii. The first distillation stage, conducted at temperatures of 125 and 135°C, yielded distillates characterized by five dominant chemical compounds, primarily encompassing alcohol, phenols, and their derivatives.
iv. The second distillation stage, performed at temperatures of 145 and 155°C, yielded distillates containing two prominent chemical compounds—phenol and its derivative—highlighting the targeted separation and enrichment achieved through the redistillation process.

v. The resulting redistillation product holds substantial promise as a versatile raw material for various applications, including food preservation, antiseptic formulations, and insecticides, all of which can benefit from the enhanced phenol compound content.

In essence, this study not only provides a comprehensive understanding of the impact of distillation and redistillation temperatures on phenol compound enrichment but also highlights the potential practical applications of the obtained redistillation product. The findings underscore the importance of temperature optimization and the subsequent utilization of the redistillation process in enhancing the value and utility of coconut shell liquid smoke, further expanding its potential applications across diverse industries.

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