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Research Article

Optimization of pyrolytic oil production from coconut shells by microwave-assisted pyrolysis using activated carbon as a microwave absorber

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Abstract. Coconut shell waste pollutes the environment and affects public health. Converting coconut shell waste to pyrolytic oil (liquid smoke) with antimicrobial properties using microwave-assisted pyrolysis and activated charcoal as an absorbent is a promising solution. The purpose of this study is to investigate the process factors involved in the manufacture of coconut shell pyrolytic oil (liquid smoke) using microwave-assisted pyrolysis, to identify the chemical components in coconut shell pyrolytic oil, and to optimize the process factors using a face-centered central composite design (FCCD). This study further used coconut shells of various sizes (1–3 mm) and employed microwave-assisted pyrolysis with different power levels (300–600 W) and pyrolysis times (5–30 min). The results revealed that the pyrolytic oil (liquid smoke) yield increased as the time and microwave power increased but decreased as the size of the materials decreased. The optimum yield obtained was 34.6% at the following conditions: power of 593.6 W, material size of 2.9 mm, and heating time of 28.5 min. The analysis of the components of the volatile compounds in the pyrolytic oil (liquid smoke) product obtained from gas chromatography-mass spectrometry (GC–MS) analysis identified a total of 14 chemical components in coconut shell pyrolytic oil (liquid smoke) at 300 W, 15 compounds at 450 W, and only 5 components at 600 W. Among these compounds, phenol, dimethoxy phenol, guaiacol, hydroxyanisole, and methoxyphenol were found to have the highest concentrations. The outcomes of this study offer valuable contributions to the development of pyrolytic oil (liquid smoke) products with enhanced quality, flavor, and potential applications in the food industry.

Keywords: Activated charcoal, coconut shell, pyrolytic oil, microwave absorber, microwave-assisted pyrolysis.

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1. Introduction

The coconut plant plays a significant role in the Indonesian economy and is cultivated in various provinces throughout the country. Riau is the largest coconut producer, followed by North Sulawesi and East Java(Alouw & Wulandari, 2020). Coconut has many uses in Indonesia and is a primary source of income for 6.3 millions farmers who utilize all parts of the coconut for handicrafts, furniture, food, beverages, and bioenergy (Alouw & Wulandari, 2020). In 2000, Indonesia had a coconut plantation area of 3.7 million hectares, according to the Asian Pacific Coconut Community (Hasanah, Mulyati, & Heliawati, 2022). In 2019, Indonesia produced 17.13 million coconuts, making it the world's top producer (Ximenes, Nurmalina, & Rifin, 2021).

Coconut production results in waste, such as coconut shells, which increase as coconut production increases (Nuryana *et al.*, 2020). The shell (12%), meat (28%), husk (35%), and water (25%) are the four basic components of the coconut (Abidin, 2021). The abundance of coconut shell waste in Indonesia leads to environmental pollution and public health into commercial products reducing the environmental pollution. Coconut shells can be transformed into pyrolytic oil (liquid smoke) (Rizal *et al.*, 2022). Coconut shells are biomass derived from agricultural residues. In this case, various organic materials, including wood, grass, straw, corn, wood waste, and agricultural wastes, can be used as biomass feedstock (Jamilatun *et al.*, 2019).

Pyrolytic oil (liquid smoke) is also known as wood vinegar, pyroligneous acid or wood acid (Wibisono, Skripsa, Prabowo, & Boedi, 2022). Because of the abundance of coconut shells, liquid smoke production is an appealing industry in Indonesia. In this case, liquid smoke can function as an inhibitor of bacterial growth (Dewi, Tuhuteru, Aladin, & Yani, 2021). Furthermore, because of the presence of two predominant compounds that act as bacteriostatic phenols and organic acids, which possess the ability to control bacterial growth and act as antioxidants and antimicrobial agents (Pemanasari *et al.*, 2020).

Rapid biomass pyrolysis can maximize liquid production. Therefore, the conversion of biomass to liquid smoke is proposed. The total liquid output used to create conventional liquid smoke by burning is typically 35% of the weight of the raw materials as shown in Figure 1 (Xin, Dell, Udugama, Young, & Baroutian, 2021). This product distribution is greatly influenced by process parameters, including pyrolysis temperature and residence duration (Pestaño & Jose, 2016).

Commonly, liquid smoke is manufactured using a conventional pyrolysis reactor. However, the use of microwave

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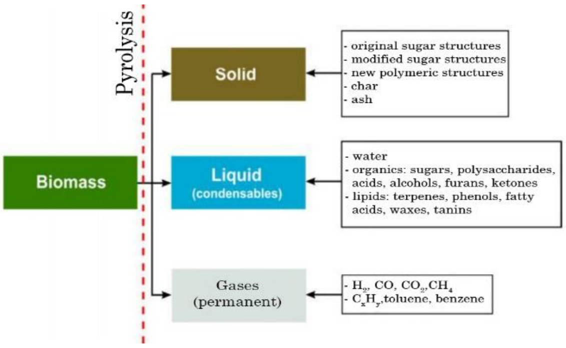

Fig. 1 Composition of the pyrolysis reaction products (Pestaño & Jose, 2016)

technology is a promising method of its production (Idris *et al.*, 2020). Microwave technology offers several advantages for biomass pyrolysis (Shi *et al.,* 2023). The use of microwave-assisted pyrolysis (MAP) technology allows fast, efficient, and selective energy transfer, precise heating, and easy control, all while being noncontact (Reddy, Shravani, Das, Dash, & R.Vinu, 2019). Electromagnetic waves with frequencies ranging from 300 MHz to 300 GHz are known as microwaves, which correspond to wavelengths ranging from 1 mm to 1 m under vacuum chamber conditions (Mumtaz, Rana, Choi, & Han, 2022). The structures of the reactor and biomass materials influence the microwave's speed and heat rate (Lin *et al.*, 2022). The material's molecules use microwave energy to heat it and transform electromagnetic radiation into thermal energy (Aziz, Yunus, Kania, & Hamid, 2021). Microwave technology can speed up the pyrolysis reaction process and can enhance the quality of the products with added value (Khelfa, Rodrigues, Koubaa, & Vorobiev, 2020). Microwave heating is faster than conventional heating because microwaves penetrate the material and simultaneously store energy to generate heat in every area of the material (Aziz *et al.*, 2021).

This is because the heat generation mechanism (dipole rotation and ionic conduction) is related to the influence of the dipole polarization mechanism and ionic conduction from microwave absorption, as shown in Figure 2. Molecules with a dipole moment will try to follow the electric field when exposed to microwave radiation. Because the electric field is wavy, the dipole will try to follow its movement constantly. In molecules with a charge, ions will move back and forth through the material and will collide with each other because of the electric field components of microwave radiation. This mechanism causes intermolecular collisions to get bigger so that the frequency factor is even greater and affects the activation energy value of the pyrolysis reaction (Foong *et al.*, 2023).

The feedstock is heated uniformly and selectively and is heated without direct contact due to the microwaves' penetration into the material, which results in vigorous dipole rotation and ionic conduction. This condition generates an outward-flow temperature gradient, which is more effective and results in a shorter processing time (Foong *et al.*, 2022).

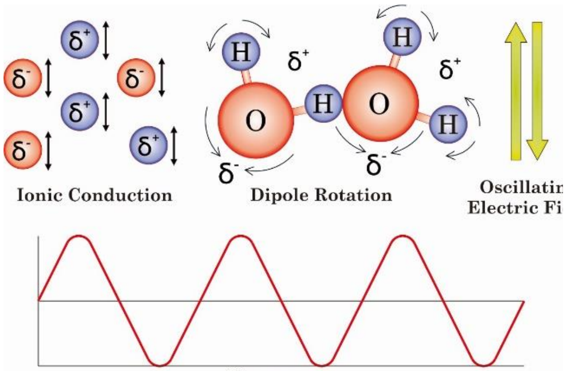

Fig 2. Mechanism of microwave heating

To speed up the pyrolysis process, a microwave absorber in the form of a carbon material is needed (Qadariyah, Aswie, Widjaya, Aditya, & Mahfud, 2021), and carbon materials are generally great absorbers of microwaves for the pyrolysis process (Gunwant & Vedrtnam, 2021). Microwave heating can potentially break down biomass molecules because of its high speed and heat efficiency through the microwave dielectric heat effect by utilizing microwave heat absorbents (Li, Li, & Shao, 2022).

Activated carbon and carbon are effective microwave absorbers and can efficiently convert microwave energy into heat, making them ideal for pyrolysis processes (Arnelli, Henrika Putri, Cholis, & Astuti, 2019). A microwave absorber has been developed to absorb energy through microwave irradiation to improve the heating rate (Torres-Moya *et al.*, 2022). This absorbent and raw material is added to the microwave, resulting in a stable pyrolysis temperature (Ethaib, Omar, Kamal, Biak, & Zubaidi, 2020). The combination of activated carbon and carbon is a superb absorber of microwaves that has a great capacity to absorb microwave radiation and to turn it into heat. Activated carbon is used to absorb microwaves and to generate heat. Activated carbon's good adsorption properties allow it to absorb microwave energy efficiently. When solid residues are immediately poured over the heated absorbent, the reactor temperature stabilizes, utilizing only a small quantity of energy from microwave radiation. This method enables the simultaneous heating of biomass using two heating methods: microwave conduction and irradiation, owing to the high absorbent temperature

Coconut shells are a hardwood type with a higher lignin content than other hardwoods. Specifically, it has a lignin composition of 33.30%, and cellulose and hemicellulose make up 30.58% and 26.70%, respectively. Meanwhile, the remaining components are water (8.86%) and ash (0.56%) (Nunes, Silva, Gerber, & Kalid, 2020). The high silica (SiO_2) content in coconut shells contributes to their hardness (Bheel, Mangi, & Meghwar, 2021). In the manufacture of coconut shell liquid smoke, specific pyrolysis process conditions can affect the content of various compounds. The maximum phenol content (7.32%) in liquid smoke made from coconut shells occurs at a 600°C pyrolysis process temperature and a 120-minute heating time. The highest content (92.30%) for acetic acid is obtained with a 120-min heating duration at a 500°C pyrolysis process temperature. By contrast, the highest carbonyl content (32.56%) is achieved with a 60-min heating duration at a 400°C pyrolysis process temperature (Maulina, Amalia, & Kamny, 2020).

Microwave-assisted pyrolysis technology is used to manufacture coconut shell liquid smoke, a novel research topic. This technology is expected to produce coconut shell liquid smoke more easily, augmented by the inclusion of a microwave absorber. Liquid smoke production, which serves as an alternative preservative, holds significant potential in advancing food technology due to its high economic value, wide availability, and ease of acquisition (Muhammad Faisal & Gani, 2018).

This research compared microwave-assisted pyrolysis methods and conventional processes that do not use microwaves. The study measured raw materials and examined key process factors, such as power, heating time, and material size, to produce liquid smoke from coconut shells. Response surface methods with a face-centered central composite design (FCCD) were also used to optimize the effect of these factors. To achieve these objectives, coconut shells of varying sizes (1–3 mm), different power levels (300–600 W), and different heating times (5–30 min) were used. Through this comparative analysis, the research aimed to identify the optimal conditions for generating liquid smoke from coconut shells using microwave-assisted pyrolysis. In addition, the study identified and analyzed the chemical components in the resulting coconut shell liquid smoke, enhancing our understanding of its composition and potential applications.

2. Materials and Methods

2.1 Materials

The main materials used in this research were coconut shells collected from East Java, Indonesia, specifically from Dampit District of Malang. These coconut shells served as the primary source material to produce liquid smoke. Coconut shells are categorized as hardwoods with high lignin levels and a composition of 33.30% lignin, 30.58% cellulose, 26.70% hemicellulose, 8.86% water, and 0.56% ash. In addition, activated carbon was utilized as a microwave absorber and was combined with the coconut shells in microwave-assisted pyrolysis. All other chemicals required for the research were obtained from the local market in Malang, Indonesia.

This ensured that the study utilized readily available materials and resources to conduct the experiments and analysis for producing liquid smoke from coconut shells using microwave-assisted pyrolysis. Using locally sourced materials, the research explored a practical and feasible approach to liquid smoke production that can be easily implemented in the region with readily available resources.

2.2 Experimental apparatus

The equipment used in the study was a microwave with the following specifications: maximum rated output power of 800 W, voltage of 220 V, rated input power of 1250 W, and magnetron frequency of 2450 MHz (12.45 GHz). In addition, the dimensions of the microwave were as follows: length = 48.5 cm, width = 37 cm, and height = 29.25 cm. Pyrex two-neck flasks of 500 mL (d = 9.85 cm) were utilized as a pyrolysis reactor for the microwave-assisted pyrolysis method. These flasks were equipped with a condenser as a coolant and a cooling trap to capture the light fraction of liquid smoke released during the process. The microwave used in the research for this pyrolysis process was a commercial microwave equipped with a thermocouple.

A thermocouple was also mounted on the microwave to monitor and adjust the temperature during the pyrolysis. A schematic of the experimental apparatus is shown in Figure 3.

2.3 Experimental Procedure

After reducing the coconut shells to sizes of 1, 2, and 3 mm, they underwent a sun-drying process. Then, 50 g of the dried coconut shell pieces was mixed with activated carbon, constituting 10% of the mixture's total weight. The resulting mixture was then subjected to microwave-assisted pyrolysis (MAP) at various power ranges (300, 450, and 600 W) and heating times (5, 15, 20, 25, 30, and 30 min). Three fractions were obtained during pyrolysis: solid charcoal, heavy tar, and light gas. Light gas passed through a condenser, where it was cooled and condensed to form liquid smoke. Condensed liquid smoke was then captured in a cooling trap, and any uncondensed methane gas was released. Solid charcoal remained inside the microwave after the pyrolysis, and heavy tar was collected and stored separately after passing through the condenser.

The experimental setup used for microwave-assisted pyrolysis to produce liquid smoke is shown in Figure 3. The setup consists of a two-neck flask placed inside a microwave, containing 50 g of coconut shells and activated carbon at a 10% ratio. Different durations and power levels were applied for heating during the pyrolysis process. As the mixture was heated, smoke was generated, and then directed through a condenser. The condenser facilitated cooling and condensation, resulting in the formation of liquid smoke. The condensed liquid smoke was collected in a cooling trap. Any residual smoke not collected in the cooling trap underwent further cooling and condensation in a second condenser, leading to tar formation. The tar produced during this process was collected and stored in a separate storage unit.

One crucial factor that assesses the process' quality is the percentage yield of liquid smoke. Yield was calculated as a percentage of the raw material used, with higher yields indicating a better quality process. The percentage yield of liquid smoke can be calculated using Equation (1) as follows (Dewi *et al.*, 2021):

$$Yield\ (\%) = \frac{weight\ of\ liquid\ smoke\ (gr)}{weight\ of\ raw\ material\ (gr)}\ x\ 100\%. \qquad (1)$$

2.4 Analysis of the chemical components of liquid smoke

Gas chromatography and mass spectroscopy were used to identify the chemical constituents of liquid smoke. The sample preparation procedure began with extracting coarse liquid smoke and distilling it with ether. The separated upper fraction (ether) was then collected, and the extraction process was repeated with the lower fraction. The resulting upper fractions were combined and concentrated by blowing nitrogen gas until the remaining volume was approximately 1 mL. The analysis was performed using Shimadzu's GC–MS-QP2010S under the following operating conditions: electron impact ionizing type 70 eV, injector temperature of 290°C, detector temperature of 280°C, Rtx-5MS column type with a length of 30 m, column

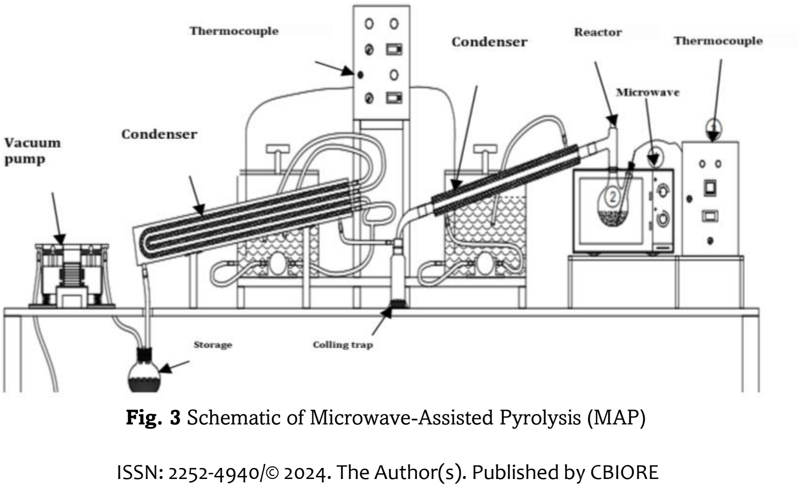

Fig. 3 Schematic of Microwave-Assisted Pyrolysis (MAP)

Table 1
Range and level of independent variables

Variables	Symbol coded	Range and levels		
		-1	0	1
Power (W)	X_1	300	450	600
Size (mm)	X_2	1	2	3
Time (min)	X_3	10	20	30

temperature ranging from 70°C to 230°C with a temperature increase of 5°C per min, and helium carrier gas with a flow rate of 60 mL/min at a pressure of 13.7 kPa.

2.5 Optimization using response surface methodology

Three operating variables were included in the optimization process utilizing response surface methodology (RSM), including raw material sizes (1–3 mm), heating times (10–30 min), and microwave power levels (300–600 W). The optimum point was determined using Design Expert Program version 13 (State-Ease Inc., Minneapolis, MN, USA), commonly called RSM. Because it involved two free variables, the face-centered central composite design (FCCD) was used, resulting in 20 experimental units. In this study, power (X_1), size (X_2), and time (X_3) were identified as the three important variables that affect the liquid smoke yield through pyrolysis. The RSM design was utilized to optimize the yield of liquid smoke from coconut shells, considering three factors, three levels of code, and three levels of power codes, including 300, 450, and 600 W power; sizes of 1, 2, and 3 mm; and times of 10, 20, and 30 min, as presented in Table 1.

The response function was estimated using regression analysis as a first-order polynomial, which can be written as shown in Equation (2)

:

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_{iX_i} + \sum_{i=1}^{k} \beta_{iiX_i^2} + \sum_{i-1}^{k-1} \sum_{j=2}^{k} \beta_{ijX_iX_j} + \varepsilon \qquad (2)$$

where Y denotes the expected response; β_0 denotes the constant regression coefficients; β_i, β_{ii}, and β_{ij} are the coefficients of linear, quadratic, and interaction effects; the variable codes are denoted by x_i and x_j; and ε is the error (Oramahi, Kustiati, & Wardoyo, 2022).

3. Results and Discussion

3.1 Thermogravimetric Analysis (TGA) of coconut shells

The behavior of the composite materials to heat exposure during chemical processes was observed in thermal decomposition. Thermal decomposition is a chemical reaction in which a single compound splits into two or more simple compounds when energized in the form of heat.

The thermal degradation was calculated using the thermogravimetric analysis behavior of coconut shells, and the results are presented in Figure 4. as a percent of the remaining weight (TGA curve) i.e., measures the amount and rate of change in the weight of the material concerning temperature or time during heating or cooling and differential scanning calorimetry (DSC) curve i.e., measures the heat absorbed or liberated during heating or cooling.

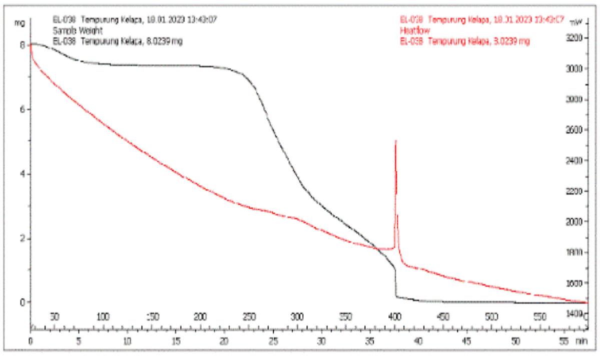

Fig. 4 TGA and DSC analyses of coconut shells. TGA/DSC curve (TGA—black, DSC—red)

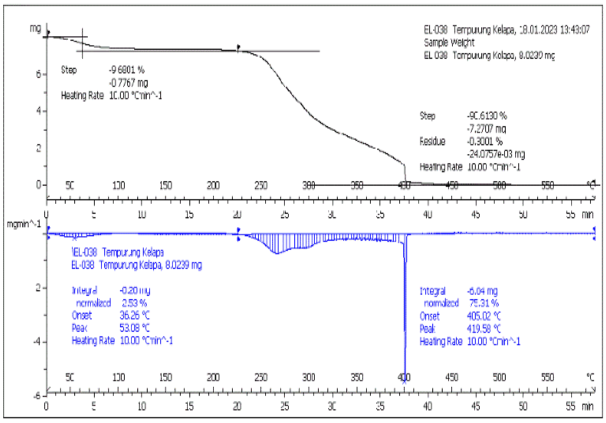

Fig. 5 TGA curve (up) and DTG curve (bottom).

3.2 Thermal analysis/Thermogram interpretation

The thermogravimetric analysis/differential scanning calorimetry (TGA/DSC) curves of the coconut shell sample, which provide information regarding the sample's thermal behavior, are shown in Figure 4, and the TGA thermogram and its first derivative (DTG) offering more insights into the coconut shell's thermal properties are shown in Figure 5. The analysis was conducted using an 8.0239 mg sample of the coconut shell, and the thermal analysis was performed under inert air circumstances with a consistent heating rate of 10°C/min in temperatures ranging from 0°C to 600°C.

The initial weight loss was observed at a temperature below 100°C. This weight loss was attributed to the moisture content loss due to water evaporation and the removal of light volatiles, resulting in a weight loss of approximately 9.68%. When the sample was heated up to a temperature close to 200°C, no significant changes in weight were observed. However, a slight change in the decomposition rate, represented by a flat curve, was identified in Figure 5. It is reported that lignin slowly decomposes over a broader temperature range (140°C–600°C) (Stefanidisa *et al.,* 2014). Hence, although not prominent, the beginning of lignin decomposition could contribute to the peak occurrence within low temperatures of 150°C–200°C. Conversely, cellulose could decompose within a slightly narrower temperature range between 250°C and 400°C.

Furthermore, the TGA/DTG plots represent overlapping peaks at approximately 200°C–420°C, indicating that rapid degradation of biomass components had occurred (90.61% weight loss), as shown in Figure 5. The peaks correspond to the degradation of lignocellulosic components, such as hemicellulose, cellulose, and lignin. Owing to its amorphous structure and low activation energy, hemicellulose could decompose at lower temperatures, over a wider temperature range between 190°C and 380°C (Escalante *et al.*, 2022). At the same time, cellulose could decompose in a slightly narrow temperature range between 250°C and 400°C (Tangsathitkulchai, Junpirom, & Katesa, 2016). Thus, it could be inferred that the appearance of the overlapping peaks of the TGA/DTG curves was mostly related to the breakdown of the cellulose and hemicellulose components. The maximum decomposition rates were noted to occur at 419.58°C are shown in Figure 5. This finding is supported by the presence of a sharp

Table 2
Comparison of percent yield using the method without microwave and the method using microwave

Method	Yield	Efficiency	Researcher	Year
Conventional	23.6 % (360 min)	6.5%	R. D. Ratnani, et al	2022
Conventional	17.5 % (29 min)	60.3%	Robert Silaban, et al	2022
Conventional	23.6 % (165 min)	14.3%	Baiti Hidayati, et al	2022
Microwave	34.40% (30 min)	114.6%	SPA Anggraini, et al	2023

exothermic peak in the DSC curve at approximately 400°C, as shown in Figure 4. Moreover, in the TGA/DTG curves' flat tailing region, a higher temperature was noticeable because of the slow rate of lignin decomposition across a huge temperature range.

Considering the thermogravimetric analysis (TGA) curve in Figure 4. Thermal degradation of coconut shell liquid smoke occurs in four stages. The initial weight loss was observed at 68°C within 3.8 min, i.e., 0.5 mg, approximately 6.25% of the weight loss during the process. This was due to the loss of moisture content during water evaporation. In the second stage, weight loss occurred at a temperature of 240°C within 20 min, which was 0.7 mg, approximately 8.75%. In the third stage, there was weight loss at 400°C for 37.5 min, which was 7.7 mg, approximately 96.25%. In the fourth stage, weight loss occurred at a temperature of 512°C within 48.8 min, which was 8 mg, approximately 10%. Many compounds with a high molecular weight have greater cracking energies above 500°C, which cause them to break down and disappear.

The maximum weight loss was 87.5%, as observed in the temperature range between 240°C and 400°C. At this stage, more devolatilization of coconut shells occurred, namely, acetic acid, acetaldehyde, carbonyl, phenol, and phenolic esters. Low-molecular-weight chemicals, like decomposition products, are lost at such temperatures. Differential scanning calorimetry (DSC) determines the variation in the heat needed to increase the reference and sample temperatures as a function of temperature. The devolatilization of the coconut shells as shown in the DSC curve appeared to have one peak pattern at 400°C with a heat flow of 2520 mW, with the peak type of the DSC curve being exothermic peak-release heat.

At 37.5 min and a temperature of 402.5°C, there was a decrease in peak to heat loss of 1800 mW. Then, there was a second decrease until heat loss of 1480 mW. At 400°C is the maximum decomposition rate, i.e., cellulose and lignin decomposition. It degraded at the peak into acetic acid, carbonyl, and phenol compounds. The third stage's climax occurred when cellulose and lignin decomposed at temperatures between 240°C and 400°C. It started with water evaporation at a temperature of 100°C. Furthermore, the first devolatilization occurred at temperatures from 100°C to 240°C. In this process, hemicellulose decomposition occurred. The second devolatilization occurred at temperatures from 240°C to 400°C, and cellulose and lignin decomposition also occurred. Lignin decomposition occurred in the third devolatilization, from 400°C to 512°C. Thus, it can be concluded that the peak appearance on the DSC curve was primarily due to cellulose and lignin decomposition components, giving a weight loss of approximately 96.25%.

The analysis temperature TGA stopped at 600°C, as shown in Figure 4. This was because the main components in the coconut shell were depleted or decomposed completely at that temperature. In the TGA analysis process, the material was subjected to gradual heating and the sample weight was monitored during the heating process. The sample weight decreased with the decomposition or reduction of the components contained in the sample.

TGA temperature analysis was intended to understand the point at which most of the organic components in a sample have decomposed or burned out. The temperature reflects the point at which the organic components in the coconut shell have been depleted or completely decomposed, leaving behind a carbon residue.

3.3 Comparison yield of conventional and microwave-assisted pyrolysis methods in producing liquid smoke

The comparison of yield obtained from conventional, and microwave-assisted pyrolysis methods in producing liquid smoke is shown in Table 2.

The difference in percent yield using a microwave resulted in a higher yield percentage of 34.40% within 30 min with efficiency of 114.6%, as shown in Table 2. For those without a microwave, R.D. Ratnani stated that liquid smoke produced a percent yield of 23.6% for 360 min with efficiency of 6.5% (Ratnani, Hadiyanto, Widiyanto, & Adhi, 2022). At the same time, Robert Silaban reported that using conventional methods produced a liquid smoke yield of 17.5% for 29 min with efficiency of 60.3% (Silaban, Lubis, Siregar, Sinaga, & N.P, 2022). Baiti Hidayati also explained that liquid smoke produced by conventional means was 23.6% for 165 min with efficiency of 14.3% (Hidayati, Sipahutar, Bizzy, & Faizal, 2022).

The use of microwave resulted in greater efficiency of 114.6%. This was due to the transfer of heat into the material and the interaction of electromagnetic field molecules. Volumetric heating treatment of materials using microwaves has advantages of significant energy savings, fast times, and improved production processes and is environmentally friendly (Paunescu, Axinte, Dragoescu, & Cosmulescu, 2020). In making liquid smoke by conventional pyrolysis, organic raw materials are heated under anaerobic conditions (without air) at high temperatures to produce liquid smoke. This process involves the thermal decomposition of raw materials into simpler products, including liquid smoke. However, in this process, several obstacles can cause lower liquid smoke yields.

One of the main constraints of conventional pyrolysis is inefficient heat transfer. The heating of organic raw materials in conventional pyrolysis is often uneven, so only a small part of the raw materials is involved in the pyrolysis reaction to produce liquid smoke. Uneven and inefficiently controlled temperatures can also lead to side reactions resulting in unwanted products or even further thermal decomposition, reducing the yield of liquid fumes.

In conventional pyrolysis, heat is supplied to the raw material externally through direct heating with a heat source such as gas, furnace, or other heating elements. This heat is then gradually conducted from the hot surface into the raw material. The convection process also occurs when hot air or gas moves around the raw material and takes its heat, aiding in heat transfer. Heat radiation can also occur between hot surfaces and raw materials. Because of the use of microwaves in the pyrolysis process, efficient dielectric heating occurs. Microwaves heat organic raw materials quickly and evenly by heating their water molecules. This allows more homogeneous and better-controlled temperatures, increasing the yield of liquid smoke. In addition, microwaves can reduce the reaction time required in the pyrolysis process, improving the efficiency and yield of liquid smoke.

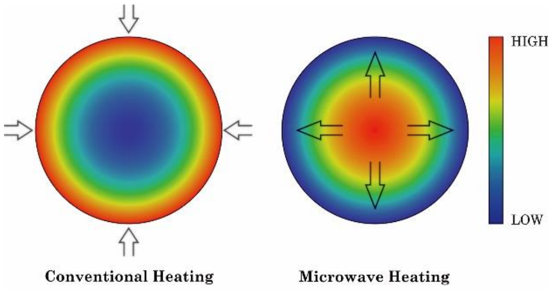

Fig. 6 Microwave induced and conventional heating patterns

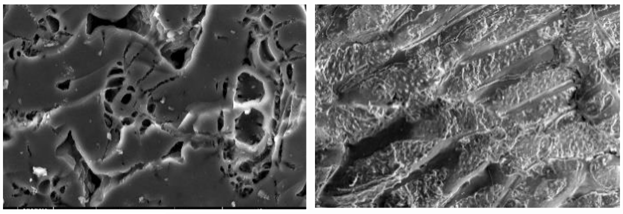
(a) (b)

Fig. 7 Activated carbon SEM test results (a) before heating and (b) at a temperature of 600°C

In microwave use, heat is generated internally in the raw material through dielectric heating. Microwaves emit electromagnetic waves with a certain frequency absorbed by water molecules in organic raw materials. In this case, microwaves polarize the water molecules and move with each other producing thermal energy, which makes the raw material heat quickly and evenly.

The main advantages of microwaves are their high heating efficiency and ability to heat raw materials uniformly. Heat is generated directly inside the feedstock, reducing the time required for heat transfer from external sources into the feedstock. In addition, microwaves can heat raw materials more homogeneously, reducing the temperature unevenness that can affect the pyrolysis reaction.

How microwaves enable internal dielectric heating, leading to quick and uniform heat distribution in organic raw materials, as shown in Figure 6. This efficient and homogeneous heat transfer allows better temperature control, resulting in increased liquid smoke yield compared to conventional methods, where heat is externally supplied and needs to be conducted to the feedstock.

Applying microwaves in the pyrolysis process ensures good and optimal performance in the reactor's evaporation process. Microwave-radiation-based heating is highly efficient, leading to significant energy savings. Compared to conventional heating approaches, microwaves require less energy input to heat the raw materials. By contrast, the conventional pyrolysis process consumes approximately 15% of the energy in the biomass feed (Zhang, Lei, Chen, & Wu, 2016).

According to Yin, it takes biomass many minutes to reach the reaction temperature during microwave-assisted pyrolysis (MAP) (Borges *et al.*, 2014). In fact, the heating rate may be boosted to decrease the reaction time to produce a higher and higher-quality yield of liquid smoke. Indeed, the addition of activated carbon in the microwave-assisted pyrolysis process offers significant benefits, enhancing both the efficiency and quality of the pyrolysis process. Activated carbon is known for its high absorption properties against microwaves. When added to organic raw materials, activated carbon acts as a microwave absorber, efficiently absorbing the microwave energy emitted by the microwave source.

This microwave absorption carried out by activated carbon leads to the generation of heat, which promotes the rapid and uniform heating of the organic raw materials. The ability of activated carbon to absorb microwave energy effectively allows precise and controlled heating, contributing to the optimization of the pyrolysis process. The inclusion of activated carbon as a microwave absorber contributes to improved heat transfer and temperature control during the pyrolysis process, leading to the enhanced yield and quality of the resulting liquid smoke. Another research project provides evidence of the positive impact of activated carbon in microwave-assisted pyrolysis, supporting its efficacy as an effective method for liquid smoke production from organic materials (Shi et al., 2023).

In pyrolysis, activated carbon is a catalyst (Ohra-aho, Lindfors, Lehtonen, Tamminen, & Siipola, 2020). As a catalyst, activated carbon helps in increasing the speed of the pyrolysis reaction without undergoing significant chemical changes. This can reduce the energy required and can speed up the decomposition of organic matter into liquid smoke. Activated carbon has a very large and complex pore structure, which results in a large surface area (Kumar & Jena, 2016). This can be seen in Figure 7.

The SEM results of activated carbon with a magnification of 2500× in Figure 7 show that the heating temperature of the reactor will open many pores in activated carbon. At a temperature of 600°C, there will be more open pores with a diameter of approximately 10 μm compared to when there will be no heating (pores with a diameter of approximately 50 μm).

With these pores, activated carbon is very suitable for use as an absorber because when radar electromagnetic waves hit activated carbon, the waves will not be reflected but will be deflected into the pores and then will be reflected into deeper pores so that the waves will damage and run out of energy. Therefore, broadly porous activated carbon can increase the absorption of electromagnetic wave radiation with low surface reflection, and heat is also better than the absorbed energy due to the fraction of high air volume or the mechanism of absorption of multiple reflection radiation.

An illustration of electromagnetic waves trapped inside the pores of activated carbon is shown in Figure 8. The more open the pores will be, the more the conductivity value will increase. With the increase in conductivity, the absorption of electromagnetic wave radiation will increase. This structure allows activated carbon to absorb materials. The material is given a microwave absorber to hasten the heating procedure during pyrolysis (Mushtaq, Abdullah, Mat, & Ani, 2015). Without the addition of activated charcoal to the coconut shell, pyrolysis on the coconut shell can still produce liquid smoke. Therefore, without activated charcoal, the result of the coconut shell pyrolysis process may produce liquid smoke, but its purity or chemical composition is not as good as when using activated charcoal.

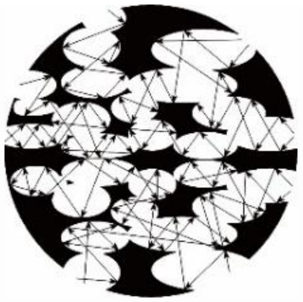

Fig.8 Illustration scheme of the absorption of electromagnetic wave radiation

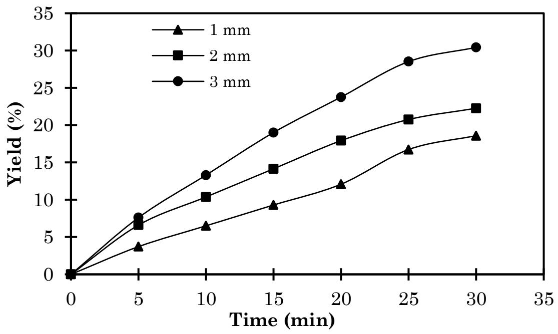

Fig. 9 Effect of heating time on % yield with various sizes of raw materials using microwave-assisted pyrolysis at 450 W power

3.4. Microwave-assisted pyrolysis technique with an activated charcoal absorber in opposition to heating time

Using coconut shells of various diameters (1, 2, and 3 mm) and activated charcoal as the absorbent, the pyrolysis process was carried out at several power levels, namely, 300, 450, and 600 W, to explore the impact of microwave power on the yield of liquid smoke. The range of the heating period was 5–30 min. The obtained liquid smoke yields at different power levels are presented in Figure 9.

The yield of liquid smoke increased as the heating times increased, as shown in Figure 9. Among the raw material sizes tested, 3 mm produced the highest yield of liquid smoke, followed by 2 mm and then 1 mm. This was because microwaves with a power of 450 W provided a stronger heat shot to decompose organic compounds, resulting in more smoke and a higher yield of liquid smoke. By contrast, liquid smoke production at 1 mm was the lowest because a microwave power of 450 W was very strong for smaller particle sizes. Thus, there was low efficiency in the liquid smoke production process. A power of 450 W was very high for particles of 1 mm size because excessive heating can damage particles and can change their chemical composition. Very high power leads to the decomposition of particles into unwanted or inefficient products. Particle size of 1 mm receiving high microwave energy at high microwave power in a short period, thus resulting in a structural damage. This is what causes low efficiency in the formation of liquid smoke. Very high power causes some energy to be wasted in the form of unnecessary heat or energy not used efficiently in liquid smoke production.

The 3 mm material size produced the highest liquid smoke after 30 min of heating. This was because the prolonged heating time increased the temperature, causing more smoke to be produced, which could then condense into liquid smoke. Microwave power affects the microwave's ability to penetrate and heat materials evenly. When the raw material is heated in a microwave, the microwave energy will be absorbed by the material and converted into thermal energy, which, in turn, causes heating of the material. In larger raw materials, a large amount of wave microenergy will be absorbed on the external surface and will then propagate slowly to the inside of the material. Owing to the larger size, this energy has more distance to propagate before reaching the center of the material.

In this case, the larger the raw material is, the more even is the heat distribution as the heat propagates into the material over a longer period. As a result, the temperature of the material at various points within the material becomes more uniform and the heating process becomes more effective. This impact may increase the yield of produced liquid smoke. Conversely, in smaller feedstock, wave microenergy can penetrate and spread rapidly throughout the material, resulting in a more uneven heat distribution. Some parts of the material get hotter than others, which can lead to imperfections in heating and lower results in the formation of liquid smoke.

The sizes of the feed particles affect the generation of liquid smoke. Temperature, reaction time, heating rate, raw material type, and feed particle size are only a few variables affecting the pyrolysis process (Ridhuan, Irawan, & Setiawan, 2020). Depending on the proportion of cellulose and lignin in the wood material, "pyrolysis oil" or "bio-oil" can be produced with yields of up to 70%–80% weight (Lyu, Wu, & Zhang, 2015). At 600 W power, it produces a higher % yield compared to 300 and 450 W, as shown in Figure 10. A higher microwave power will provide a higher thermal energy in less time. This will increase the heating speed of the raw material in the pyrolysis process, thus achieving the optimal reaction temperature faster.

The larger wave microwave power of 600 W can also help in solving the problem of uneven heating in the pyrolysis process. Even heating will ensure that all raw materials reach the required temperature for the pyrolysis reaction more consistently, thereby increasing the yield of liquid smoke. The higher the micropower of the waves is, the higher is intensity of the electromagnetic energy. This intensity can stimulate more molecules in the raw material to move and interact with each other, increasing the rate of the pyrolysis reactions. The higher micropower of the waves provides a higher intensity of electromagnetic energy. This intensity can stimulate more molecules in the raw material to move and interact with each other, increasing the rate of the pyrolysis reactions.

The smaller microwave power of 300 W provides lower thermal energy. This means that the raw material will take longer to reach the optimal reaction temperature. A slower heating process can cause the pyrolysis reaction to be less efficient, so the yield of the produced liquid smoke is also lower. The waves' low micropower does not reach the temperature required for the pyrolysis reaction.

Lower reaction temperatures can result in less liquid smoke product formation. A lower microwave power can also cause uneven heating problems in raw materials. Uneven heating can inhibit pyrolysis reactions and can cause certain parts of the feedstock not to degrade properly, reducing the yield of liquid smoke. A lower microwave power provides lower-intensity electromagnetic energy. A low intensity can reduce the number of molecules stimulated to interact and respond to the pyrolysis process, thus inhibiting the formation of liquid smoke products.

3.5 GC–MS characterization of liquid smoke

The chromatograms presented illustrate the chemical constituents of liquid smoke obtained from coconut shells using

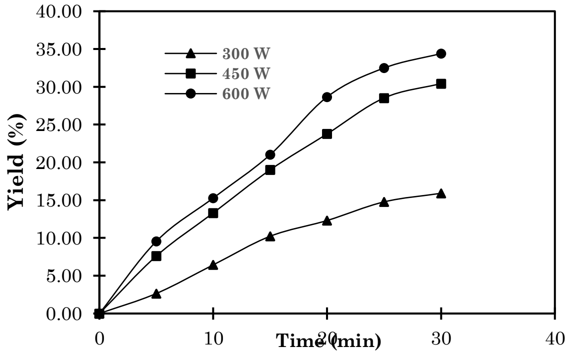

Fig. 10 Effect of heating time on % yield with various power values using microwave-assisted pyrolysis using a raw material with a size of 3 mm

Table 3
GC-MS analysis of liquid smoke products

Chemical content	Area (%)		
	300 W	450 W	600 W
1,2-cyclopentanedione, 3-methyl, 2-Hydroxy.	7.53		
Phenol, 2-methyl.	3.37		
Phenol, 4-methyl, oxytloluene.	6.04		
Phenol; 2-methoxy; guaiacol; methoxyphenol.	14.70		
2-methoxy-4- methoxyphenol; phenol; methylguaiacol; benzene	3.69		
1,2-benzenediol.	20.02		
1,4-dimethoxy-2-methyl; toluene; 1,4-dimethoxy-2-methylbenzene.	3.16		
1,2-benzenediol; 4-methyl-3, 4-dihydroxytoluene.	3.80		
Phenol, 2,6-dimethoxy-2, 6- dimethoxyphenol.	25.07		
Benzaldehyde, 4-hydroxy-3-methoxy.	2.06		
Benzene; 1,2,3-trimethoxy; methylsyringol.	4.68		
Benzene; 1,2,3-trimethoxy-5-methyl-toluene.	2.24		
2-propanone; 1,4-hydroxy-3- methoxyphenyl.	2.31		
2,4-hexadienedioic acid; 3,4-diethyl, dimethyl ester.	1.33		
Acetic acid; 3-methylphenyl ester; benzene.		4.88	
Phenol, 2-methoxy; guaiacol; methoxyphenol.		30.67	
4-hexen-3-one; 4,5-dimethyl.		6.89	
2-methoxy-4-methylphenol; phenol; 2-methoxy.		5.36	
1,2-benzenediol pyrocatechol; benzenediol.		4.19	
2-pentanone; methyl propyl ketone; ethylacetone; acetate.		2.55	
3-methoxy-pyrocatechol; 1,4-benzenediol.		1.09	
Phenol, 4-ethyl-2-methoxy; ethylguaiacol.		4.05	
3-acetyl-methyl.		2.09	
Phenol; 2,6-dimethoxy; syringol 1,3, phenol.benzaldehyde.		21.01	
Benzaldehyde, 4-hydroxy-3methoxy.		1.25	
Benzene; 1,2,3-trimethoxy; methylsyringol; 2,5-dimethoxy; methanol.		5.05	
Benzene; 1,2,3-trimethoxy-5-methyl; toluene; 2,5-dimethoxy; phenol.		3.87	
2-propanone; 1-4-hydroxy-3-methoxyphenyl.		4.05	
2,4-hexadienedioic acid, 3,4-diethyl-, dimethyl ester.		3.01	
2-cyclopenten-1-one; 2-hydroxy-3-methyl.			5.50
Phenol, 2-methoxy; guaiacol; methoxyphenol.			64.45
2-methoxy-4-methylphenol; phenol; 2-methoxy.			6.80
Phenol; 4-ethyl-2-methoxy; benzene; toluene.			4.43
Phenol; 2,6-dimethoxy.			18.82

the microwave-assisted pyrolysis method (shown in Table 3). The relative percentage of regions under the chromatographic peaks was calculated to perform a semiquantitative analysis.

The relative percentage of the area under each chromatographic peak was calculated, providing valuable insights into the main chemical components present in the liquid smoke. The higher the percentage of each area is, the greater is the content of the corresponding chemical in the liquid smoke.

The major chemical components of the liquid smoke made from coconut shell, with the corresponding percentages of regions, are shown in Table 3. Upon conducting GC-MS analysis tests, the results for liquid smoke components derived from the microwave-assisted pyrolysis method at different power levels: 300 W, 450 W, and 600 W, respectively, as shown in Table 3. Each table provides a breakdown of the identified compounds and their corresponding percentages, thereby revealing how the chemical composition of the liquid smoke varies with the applied power during the pyrolysis process. These comprehensive analyses offer essential information regarding the chemical makeup of coconut shell liquid smoke and the influence of microwave power levels. This knowledge is crucial for understanding the factors that contribute to the distinct flavor and aroma profiles of liquid smoke and can aid in optimizing the production process to achieve the desired characteristics in the final product.

The comprehensive GC–MS analysis provided valuable insights into the intricate chemical makeup of coconut shell liquid smoke, as shown in Table 3. At 300 W power, the analysis revealed the presence of 14 distinct components in the liquid smoke. Notably, phenol and dimethoxy phenol had the highest concentrations, indicating their significance in the overall composition. Furthermore, 450 W showed a more complex chemical profile, with 15 identified compounds. Phenol, guaiacol, and methoxyphenol emerged. Phenol, guaiacol, methoxyphenol, and hydroxyanisole emerged as the predominant components at this power level, contributing significantly to the intense aroma and flavor of the liquid smoke. At a power of 600 W, a more straightforward composition consisting of five compounds in the liquid smoke of a coconut shell was observed. Among them, phenol, 2-methoxy, guaiacol, and methoxyphenol were the most abundant components. These findings demonstrate the impact of power levels on the chemical composition of the liquid smoke derived from coconut shells.

The varying concentrations of specific compounds at each power level result in distinct sensory attributes in the liquid smoke. Understanding these nuances is crucial for optimizing the production processes of liquid smoke and harnessing the desired flavor and aroma profiles from coconut shells. The research sheds light on the importance of power control during coconut shell liquid smoke production, allowing manufacturers to tailor the chemical makeup to meet specific preferences and applications. Ultimately, this knowledge can lead to the development of high-quality liquid smoke products with consistent and desirable characteristics.

The use of different microwave powers during liquid smoke production from coconut shells can significantly affect the chemical composition and the number of compound components present. When lower power levels are employed, slower heating occurs, allowing complex compounds to decompose or undergo various chemical reactions over a longer duration. This can form a greater variety of compounds in liquid smoke. The structural integrity of complex compounds is better maintained at lower temperatures, preventing their decomposition into simpler components.

Conversely, a higher microwave power leads to faster and more intense heating, increasing the risk of thermal decomposition for compounds exposed to high temperatures. This can result in the loss or alteration of certain compounds, generating unwanted byproducts. In addition, rapid heating may not allow sufficient time for complete molecular breakdown or equilibrium of chemical reactions, potentially reducing the number of compound components formed.

The increased power can also elevate the evaporation rate of more volatile compounds in the sample. Consequently, these highly volatile compounds may vaporize before they can be properly detected or separated in the analysis process, leading to fewer detected and measured compound components in liquid smoke. Overall, the GC–MS analysis provides useful information regarding the chemical constituents present in the liquid smoke derived from coconut shells, which can have important implications for its usage in the food industry.

3.6 *Statistical analysis model fitting*

The optimization process used the face-centered central composite design technique based on response surface methodology (RSM). A comparison of the 20 experimental data with the data obtained from the model is shown in Table 4. The % yield response values ranged from 6.50809% to 30.4262%. The ANOVA results indicate that the model chosen for the yield response is a 2Fl model, as shown in Table 5. The significance of the selected model is indicated by the p-value being less than 0.05 (< 0.0001), demonstrating that each variable (power, size, and time) has a significant effect on the yield response.

Fit statistics demonstrated a negligible difference of less than 0.20 between the predicted R^2 of 0.9128 and the adjusted R^2 of 0.9624. The signal-to-noise ratio measured by Adeq Precision was 30.361, which is acceptable because a ratio of at least 4 is preferred. This model can be utilized to navigate the design space effectively.

Table 4
Experimental result

Run	Factor 1 A: power (W)	Factor 2 B: size (mm)	Factor 3 C: time (min)	Response Yield (%)
1	300	1	20	11.0913
2	300	2	10	7.7784
3	300	2	30	21.298
4	300	3	20	12.311
5	450	1	10	6.50809
6	450	1	30	18.5945
7	450	2	10	10.378
8	450	2	20	17.9256
9	450	2	30	22.2655
10	450	3	10	13.3115
11	450	3	30	30.4262
12	450	1	20	14.1319
13	600	2	10	12.2188
14	600	2	30	27.2574
15	600	3	20	28.6664
16	600	2	20	16.3218
17	450	2	20	18.1818
18	450	2	20	17.747
19	450	2	20	17.9256
20	450	2	20	18.1818

Overall, the optimization process utilizing RSM, and the face-centered central composite design technique yielded a reliable and significant model for predicting the % yield of liquid smoke. The results offer valuable insights into the influential factors (power, size, and time) and provide a practical approach for enhancing liquid smoke production from coconut shells.

The model presented is indeed significant, as evidenced by the model F-value of 82.03, as shown in Table 5. A large F-value like this could occur because of noise only 0.01% of the time. Their P-values determine the significance of model terms, and in this case, terms A, B, C, and AB are considered significant, with P-values less than 0.0500. However, if a term's P-value is greater than 0.1000, it is considered nonsignificant. The lack of fit F-value of 4.70 indicates a 5.26% probability that the observed lack of fit could be attributed to noise.

In other words, the lack of fit F-value suggests that noise may not significantly influence the model's fit. Overall, the statistical analysis confirms the significance of the model and identifies the relevant terms that contribute significantly to the % yield of liquid smoke, as shown in Table 5. These findings provide valuable insights for understanding the factors that affect the production process and can be utilized to optimize the process further.

3.7 *Optimization of liquid smoke from coconut shells*

The maximum response will be obtained from the curve of the plotted response surface so that it will be formed from the relationship between variables and the ideal grade for each variable. The response surface curve for liquid smoke from coconut shells using activated charcoal as an absorber by the microwave-assisted pyrolysis method is presented in Figures 11–13. The interaction of power (W) and size (mm) on the % yield of liquid smoke is shown in Figure 11. The interaction of power (W) and time (min) on the % yield liquid smoke is shown in Figure 12, and the interaction of size (mm) and time (min) on the % yield liquid smoke is shown in Figure 13. The final equation in terms of the real variables is shown in Equation 3. In the actual Equation, an equation will be obtained as a first-order regression model, which is usually expressed by a polynomial linear regression equation of order one as follows.

$$Y = 17.13 + 3.72\,A + 4.30\,B + 6.96\,C + 3.33\,AB + 0.3797\,AC + 1.26\,BC \qquad (3)$$

The optimum response can be visually depicted through the plot's contours and response surface (Figures 11–13). The plot can be used to determine the relationship between the response and its free variables (Ugwu, Sen Gupta, Adeloye, & Martínez-Villegas, 2022). The contour of the plot is a visual illustration of the shape of the surface % yield in response and changes in free variables, namely, power (W), the size of the raw material (mm), and the heating time (min). If the contour of the interaction plot of the three variables to the response has an optimum point in the center, it shows that the optimization model correctly describes the connection between the variable and the response. Meanwhile, in the response of the surface plot, the curve formed is a parabola.

The optimum obtained yield was 34.6% at the following conditions: power of 593.6 W, material size of 2.9 mm, and an optimal heating time of 28.5 min for the operating process. These parameters are recommended as the optimal formula solution. Power, raw material size, and heating time are crucial optimized components with a % yield of liquid smoke, and they

Table 5
Identification of major influences on the % yield liquid smoke using Analysis of Variance (ANOVA) for the response surface 2Fl model.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	795.09	6	132.51	82.03	< 0.0001	significant
A-power	110.97	1	110.97	68.70	< 0.0001	
B-size	147.83	1	147.83	91.51	< 0.0001	
C-time	485.07	1	485.07	300.27	< 0.0001	
AB	44.32	1	44.32	27.44	0.0002	
AC	0.5768	1	0.5768	0.3571	0.5604	
BC	6.32	1	6.32	3.91	0.0695	
Residual	21.00	13	1.62			
Lack of Fit	18.53	8	2.32	4.70	0.0526	Not significant
Pure Error	2.47	5	0.4933			
Cor Total	3816.09	19				

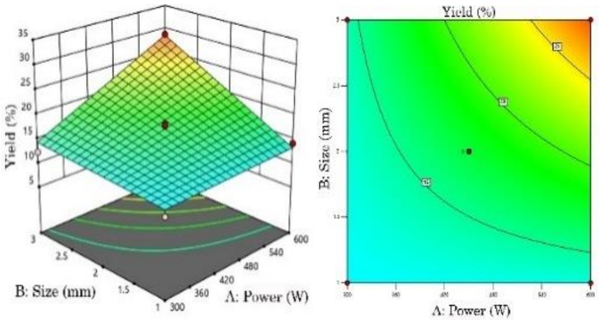

Fig. 11 Response surface showing effect of power (W) and size (mm) on the % yield liquid smoke (a) Plot contours (b) Response surface plot

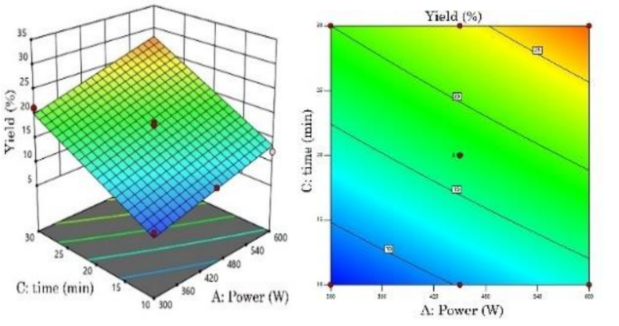

Fig. 12 Response surface showing effect of power (W) and time (min) on the % yield liquid smoke (a) Plot contours (b) Response surface plot

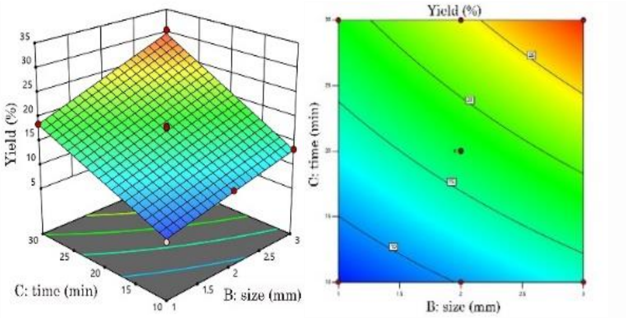

Fig. 13 Response surface showing effect of size (mm) and time (min) on the % yield liquid smoke (a) Plot contours (b) Response surface plot.

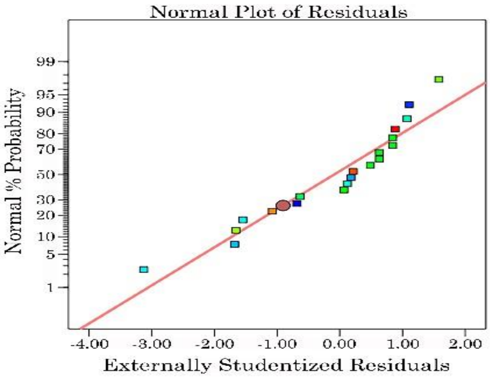

Fig. 14 Plot of normality of residual response % yield of liquid smoke

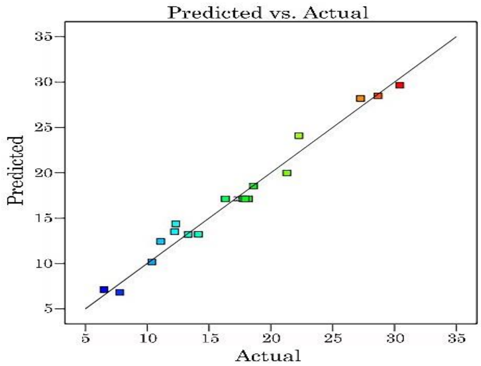

Fig. 15 Results of liquid smoke production experiments against liquid smoke production predicted

hold an importance level of 5 (+++++). This high importance level underscores the significant impact of these factors in achieving the maximum yield of liquid smoke from the pyrolysis process.

By utilizing these optimized values for power, raw material size, and heating time, liquid smoke production from organic raw materials can be highly efficient and can result in an increased yield of 34.6%. This optimized formula solution represents a substantial improvement in the liquid smoke production process, offering enhanced efficiency and quality, and it paves the way for more economically viable and sustainable applications of liquid smoke in various industries. It is the optimal formula solution in this condition because it has a desirability value of 1. A desirability value of 1 is interpreted as perfect between the target and the resulting optimization (Gutema, Gopal, & Lemu, 2022).

A graph of the normal residual response % liquid smoke yield illustrating the relationship between the actual value and the predicted value is shown in Figure 14. Fig 14 depicts that the response data % liquid smoke yield diffuses normally with close to the normal line. Thus, the relationship between the actual results and those predicted by Program Design Expert 13 has close results.

In the predicted vs. actual graph, the x-axis represents the values predicted by the model and the y-axis represents the actual measured values of the experiments performed. Each point in the graph represents one measurement from the experiment. The points of the experimental results get closer to the diagonal line (y = x), as shown in Figure 15. Therefore, the better the model's prediction with the actual value is, the better the prediction model is considered good and accurate.

4. Conclusion

Liquid smoke production from coconut shells using microwave-assisted pyrolysis was comprehensively explored, revealing the significant influence of both microwave power level and raw material size on the yield of liquid smoke. Through RSM analysis, optimal operating conditions were identified, resulting in an impressive yield of 34.6% with a microwave power of 593.6 W, raw material size of 2.9 mm, and a heating duration of 28.5 min.

GC–MS analysis of the liquid smoke produced at different power levels provided valuable insights into the chemical composition. Specifically, 14, 15, and 5 chemicals were identified at 300, 450, and 600 W power levels, respectively. The results showcased various chemical compounds with varying quantities at different power levels. Notably, phenol, dimethoxy phenol, guaiacol, hydroxyanisole, and methoxyphenol emerged as the most abundant compounds.

These findings underscore the potential of microwave-assisted pyrolysis as a viable and efficient method for producing high-quality liquid smoke from coconut shells. The research highlights this innovative approach's effectiveness, enabling liquid smoke generation with desirable yields and favorable chemical compositions. The outcomes of this study offer valuable contributions to the development of liquid smoke products with enhanced quality, flavor, and potential applications in the food industry.

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Author Contributions: S.P.A.A.: Preparation of equipment and materials, collection of experimental data, data analysis, draft manuscript, S.R.J.; analysis, review, monitoring and editing, S.S.; methodology, manuscript review and editing. M.M.; conceptualization, supervision, validation, manuscript review and final editing. All authors have read and agreed to the published version of the manuscript.

Conflicts of Interest: The authors declare no conflict of interest.

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