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EXTRACTION OF DAMMAR RESIN (*Agathis dammara*) USING MICROWAVE-ASSISTED HYDRODISTILLATION (MAHD)

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Abstract

Dammar resin (*Agathis dammara*) is a mixture of various organic polymers in a solid or semi-solid form that can be utilised as an essential oil source. This study extracted essential oil from dammar resin using microwave-assisted hydrodistillation (MAHD), considering its ability to extract the product in a short period with minimal solvent. This study investigates the effects of solvent type and extraction time on the yield of dammar resin extract. Results showed that the highest yields are 27.500, 22.902, and 15.392% for distilled water, a 1:1 v/v mixture of ethyl acetate and distilled water, and a 1:1 v/v mixture of hexane and distilled water as solvents, respectively. The optimum extraction time was different for different solvents, in the 60–90 minute range. Based on gas chromatography-mass spectrometry (GC-MS) analysis, the dammar resin extract contains several groups of components, including alcohols and sesquiterpenes. The alcohol group was higher after extraction using an ethyl acetate and distilled water mixture, especially 2-Butoxyethanol, with a yield of 39.706%. Meanwhile, a mixture of hexane and distilled water increased the sesquiterpene compounds in products such as *Germacrene D*, yielding 19.975%.

Keywords: Dammar; Extraction; Microwave; Resin

Abstrak

Getah damar (Agathis dammara) merupakan campuran dari beberapa polimer organik dalam bentuk padatan dan semi-padatan yang dapat dimanfaatkan sebagai sumber minyak atsiri. Penelitian ini mengekstrak minyak atsiri dari getah damar menggunakan microwave-assisted hydrodistillation (MAHD) yang dapat menghasilkan ekstrak dalam waktu singkat dengan volume pelarut minimal. Tujuan penelitian ini adalah untuk mempelajari pengaruh pelarut dan waktu ekstraksi terhadap rendemen ekstrak getah damar. Hasil menunjukkan bahwa rendemen tertinggi yang didapatkan sebesar 27,500%, 22,902%, dan 15,392% setelah diekstrak menggunakan air destilasi, campuran etil asetat dan air destilasi 1:1 v/v, serta campuran heksana dan air destilasi 1:1 v/v. Waktu optimum ekstraksi berbeda untuk pelarut yang berbeda, dengan rentang antara 60 – 90 menit. Berdasarkan analisis gas chromatography-mass spectrometry (GC-MS)*, ekstrak getah damar mengandung beberapa kelompok komponen, seperti golongan alkohol dan seskuiterpena. Golongan alkohol lebih banyak diperoleh pada ekstraksi menggunakan campuran etil asetat dan air destilasi, terutama 2-Butoxyethanol sebanyak 39,706%. Sedangkan, ekstraksi menggunakan campuran heksana dan air destilasi lebih banyak menghasilkan golongan seskuiterpen seperti Germacrene D sebanyak 19,975%.*

***Kata Kunci**: Damar; Ekstraksi;* Microwave*; Resin*

1. INTRODUCTION

The dammar tree (genus *Agathis*) is commonly found in Indonesia and is widely known for its high-quality resin (Asrorie et al., 2021). Dammar resin is a mixture of various organic polymers in solid or semi-solid form. According to Putra et al. (2021), dammar resin (*Shorea javanica*) from Krui (Pesisir Barat, Lampung) contains G*ermacrene D*, a type of sesquiterpene. Sesquiterpenes is a C15 terpenoids consisting of three isoprene units and are categorized as essential oils with aromatic components (Ninkuu et al., 2021). Its compounds are usually found in linear, cyclic, bicyclic, and tricyclic forms, and the form of lactone rings (Perveen, 2018). Moreover, sesquiterpenes are a subclass of terpenes with numerous benefits, such as antimicrobial, anti-tumour, anti-inflammation, and affecting the central nervous system health (da Silveira e Sá et al., 2015). Other studies identified the applicability of dammar resin as an alternative fuel (Jamal & Aisyah, 2022) and in composite materials (Stănescu & Bolcu, 2020).

Given its potential as a high-value product, dammar resin extraction is necessary to enhance its quality. A solvent can assist in extracting several compounds from dammar resin. Solvent binds a specific component in a plant during the extraction process. The solvent selection is based on the extract characteristics. In addition, the type and quality of solvent affect the extraction process and the extract quality. The type of solvent must be selected based on the nature of the material, using the principle that polar solvents will dissolve polar compounds and non-polar solvents will dissolve non-polar compounds (Nawaz et al., 2020). Dammar resin is insoluble in water but soluble in organic solvents. Thus, according to the theory, it can be effectively extracted using non-polar solvents.

There are two methods in the dammar resin extraction process: conventional and modern. Maceration, fractionation, hydro distillation, and Soxhlet are mostly known conventional methods. However, these methods require long processes, use hazardous solvents, and produce a low yield of extracts (Barão et al., 2024). Modern methods, such as ultrasound-assisted extraction, pulsed-electric field, and microwave-assisted extraction, improve the yield and quality of extracts due to the shorter process and lower solvent usage (Barão et al., 2024; Mathews et al., 2024).

Previous studies extracted essential oil from the cat's eye dammar using distillation (Mulyono, 2013; Wiyono, 1998). The results showed that the extract yield was 0.05–3.90% after 1–7 hours of extraction. Compared to microwave-assisted distillation (MAHD), the extract yield of pine resin oil was much higher, ranging from 12–30% (Sarah & Juwairiah, 2021)As mentioned before, MAHD is more efficient in extraction due to the introduction of heat from microwaves. However, studies that discuss the extraction of dammar resin using microwave-assisted distillation are limited. Therefore, this research investigates the effect of solvent and extraction time on the yield of dammar resin extract and its components.

2. MATERIALS AND METHODS

2.1 Materials

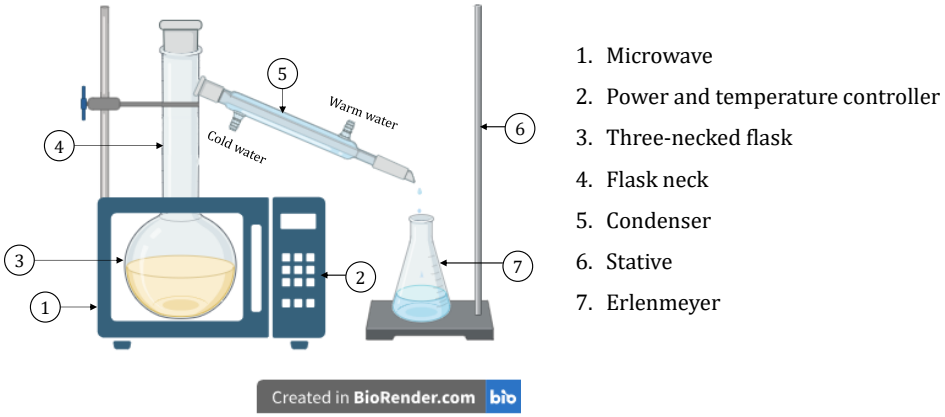
The dammar resin (*Shorea javanica*) was sourced from Pahmongan, Krui, Pesisir Barat, Lampung. This study used technical grade ethyl acetate 99%, N-hexane 99%, and distilled water for solvent variations.

2.2 Dammar Resin Extraction

Before extraction, the resin was crushed and sieved using a 50-mesh sieve. The fine resin (50 grams) was then mixed with 500 mL of solvent (1:10 w/v ratio). In this process, the solvent variations are distilled water (DW), ethyl acetate and distilled water (EA-DW), and hexane and distilled water (H-DW). The solvent ratio were 1:1 v/v for the mixtures of EA-DW and H-DW. The extraction times were 60 and 90 minutes at a constant microwave power of 375 watts. Extract from DW solvent was directly processed for distillation, while other extracts were filtered and evaporated. The evaporation process was conducted using a rotary evaporator (IKA RV 8) under vacuum conditions. Samples with different treatments were weighed to calculate the yield. The method is illustrated in Figure 1.

2.3 Sample Analysis

Several analyses were conducted to determine the optimal conditions for the dammar resin extraction



1. Microwave
2. Power and temperature controller
3. Three-necked flask
4. Flask neck
5. Condenser
6. Stative
7. Erlenmeyer

Figure 1. Illustration of the resin extraction method

process. The density was evaluated by filling the dammar resin extract into a pycnometer. The ratio of the total mass of the extract to volume was the density of the resin extract. Chemical compounds in the highest yield extract were analyzed using gas chromatography-mass spectroscopy (PerkinElmer–Clarus SQ 8C G-MS).

3. RESULTS AND DISCUSSION

3.1 Physical Appearance of the Extract

Table 1 displays the effect of the extraction solvent and time on the extract result. As can be seen in Table 1, the highest result was 15.41 mL with distilled water at 60 minutes. The maximum results for ethyl acetate and hexane solvents were 14.93 mL and 11.66 mL after 60 and 90 minutes of extraction, respectively. These data are supported by Figure 1, which shows the physical appearance of the extract.

Table 1. Extract after MAHD extraction

No	Solvent	Volume (mL)	Time (minutes)	Result (mL)
1	DW	500	60	15.41
			90	15.31
			120	14.69
2	EA-DW	500	60	14.93
			90	6.19
3	H-DW	500	60	4.67
			90	11.66

According to Figure 2, different solvents affected the extract's color. After being extracted using distilled water, the extract was more yellow than the others. However, the other crucial factor of the extract's color is the turbidity of the precursor. Furthermore, the appearance of the extract affected the appearance of the final product, dammar resin. Figure 3 shows the appearance of the dammar resin in a solid phase. Following the color of the extract, the resin of distilled water-assisted extraction showed the most yellow appearance. The color difference was evaluated using MATLAB by calculating the color change value (ΔE) according to the RGB color scale (Finn, 2021). The value is then categorized, as can be seen in Table 2.

Table 2. The color difference between samples (Finn, 2021)

Comparison	ΔE	Category
EA-DW vs H-DW	3.26	Noticeable with close inspection
EA-DW vs DW	5.68	Obvious color difference
H-DW vs DW	8.4	Obvious color difference

Category:
Low ΔE (< 1): Less color shift
High ΔE (> 5): Significant color shift

The color change in Figure 3 was also caused by solvent evaporation. The dammar resin's surface structure was broken during the evaporation heating process. An extended period of the heating process causes a thermo-oxidative degradation, changing the aromatic structure to other compounds (Pecora et al., 2016). Terpene oxidation also occurred during this process, darkening the dammar resin. One type of terpene found in dammar resin is *Caryophyllene*. As a result of *Caryophyllene* oxidation, *Caryophyllene* oxide (alcohol) is formed in the dammar resin (Putra et al., 2021).

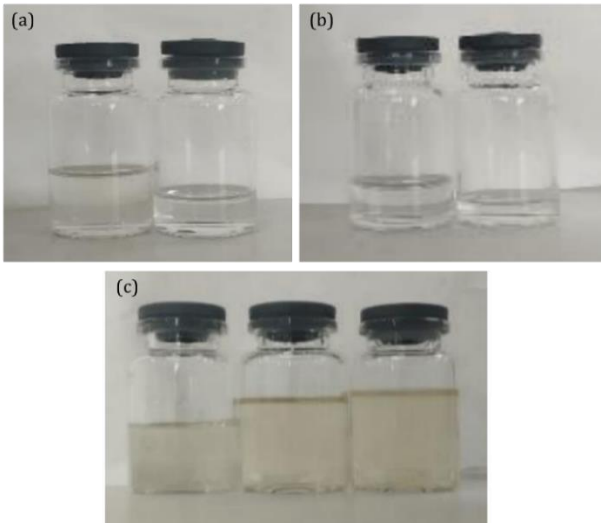


Figure 2. The physical appearance of dammar extract after MAHD extraction using (a) EA-DW, (b) H-DW, and (c) DW as solvents

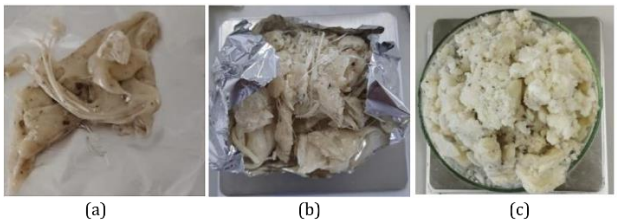


Figure 3. Dammar resin after extraction using (a) DW, (b) EA-DW, and (c) H-DW

The density of dammar resin was analyzed to assess its physical properties following extraction with various solvents. Table 3 presents the densities obtained using distilled water and ethyl acetate, while data for hexane were excluded due to insufficient resin yield. The result indicates that different solvents and extraction times produced different resin densities, primarily due to differences in solvent density (distilled water > ethyl acetate). Density is a vital parameter depending on the application. As an example, the density of dammar resin in this study is larger than needed for fuel application (Jamal & Aisyah, 2022).

Table 3. Density of dammar resin after extraction

Solvent	Extraction time (min)	Density (g/mL)	Average Density (g/mL)
DW	60	1.012	1.012
	90	1.012	
EA-DW	60	0.904	0.871
	90	0.839	

3.2 Effect of Extraction Solvent on the Extract Yield

Solvent type is an important factor in the extract yield. The extraction effectiveness highly depends on the solubility of compounds in the solvent. This phenomenon follows the 'like dissolves like' principle, which states that substances with similar properties dissolve in one another (Verdiana et al., 2018). This study used distilled water, ethyl acetate, and hexane solvents. These solvents were chosen based on their properties (polar, semi-polar, and non-polar) to compare the resin extract yields (Nawaz et al., 2020). Figure 4 represents the effect of solvents and extraction time on the extract yield.

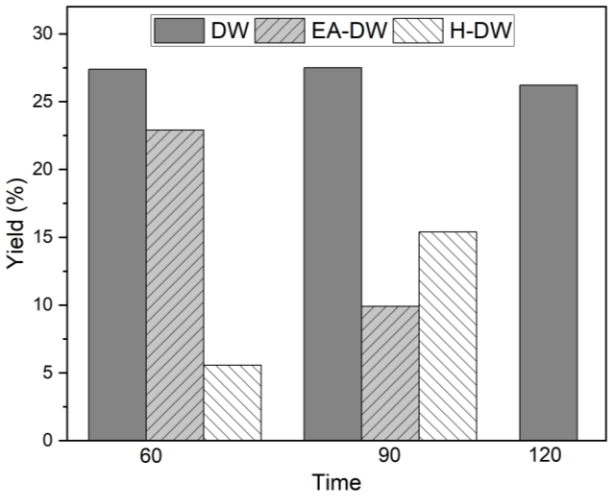


Figure 4. Extract yield at different solvents and extraction times

Solvent type is an important factor in the extract yield. Based on the yield, the solvent order from highest to lowest is distilled water > ethyl acetate > hexane. A previous study stated that the solvent polarity impacts yield (Maghfiroh et al., 2019). Materials' polarity can be seen from their dipole moment and dielectric constant. A solvent with a high dipole moment and dielectric constant is categorized as a polar solvent, and vice versa (Liu et al., 2013). According to the data, water has a higher dielectric constant than ethyl acetate and hexane (Maryott & Smith, 1951). Water has a dielectric constant of 78.54, while ethyl acetate and hexane have dielectric constants of 6.02 and 1.89, respectively. Moreover, the dipole moments of distilled water, ethyl acetate, and hexane are 1.85, 1.81, and 0.08, respectively (Li & Du, 2011). The molecular structure of solvents in this research is shown in Figure 5.

Dammar resin contains polar and non-polar compounds, which are discussed in the GC-MS analysis. Polar compounds, such as ethanol, are highly soluble in distilled water. Non-polar compounds in dammar resin (sesquiterpenes) are highly soluble in hexane, a non-polar solvent. The molecular structures of sesquiterpenes are represented in Figure 6**,** indicating non-polar compounds.

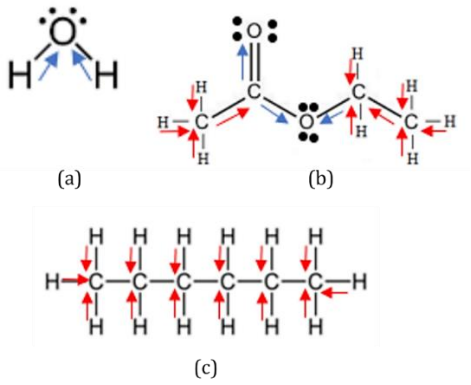


Figure 5. Molecular structure of (a) water, (b) ethyl acetate, and (c) hexane

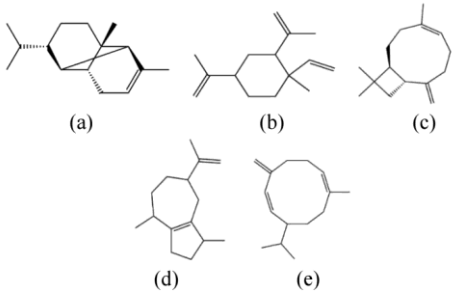


Figure 6. Molecular structure of sesquiterpenes: (a) *Ylangene*, (b) *Elemene,* (c) *Caryophyllene*, (d) *alpha-Guaiene*, and (e) *Germacrene D*

Introducing microwave radiation increases the oscillation and rotation of ions in the solvent. During this period, the ions are adjusted to the microwave direction (Figure 7) (Anwar et al., 2015). Thus, these ions collide and generate heat. When the solvent is non-polar with positively charged ions, the heating process occurs poorly due to the fewer electrons that respond to the microwave (Llompart et al., 2019).

Non-polar solvents can still be used for extraction by combining them with a polar solvent at a ratio of 1:1 (Llompart et al., 2019). The purifying process is an important stage in material extraction. This study used a simple distillation process to separate the distilled water solvent and extract. This method is less effective due to its long period and periodic temperature checking, so the temperature does not exceed the boiling point.

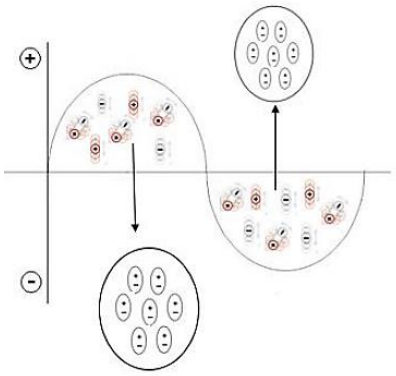


Figure 7. Mechanism of polar solvent heating using a microwave

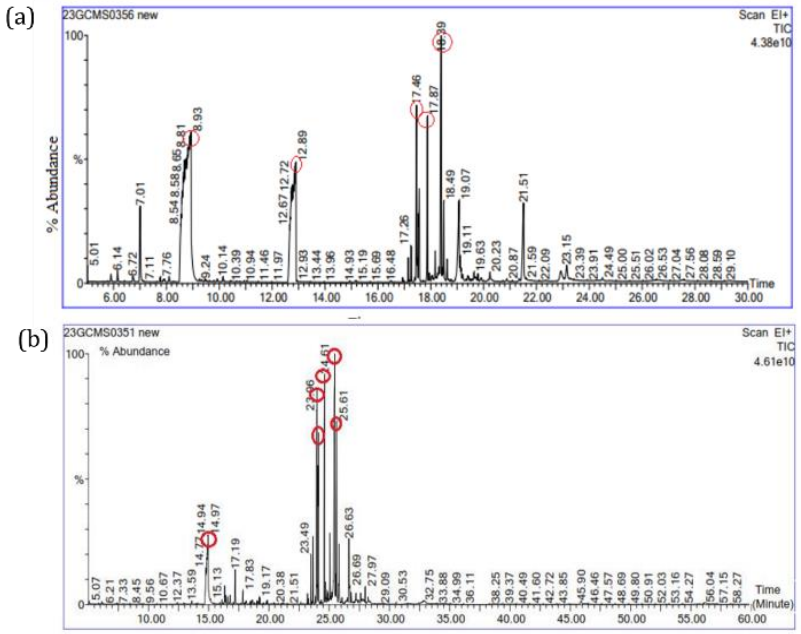


Figure 8. GC-MS analysis of dammar resin extract with solvents: (a) ethyl acetate and (b) hexane

A rotary evaporator separated ethyl acetate and hexane solvents from the extract. The challenge of using a rotary evaporator is the pressure fluctuation. However, in this method, the pressure is controlled under a vacuum condition. Under these conditions, the pressure is low and the solvent's boiling point is decreased. The evaporation becomes possible at lower temperatures (Chen et al., 2024).

3.3 Effect of Extraction Time on the Extract Yield

The extraction time can be different based on the sample and solvent properties (Llompart et al., 2019). According to Valarezo et al. (2020), the extraction time for dammar using microwave-assisted hydrodistillation was in the range of 1–2 hours. However, the optimum extraction time was at 1.5 hours. Figure 4 shows the effect of extraction time on the extract yield.

The data show no significant change in the extract yield after 60 minutes, especially for distilled water solvent. This result is similar to that of Moradi et al. (2018) who observed that the extraction process was fast at first, but became slow and constant after a longer extraction time. For ethyl acetate extraction, the yield decreased with longer extraction times. In contrast, after extraction using a hexane-distilled water mixture, the yield was higher at a longer time (90 minutes). A similar pattern was discovered in a previous study, finding that different solvents had different optimum extraction times (Mahmudah et al., 2025). After reaching the optimum time, the oxidation reaction controlled the process and led to the extract degradation. In this research, the oxidation was faster when the sample was extracted using a mixture of ethyl acetate and distilled water.

3.4 Dammar Resin Extract Composition

The chemical composition of the dammar resin extract was analyzed using GC-MS analysis. The analyzed samples had the highest extract results, which are 60 minutes for ethyl acetate and 90 minutes for hexane. The chemical composition is represented in Figure 8.

After extraction using ethyl acetate (Figure 8 (a)), several peaks were found at retention times of 8.925, 12.892, 17.458, 17.874, and 18.394 minutes. Several peaks indicate sesquiterpenes, including Ylangene at 17.458 minutes, Caryophyllene and Cyclodecadiene, 1-methyl-5-methylene-8-(1-methylethyl)-, [S-(E,E)]- at 17.874 and 18.394 minutes. This extract also contains alcoholic compounds, shown at retention times 8.925 and 12.892 minutes for 2-butoxyethanol and 2-butoxyethyl acetate.

Figure 8 (b) displays the chemical compounds of dammar resin after extraction by hexane. The curve shows 50 chemical compounds. Some of the chemicals are sesquiterpene, including Cyclohexane, 1-ethenyl-1-methyl-2,4-bis(1-methylethenyl)-, [1S(1à,2á,4á)] (24.126 minutes), Germacrene D (25.472 minutes), Ylangene (23.976 minutes), caryophyllene (RT 24.607 minutes), and Azulene, 1,2,3,3a,4,5,6,7-octahydro-1,4-dimethyl-7-(1- methylethenyl)-, [1R-(1à,3aá,4à, 7á)]- (25.612 minutes). This sample also contains alcohol, 2-butoxyethanol, at 14.968. The chemical compounds found in the dammar resin extract are consistent with previous research (Jamal et al., 2015).

The chemical content of the dammar resin extract is summarized in Table 4. Polar compounds (alcohol) in dammar resin were highly extracted using ethyl acetate as a solvent. In contrast, hexane solvent extracted more non-polar compounds (sesquiterpenes) from dammar resin. This result shows that solvent selection is essential, depending on the application of dammar resin. The alcoholic compounds show their potential as renewable energy (Jamal & Aisyah, 2022) and the sesquiterpenes have been used in medical applications due to their antimicrobial and anti-inflammatory activities (da Silveira e Sá et al., 2015).

Table 4. The chemical compound in the dammar resin extract

Compound	Chemical formula	Common name	Percentage (%)	
			Ethyl acetate	Hexane
Ethanol, 2-butoxy-	$C_6H_{14}O_2$	Ethylene glycol monobutyl ether	39.706	12.640
2-Butoxyethyl acetate	$C_8H_{16}O_3$	Butyl glycol acetate	19.270	-
1,6-Cyclodecadiene, 1-methyl-5methylene-8-(1-ethylethyl)-,[s(E,E)]	$C_{15}H_{24}$	Germacrene D	6.996	19.975
Ylangene	$C_{15}H_{24}$	Ylangene	3.815	11.176
Caryophyllene	$C_{15}H_{24}$	Caryophyllene	2.890	9.522
Cyclohexane, 1-ethenyl-1-methyl-2,4-bis(1-methylethenyl)-,[1S-(1à,2á,4á)]	$C_{15}H_{24}$	Elemene	2.505	5.375
Azulene, 1,2,3,3a,4,5,6,7-octahydro-1,4-dimethyl-7-(1-methylethenyl)-, [1R(1à,3aá,4à, 7á)]-	$C_{15}H_{24}$	alpha-Guaiene	-	6.712

Table 4 shows that the identified alcohol groups for ethyl acetate and hexane solvents are 39.706% and 12.650%, respectively. This result is higher than the dammar extraction using the distillation method, which only produced 0.05–3.90% extract yields (Mulyono, 2013; Wiyono, 1998). The higher value of alcohol in ethyl acetate solvent is due to the solvent and resin extract polarity. Because of the similar polarity of ethyl acetate and dammar resin, the amount of alcohol group found in this extraction was higher than that of the hexane solvent (Putra et al., 2021). Polar molecules in dammar resin, distilled water, and ethyl acetate interact through dipole-dipole interactions, causing unequal electron distribution. The positive end of one molecule is attracted to the negative end of another (Han, 2023).

The heating process during the purification also affects the alcohol group formation. Microwave power induced oxidation by converting the microwave energy into heat and triggering oxidative degradation of sesquiterpenes (Tian et al., 2022). When the dammar resin was exposed to heat, *Germacrene D* inside was oxidized and formed alcohol. Finally, the heat exposure oxidized the alcohol and formed aldehyde, carboxylic acid, and ketone (Putra et al., 2021). *Germacrene D* is one of the compounds contained in copal oil. In this study, *Germacrene D* obtained from hexane solvent was 19.975%, and ethyl acetate was 6.996%. In copal oil, *Germacrene D* compound is 0.155%. These differences are due to the geographic factor. Two factors cause different dammar resin content in each region. First is the internal factor, such as the dammar tree genes. Second, the external factors, including light exposure, pH, nutrients in soil, altitude, and environmental conditions (Katuuk et al., 2018).

3.5 Statistical Analysis

The effect of extraction solvent and time was analyzed using a statistical approach, analysis of variance (ANOVA). Table 5 presents the ANOVA of this research. The assessment indicates that the variations have no statistically significant effect on the extract yield (p-value > 0.05). This result may be attributed to the narrow range of extraction times (only 60 and 90 minutes), which may have limited the observable differences. A broader time range or additional intermediate time points could reveal more distinct patterns.

Table 5. ANOVA of extract yields

Source of Variation	SS	df	F	*P-value*	*F crit*
Time	0.570	1	0.018	0.905	18.513
Solvent	53.696	2	0.865	0.536	19.000
Error	62.058	3			
Total	116.325	5			

*Significant if p-value < 0.05

4. CONCLUSION

Dammar resin is successfully extracted using a microwave-assisted hydrodistillation. This research observed the extract result by varying the extraction solvent and time. Results show that distilled water solvent produced the highest dammar resin extract, yielding 27.50%. Meanwhile, the highest ethyl acetate and hexane yields were 22.90 and 15.39%, respectively. Based on the experiment, increasing the extraction time increased the dammar resin extract for distilled water and ethyl acetate solvents, in contrast with that of hexane. GC-MS analysis revealed that ethyl acetate extraction produced a higher concentration of alcohol groups, whereas hexane extraction resulted in a greater formation of sesquiterpenes. Future research could explore using specific solvents to optimize the extraction efficiency for specialized applications.

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