

MICROWAVE-ASSISTED HYDRODISTILLATION EXTRACTION OF CRUDE OIL FROM ARUMANIS MANGO KERNEL (*Mangifera indica* L.)

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Abstract

Arumanis mango seeds (*Mangifera indica* L.) are generally underutilized and still considered agricultural waste. Mango seeds offer various benefits, including antioxidant properties, cholesterol-lowering effects, and natural antibiotic activity. This study aims to determine the yield of mango kernel extract, evaluate the influence of solvent type and extraction time on extraction efficiency, and identify the chemical composition of the extract. The extraction process was carried out using the microwave-assisted hydrodistillation (MAHD) at 375 watts, employing three types of solvents: ethanol, ethyl acetate, and n-hexane, with extraction times of 30, 60, and 90 minutes. The chemical composition of the extracts was analyzed using gas chromatography-mass spectrometry (GC-MS) analysis. The results showed that the highest yield was obtained using ethanol at an extraction time of 30 minutes, yielding 40.342%. Based on GC-MS analysis, in the n-hexane at a 90-minute extraction time, the most abundant fatty acid detected was butyric acid, with a retention time of 14.06 minutes and a peak area of 9.852%. Meanwhile, the most dominant compound was hydroxymethyl furfuraldehyde (HMF), which appeared at a retention time of 22.09 minutes with a peak area of 12.437%.

Keywords: Extraction time; Fatty acid; Mango seed; Microwave-assisted hydrodistillation; Solvent

Abstrak

Biji mangga Arumanis (*Mangifera indica* L.) umumnya belum dimanfaatkan secara optimal dan masih dianggap sebagai limbah pertanian. Padahal, biji mangga memiliki berbagai manfaat, antara lain sebagai antioksidan, penurun kadar kolesterol dalam tubuh, serta sebagai antibiotik alami. Penelitian ini bertujuan untuk mengetahui hasil perolehan yield ekstrak biji mangga, mengevaluasi pengaruh jenis pelarut dan waktu ekstraksi terhadap efisiensi ekstraksi, dan mengidentifikasi komposisi senyawa dalam ekstrak biji mangga. Ekstraksi dilakukan menggunakan metode microwave assisted hydro-distillation (MAHD) dengan daya 375 watt, menggunakan tiga jenis pelarut, yaitu etanol, etil asetat, dan n-heksana, serta variasi waktu ekstraksi selama 30, 60, dan 90 menit. Analisis kandungan senyawa dalam ekstrak dilakukan menggunakan analisis gas chromatography-mass spectrometry (GC-MS). Hasil penelitian menunjukkan bahwa yield tertinggi diperoleh pada pelarut etanol dengan waktu ekstraksi 30 menit, yaitu sebesar 40,342%. Berdasarkan analisis GC-MS, pada variasi pelarut n-heksana dengan waktu ekstraksi 90 menit, senyawa asam lemak terbesar yang terdeteksi adalah asam butirat dengan waktu retensi 14,06 menit dan luas area sebesar 9,852%. Sementara itu, senyawa dengan komposisi paling dominan secara keseluruhan adalah

hydroxymethyl furfuraldehyde (HMF), yang muncul pada waktu retensi 22,09 menit dengan luas area sebesar 12,437%.

Kata kunci: Asam lemak; Kernel biji mangga; Microwave-assisted hydrodistillation; Pelarut; Waktu ekstraksi

1. INTRODUCTION

Most activities carried out by living beings produce waste. Excessive waste production's side effects will indirectly produce environmental problems. In general, mangoes are only utilized as part of the fruit, but the seed and peel are thrown away, even though there are still many benefits that can be taken from the mango fruit waste, especially in the seeds (Mas'ud & Puspitasari, 2017). Data from the Central Bureau of Statistics (BPS) recorded that mango production in Indonesia reached 3.3 million tons in 2023 (Badan Pusat Statistik, 2024). Arumanis mango is widely sold at juice stalls due to its sweet taste, smooth texture, and distinctive aroma, making it superior to other mango varieties. However, increased production and sales have also led to a rise in arumanis mango seed waste. Generally, around 40–60% of mango fruit waste is generated during processing, with 12–15% peel and 15–20% seed. Mango seeds are valuable due to their high extract content (16–20%), which contains 44–48% saturated and 52–56% unsaturated fatty acids (Karunanithi et al., 2015). An extraction method is needed to efficiently extract the oil content from the mango seed kernel (Mariod et al., 2017).

The oil extracted from mango kernel contains various fatty acids that are beneficial to the body, such as stearic acid, which is helpful as a lubricant in tablet manufacturing (Wu et al., 2015); oleic acid, which helps lower blood pressure; linoleic acid, which maintains skin health, helps maintain skin elasticity, prevents premature aging, and helps prevent inflammation; and palmitic acid, which is a barrier that makes the skin smoother and helps fight skin cancer (Yarova et al., 2022). The choice of solvent in mango seed kernel extraction significantly influences the types of compounds that can be extracted. As a polar solvent, ethanol is effective in extracting hydrophilic compounds such as sugars, organic acids, and phenolic compounds (Mas'ud et al., 2021). Ethyl acetate, with its semi-polar nature, can dissolve moderately polar compounds such as esters and aldehydes (Pratiwi et al., 2016; Saputri et al., 2023). Meanwhile, n-hexane, a non-polar solvent, is suitable for extracting lipophilic compounds such as fatty acids (Balacuit et al., 2021).

Mango seed kernels can be extracted using various conventional methods, such as maceration and soxhlet extraction (Mariod et al., 2017; Gurjar & Raj, 2022). Conventional extraction methods have several drawbacks: long extraction time, high energy consumption, and excessive use of solvents, which not only elevate operational costs but also result in low extraction efficiency (Bitwell et al., 2023). These drawbacks highlight the need for alternative extraction approaches that are both energy-efficient and environmentally sustainable. The innovative extraction

technique enables the mango kernel oil to be obtained in a shorter time, with reduced energy consumption and less solvent usage (Balacuit et al., 2021). This approach typically involves using microwave or ultrasonic technology. The latest method, microwave-assisted hydrodistillation (MAHD), offers the benefits mentioned earlier. Gurjar & Raj (2022) reported that the soxhlet extraction of mango kernel oil yielded 10.5% after 4 hours. Compared to microwave-assisted distillation (MAHD), the extract yield of pine resin oil was much higher, ranging from 12 to 30% (Sarah & Juwairiah, 2021). As previously noted, MAHD enhances extraction efficiency by utilizing microwave-induced thermal energy. However, there is still a lack of research specifically focusing on the application of MAHD for extracting oil from mango seed kernels. Therefore, this study assesses how solvent selection and extraction time variations impact mango seed kernel extract yield and its components.

2. MATERIALS AND METHODS

2.1 Materials

The materials used included arumanis mango seeds obtained from fruit juice stalls, 96% ethyl acetate, 96% n-hexane, and 96% ethanol technical grade.

2.2 Extraction of Arumanis Mango Kernel

2.2.1 Preparation of raw material

The mango was peeled to obtain the kernel. Then, the kernel was ground using a blender and dried in an oven at 100 °C for 2 hours. The resulting kernel powder was sieved using a 50-mesh filter.

2.2.2 Extraction and distillation

About 40 grams of powdered kernel were mixed with 320 mL of solvent, prepared by combining 180 mL of distilled water and 180 mL of either ethanol, ethyl acetate, or n-hexane. This gave a material-to-solvent ratio of 1:8 (m/v). The modified microwave was turned on at 375 W, and various time intervals (30, 60, 90 minutes) were employed. The schematic of the device and the extraction process using a microwave-assisted hydro-distillation unit is shown in Figure 1. The oil extracted from the kernel was distilled at 100 °C and 1 atm for 4 hours.

2.2.3 Yield analysis

After the purification process, it was first transferred into bottles and weighed. The yield was calculated using Equation (1).

$$\text{yield (\%)} = \frac{\text{mass of extract}}{\text{mass of sample}} \times 100\% \quad (1)$$

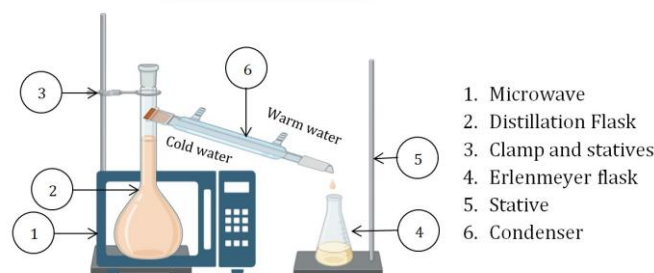


Figure 1. Schematic of the extraction device (microwave-assisted hydro-distillation)

2.2.4 Gas chromatography-mass spectrometry (GC-MS) analysis

Quantitative and qualitative analyses of the compound were performed using Gas Chromatography–Mass Spectrometry (PerkinElmer Clarus SQ 8C GC-MS) to determine its concentration and molecular structure.

3. RESULTS AND DISCUSSION

3.1 Extraction Results of Arumanis Mango Seed Kernel

Based on the data in Table 1, the highest yield percentage was obtained using ethanol with an extraction time of 30 minutes, which was 40.342%. In the variation of ethyl acetate solvent, the highest yield was achieved at an extraction time of 30 minutes, with a value of 39.434%. Meanwhile, using n-hexane produced a maximum yield of 39.547% at the same extraction time. Although ethanol solvent produced the highest yield quantitatively, the fatty acid content obtained with n-hexane was higher than that obtained from ethanol and ethyl acetate. This difference is caused by the nature of non-polar fatty acids, which are more easily soluble in n-hexane, which is also non-polar (Balacuit et al., 2021).

Table 1. Extraction result of the arumanis mango seeds

Solvent	Extraction Time (Minutes)	Yield (%)
Ethanol (96%)	30	40.342
	60	25.986
	90	22.752
Ethyl Acetate (96%)	30	39.434
	60	35.964
	90	16.745
N-Hexane (96%)	30	39.547
	60	36.842
	90	25.175

Figure 2 shows the dark brown color of the kernel extract, which is caused by the extraction of methyl 3,4,5-trihydroxybenzoate or methyl gallate in significant amounts. This occurs because of the

extraction using ethanol as a solvent (Saleem et al., 2013), which has polar covalent bonds. A previous study stated that mango seed extract also contains a phytochemical compound called gallotannin, which has a brownish-yellow color (Yadav et al., 2022). This compound acts as an antibacterial against various gram-positive and harmful bacteria (Siregar et al., 2024).



Figure 2. Mango kernel extract with ethanol solvent



Figure 3. Mango kernel extract with ethyl acetate solvent

Figure 3 shows a thick brown color, slightly lighter than the sample with ethanol solvent. Ethyl acetate, a semi-polar solvent with a polarity level of 4.4, in contrast to other polar solvents such as ethanol, which has a polarity level of 5.2. Because ethyl acetate has a lower polarity than ethanol, the polar compounds extracted were less than those extracted with ethanol solvents such as penta-O-galloyl- β -D-glucose, gallatin, and methyl gallate (Siregar et al., 2024). Some fatty acids, such as stearic acid, butyric acid, and oleic acid,

can be extracted with ethyl solvents (Saputri et al., 2023). This also explained why the kernel extract obtained with ethyl acetate appears lighter brown than that obtained with ethanol.



Figure 4. Mango seed extract with n-hexane

Figure 4 shows a darkest color extract. N-hexane can extract various fatty acid content, such as butyric acid, caprylic acid, and stearic acid (Mariod et al., 2017). The color of the extract will become more intense (dark) as the concentration of hydroxymethyl furfuraldehyde (HMF) increases because oxygen from the environment will react with HMF and produce extra dark pigments (Shapla et al., 2018). After the extraction, the extracts were separated using a simple distillation method, and then the volume of each result was recorded.

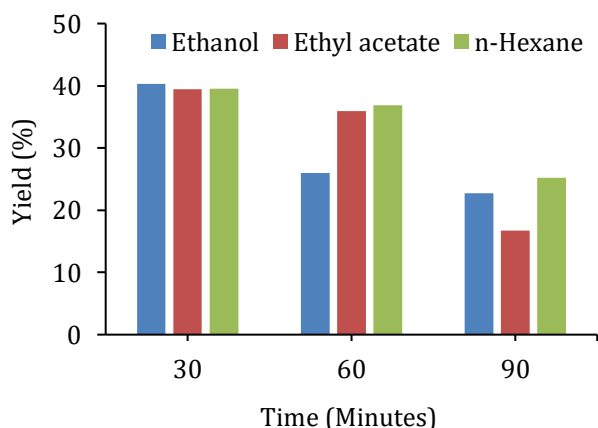


Figure 5. The effect of the solvent on the yield obtained

Figure 5 shows the impact of solvent on extract yield. The highest yield for each solvent was obtained at 30 minutes: 40.342% for ethanol, 39.434% for ethyl acetate, and 39.547% for n-hexane. This phenomenon occurs because each solvent has a different preference for the types of compounds it extracts. The dielectric constant of each solvent also affects its ability to extract certain compounds.

The solvents utilized in this study exhibited varying dielectric constants: ethanol of 24.3, ethyl acetate of 6.02, and n-hexane of 1.89, which directly correlate with their respective polarity levels. A higher dielectric constant indicates a higher degree of polarity,

influencing the solvent's ability to dissolve specific target compounds (Manna et al., 2023). In general, polar solvents are more effective at solubilizing polar compounds, while non-polar solvents exhibit greater affinity toward non-polar molecules (Saputri et al., 2023).

In mango kernel oil extraction, solvent polarity plays a critical role in determining extraction efficiency and compound selectivity. Due to its polar nature and hydrogen bonding capability, ethanol facilitates the extraction of polar compounds such as phenolics, flavonoids, and organic acids (Ghaffar & Perveen, 2024). Conversely, n-hexane, with its low polarity, selectively extracts non-polar components such as fatty acids and other lipophilic substances (Queffelec et al., 2024). Furthermore, in microwave-assisted extraction, polar solvents with higher dielectric constants absorb microwave energy more effectively through dielectric heating. This process enhances cellular disruption and mass transfer, ultimately improving the extracted compounds' yield and specificity (Chy et al., 2024).

The data in Figure 5 shows that ethanol produces more yield than other solvents, since ethanol is capable of dissolving almost all types of compounds, regardless of polarity. Therefore, although some components of mango kernel extract are non-polar, they can still be extracted by ethanol, a polar solvent. So, the greater the level of polarity of a solvent, the more compounds in a material the solvent can absorb.

In Figure 5, it can be seen that the longer the extraction time, the lower the yield obtained. This phenomenon occurs because the optimum extraction time is at 30 minutes. After this time, there is an increase in solvent evaporation due to the accumulation of heat energy from microwaves, especially when the cell structure of the mango kernel begins to degrade (López-Salazar et al., 2023). As a result, the amount of extracted compounds decreases, and the percentage yield produced tends to decrease.

The heat mechanism in microwaves involves polar rotation, which arises because the polar moments in matter are altered by radiation from electric and magnetic fields. Microwaves, which have mutually perpendicular electric and magnetic fields, cause a change in the polar orientation of matter. This momentary change takes place rapidly, resulting in heat generation. One of the unique characteristics of microwave heating is that it heats the entire volume, where even heating occurs in all parts of the sample, allowing uniform and rapid heating. It contrasts with conventional heating, where heat is transferred through conduction, convection, and radiation. In conventional heating, heat transfer occurs due to temperature differences within the sample (Anwar et al., 2015).

3.2 Gas Chromatography Mass Spectrometry (GC-MS) Analysis

A GC-MS analysis was conducted to determine the fatty acid content of the mango kernel extract. The characterization was carried out on samples extracted

using n-hexane as the solvent with an extraction time of 90 minutes. Although the extract obtained with ethanol yielded a higher amount, n-hexane was specifically chosen for this analysis due to its higher efficiency in extracting fatty acid components (Mariod et al., 2017; Gurjar & Raj, 2022). The characterization process was conducted with a retention time of 60 minutes.

Figure 6 shows that at retention times of 8.36, 14.06, 22.09, 26.74, 47.50, 47.52, 57.73, and 57.76 minutes, the content of fatty acids and several other valuable contents was found. Among these, fatty acids were prominently identified in the mango kernel extract. At a retention time of 14.06 minutes, the highest concentration of fatty acids was found, specifically butanoic acid, with a peak area of 9.852% of the total extract. Additionally, acetic acid was detected at a retention time of 7.765 minutes, with an area of 1.296%, and dodecanoic acid, a saturated fatty acid, appeared at 26.742 minutes with a peak area of 0.987%. Both butanoic and acetic acids are classified as volatile fatty acids and are widely applied in food industries, particularly in flavoring, preservation, and fermentation processes (Deshmukh & Manyar, 2020). Their commercial applications are extensive, yet they often require further purification to comply with regulatory standards. The fatty acid composition obtained in this study differs from that reported in the literature. Balacuit et al., (2021) found propanoic, oleic, palmitic, and stearic acid in mango kernel oil. The differences in composition are caused by the extraction method, as occurred in other studies with different methods, the soxhlet method and microwave-assisted extraction.

In addition to some of the fatty acid content obtained, it was also found that the largest content of mango seed extract at retention time 22.09 was 2-furancarboxaldehyde, 5- (hydroxymethyl) or hydroxymethyl furfuraldehyde (HMF), with a large area of 12.437%. HMF is not typically reported as a predominant compound in mango kernel extracts. As observed in this study, the notable presence of HMF in

mango seed kernel extracts suggests a unique phytochemical profile that may result from extraction methods or thermal degradation during preparation. HMF is typically formed through the thermal decomposition of sugars, which can occur during processing (Choudhary et al., 2020). Given the high concentration of HMF, the extract is more suitable for use in the bio-based chemical industry. HMF is a well-known platform chemical derived from biomass. It serves as a key precursor in the production of bioplastics (such as polyethylene furanoate/PEF), biofuels (such as 2,5-dimethylfuran), and various green solvents and resins (Fan et al., 2019). In addition to HMF, this mango seed extract contained another functional composition, furfural, which was found at a retention time of 11.576 minutes with a large area of 2.827%. Furfural is an aromatic aldehyde compound that can be produced from raw materials containing pentosan. Furfural is a chemical substance with several uses, one is as a solvent in separating saturated and unsaturated compounds in the petroleum extraction industry. In addition, it also acts as an intermediate compound in the production of various other industrial chemicals (Koesprimadisari et al., 2018). At a retention time of 8.360, the compound 2-Propanone, 1-hydroxy—commonly called acetol—was found. Acetol is a primary alcohol and the simplest ketone hydration structure. It is also an intermediate material for propylene glycol compounds, greatly benefiting the chemical industry (Mohamad et al., 2011).

At the retention time of 47.50-47.52 minutes, the compound hexadecanoic acid, 2-hydroxy-1-(hydroxymethyl) ethyl ester, was found with a large area of 10.843%. The compound is known to have good antioxidant activity because it belongs to the phenolic group. The high phenolic content in hexadecanoic acid, 2-hydroxy-1-hydroxymethyl-ethyl ester, reduces free radicals from the mango seed and can be implemented into beauty or health ingredients (Al-Marzoqi et al., 2015).

At the retention time of 57.73-57.76 minutes, the compound 9-octadecenoic acid (Z)-, 2,3-

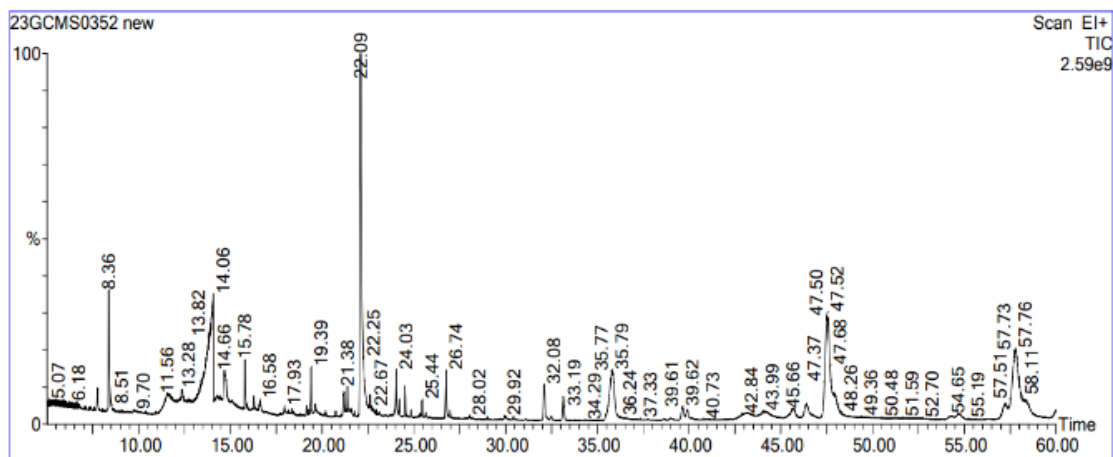


Figure 6. The GC-MS chromatogram of mango seed extract

dihydroxypropyl ester or glycerol monooleate (GMO) was found with a large area of 10.845%. GMOs are lipid excipients consisting of several monoglycerides, have antioxidant activity, are characteristic of brownish yellow, and have a distinctive odor (Loi et al., 2020). The chemical compounds of the mango seed kernel extract is summarized in Table 2.

Table 2. The chemical compounds of mango kernel oil extract

Compound	Chemical formula	Percentage (%)
<i>Butanoic acid</i>	$C_4H_8O_2$	9.852
<i>Acetic acid</i>	$C_2H_4O_2$	1.296
<i>Dodecanoic acid</i>	$C_{12}H_{24}O_2$	0.987
<i>2-Furancarboxaldehyde, 5- (hydroxymethyl) (HMF)</i>	$C_6H_6O_3$	12.437
<i>Furfural</i>	$C_5H_4O_2$	2.827
<i>2-Propanone, 1-hydroxy- (acetol)</i>	$C_3H_6O_2$	2.933
<i>Hexadecanoic acid, 2-hydroxy- 1- (hydroxymethyl) ethyl ester</i>	$C_{19}H_{38}O_4$	10.843
<i>9-Octadecenoic acid (Z)-, 2,3- dihydroxypropyl ester</i>	$C_{21}H_{40}O_4$	10.845

3.3 Comparison with Previous Research

The comparison of several extraction methods and their results is presented in Table 3. There are notable differences in the yields obtained from the three methods. Among them, the microwave-assisted hydrodistillation (MAHD) method using ethanol at 100 °C for 30 minutes produced the highest yield at 40.342%, making it the most efficient method in terms of both extraction time and yield. In comparison, the soxhlet extraction required a longer duration and resulted in moderate yields depending on the solvent used: ethanol (17.45%), *n*-hexane (10.60%), and ethyl acetate (11.03%). Ethanol consistently yielded higher extract amounts compared to the other solvents. Meanwhile, the maceration method, carried out at room temperature (28 °C) for 8 hours using ethanol, achieved a yield of 17.28%, comparable to soxhlet extraction with ethanol, despite the absence of heating.

The maceration method requires a longer and slower process because this extraction process does not involve heating; it relies solely on soaking the material (Rudiana et al., 2023). This method is known for its ability to extract heat-sensitive compounds, as it is conducted at room temperature and is relatively simple (Yoswathana & Eshiaghi, 2013).

The microwave-assisted hydrodistillation (MAHD) method is a combination of the principles of both maceration and soxhlet extraction, enhanced by the use of microwave heating and the addition of distilled water to the solvent. In this method, microwaves are the heat source that accelerates the extraction process rapidly and efficiently heat the solvent. This approach significantly reduces the extraction time and allows for the use of less solvent due to improved efficiency (Balacuit et al., 2021). However, it is essential to note that elevated temperatures during extraction, particularly in microwave-assisted methods, may pose a risk to heat-sensitive compounds. Prolonged exposure to high heat can lead to degradation, oxidation, or loss of bioactive components such as phenolics, flavonoids, and specific fatty acids (Antony & Farid, 2022). Therefore, temperature and power settings must be carefully optimized to ensure effective extraction while minimizing the potential breakdown of these valuable compounds. In this study, a medium microwave power of 375 watts was applied to avoid excessive temperatures and prevent overheating during extraction. Variations in yield may result from differences in the treatment conditions of each method, including temperature, type of solvent, and extraction time.

3.4 Statistical Analysis

The effect of the extraction solvent and duration time was evaluated using statistical methods, specifically analysis of variance (ANOVA), as shown in Table 4. The analysis shows that extraction time significantly affects the yield, with an F-value of 12.29 and a P-value of 0.0196 ($P < 0.05$). This indicates that differences in extraction time have a meaningful impact on the outcome. In contrast, the type of solvent does not show a significant effect, with an F-value of 0.683 and a P-value of 0.5558 ($P > 0.05$). Therefore, it can be concluded that extraction time plays a more critical role

Table 3. The comparison of extraction methods in mango seed extract

Extraction Method	Extraction Time	Temperature (°C)	Solvent	Yield (%)	Reference
MAHD	30 minutes	100	Ethanol	40.342	Research result
	30 minutes	100	<i>n</i> -hexane	39.547	Research result
	30 minutes	100	Ethyl acetate	39.434	Research result
Sохhlet	8 hour	90	Ethanol	17.45	(Balacuit et al., 2021)
	4 hour	70	<i>n</i> -hexane	10.60	(Gurjar & Raj, 2022)
	4 hour	70	Ethyl acetate	11.03	(Sipra et al., 2023)
Maceration	8 hour	28	Ethanol	17.28	(Yoswathana & Eshiaghi, 2013)

in determining the extraction yield than the type of solvent used.

Table 4. ANOVA of extract yields

Source of Variation	SS	df	F	P-value	F crit
Solvent	28.221	2	0.683	0.556	6.944
Extraction time	508.048	2	12.291	0.019	6.944
Error	82.670	4			
Total	618.939	8			

*Significant if p-value < 0.05

4. CONCLUSION

The arumanis mango kernel was successfully extracted using the microwave-assisted hydrodistillation method. This study observed that solvent type and extraction time influenced the extraction yield. The results show that ethanol as the solvent with a 30-minute extraction time produced the highest yield of mango kernel oil, 40.342%. Furthermore, GC-MS analysis of the extract obtained using n-hexane with a 90-minute extraction time revealed that the dominant compound was 2-furancarboxaldehyde, 5-(hydroxymethyl) or hydroxymethyl furfuraldehyde (HMF) with a peak area of 12.437% and the most abundant fatty acid detected was butyric acid, with a peak area of 9.852%. These findings indicate that while ethanol is optimal for maximum oil yield, n-hexane can be useful for isolating specific compounds such as fatty acids and HMF.

5. ACKNOWLEDGEMENT

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