



## Research Article

# Essential Oil from Hibiscus Flowers through Advanced Microwave-Assisted Hydrodistillation and Conventional Hydrodistillation

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Received 7 July 2022; Revised 21 August 2022; Accepted 10 September 2022; Published 30 September 2022

Academic Editor: Romina Alina Marc (Vlaic)

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Due to the increased demand and importance of essential oils in medicinal applications, advanced essential oil extraction techniques have been employed. Both conventional hydrodistillation (HD) and microwave-assisted hydrodistillation (MAHD) were employed to extract the essential oils from the hibiscus flower. Extraction time and solvent polarity were the most critical factors. Scanning electron microscopy (SEM) was used to investigate the surface morphologies of raw powdered hibiscus flowers (not exposed to any pretreatment) and pretreated powdered hibiscus flowers (exposed to methanol absorption for 60 minutes prior to extraction). Extractive chemistry analysis utilizing Fourier transform infrared (FTIR) spectroscopy was performed on the volatile oil obtained by MAHD. Different peaks in the gas chromatography/mass spectrometry (GC/MS) analysis indicated the presence of thirty-seven different compositions. MAHD was more energy efficient, had higher yield production, and was environmentally friendly, reducing HD's overall carbon footprint by 40%. Oxygenated monoterpene, sesquiterpene, and sesquiterpene hydrocarbons were found in the hibiscus flower's crude extract. Moreover, the methanolic extract of *Hibiscus rosa-sinensis* has potent antioxidant properties. A hibiscus flower extract had scavenging activities of 51.2% at 0.2 mg/mL, 0.3% at 0.6 mg/mL, 0.8% at 1.0 mg/mL, and 68.5% at 1.2 mg/mL against DPPH free radicals. Therefore, the MAHD method is well-suited to extracting essential oils from hibiscus flowers, and the resulting oil has the potential to provide significant therapeutic advantages.

## 1. Introduction

Aromatic chemical compounds are found in essential oils, which are volatile industrial oils. Important chemical compounds in essential oils include phenols, hydrocarbons, aldehydes, ketones, alcohols, and esters [1]. Essential oils extracted from several plants can be purchased commercially [2]. A ton of research shows that even tiny amounts of essential oils can significantly impact biological activity [3, 4].

Numerous bioassays are frequently performed to detect the antioxidant from plant extracts such as flowers. Neoteric reports indicate an inverse connection between the consumption of antioxidant-rich foods and the incidence of human disease [5]. Therefore, the food industry heavily uses synthetic antioxidants such as butylated hydroxyanisole (BHA) and butylated hydroxytoluene (BHT), which may contribute to cancer development and liver toxicity [6]. Plants have been the basis of conventional medicine

worldwide [7–9]. Using currently known methodologies and techniques for finding natural antioxidants in plants has much potential. Many experts have discussed many ways in which these plants can be put to good use [10–12].

*Hibiscus rosa-sinensis* is a small, evergreen tree growing up to 1.5–3.0 m (5.0–10 ft) wide and 2.5–5.0 m (8.0–16 ft) tall, with shiny leaves and red flowers in summer. Different parts of this plant, such as leaves, roots, and flowers, have been known to possess medicinal features such as laxatives, aphrodisiacs, and contraceptives. *Hibiscus rosa-sinensis* is well-known in the *Malvaceae* plant family as an evergreen grassy plant.

Fatty acids, flavonoids, carbohydrates, proteins, and minerals are all found in hibiscus flowers [13]. The flowers have been studied for their anti-inflammatory, antihypertensive, hepatoprotective, anticancer, antidiabetic, antinociceptive, cytotoxicity, antibacterial, and antioxidant properties [14].

Techniques such as solvent extraction, steam distillation, hydrodistillation (HD), maceration, and expression are commonly used to extract essential oils from plants [15, 16]. Even though these methods are labeled as “traditional extraction procedures,” HD is utilized multiple times throughout [15, 17]. However, several disadvantages are associated with traditional techniques, such as damage to volatile compounds, high energy consumption, and long extraction time [17–19]. Extraction methods have evolved due to increase in extraction yield, decrease in extraction time, improvement in soil quality, and decrease in operating costs. Modern styles include pressurized solvent extraction, ultrasound-assisted extraction, supercritical fluid extraction, microwave-assisted extraction, and microwave-assisted hydrodistillation (MAHD) [15, 20]. There are many advantages of using microwave-assisted hydrodistillation, such as its low cost, and it is one of the modern and fast methods, and it is possible to get better yield from the extracted oils [21, 22].

Recent studies [23, 24] have shown that microwave ovens can be used to remove active plant components effectively. There is a close relationship between the dielectric constant of the solvent and the sample and the effectiveness of MAHD [25, 26]. The extraction process limits the examination of various components in plant material because traditional procedures are thermally hazardous, solvent-intensive, and time-consuming [27, 28]. This novel, promising MAHD method can potentially reduce solvent usage, protect thermolabile elements, and facilitate rapid, high-yield extraction.

Since MAHD allows for solvent selectivity, it can reduce extraction time and allows for precise temperature control, and it is widely employed as a method for chemical extraction. As it produces less carbon dioxide (CO<sub>2</sub>), MAHD is better for the environment [29]. Conventional methods for bioactive component extraction are laborious and lack precise temperature regulation [27]. Many bioactive components of plants have also been extracted using this method. Although it is more effective than traditional steam distillation [23, 30], its use depends on the solvent's dielectric constants and the sample [25].

Healing with MAHD relies on its direct effect on polar materials or solvents because this is where the action is most concentrated. Ionic conduction and the rotation of two dipoles drive this phenomenon [31]. Some of the many advantages of MAHD include targeted heating, higher output, sufficient temperature maintenance, fewer required process steps, quicker start-up, smaller equipment footprint, and lower temperature disparities. Moreover, unlike conventional oil, the oils produced through MAHD do not harm the planet [32]. Furthermore, there are scant studies on functional group analysis of the chemicals recovered from hibiscus flowers using MAHD or on the impact of pretreatment input materials. Furthermore, no reports compare the effectiveness of MAHD and the traditional HD method in extracting the oil from the hibiscus flower's functional groups. This study compares MAHD to HD to extract the volatile oils found in hibiscus blooms. The hibiscus flower was studied morphologically before and after pretreatment to determine the impact of the process. GC-MS was used to determine the chemical makeup of the sample. Parts of the compounds recovered by each method were compared, and FTIR analysis was used to look at structural changes in the functional groups of the components. The financial, energy, and ecological effects of MAHD and HD extraction were also studied.

## 2. Materials and Methods

**2.1. Materials.** The mature and fresh flowers of hibiscus were collected in February 2016 from a location in Gambang Campus, Universiti Malaysia Pahang, Malaysia. In addition, the ecosystem for planting the flower was the system accustomed to climate change; whether it is summer or winter, it grows throughout the year. It does not tolerate low temperatures below 10°C. As defined by the climate of Malaysia, temperatures do not reach this degree in winter. This study used chemicals exclusively for analysis to verify that the extracted essential oil is safe for the environment. A small number of chemicals utilized in this work, including dimethyl sulfoxide (DMSO), anhydrous sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>), and potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>), were supplied from Sigma-Aldrich (USA).

**2.2. Pretreatment.** Some dirt and sticky material, such as tiny sand grains, were found in the hibiscus flowers. The material was cleaned to eliminate harmful chemicals as much as possible. This method required fresh hibiscus flower samples, which were washed in distilled water for 60 minutes and then air-dried for 24 hours at temperature between 60 and 70°C. After that, the particle size of the sample was reduced by crushing and grinding about 70 g of dried hibiscus flower petals. Using a mechanical sieve shaker, 80 μm of hibiscus flower powder was obtained after grinding and sieving the samples. It was determined that drying the flower powder at 105 ± 5°C would provide a constant weight. The following formula determined the flower powder's moisture content:

$$\text{Moisture content (\%)} = \frac{W_1 - W_2}{W_1} \times 100, \quad (1)$$

$W_1$  and  $W_2$  are the weight of the flower powder before and after drying (g), respectively. The moisture content of the flower powder was about 22.2%.

The thoroughly dried hibiscus flower powder sample was kept as an indicator. Before extraction, 35 g of the powdered hibiscus flowers were weighed and presoaked for 1 h in distilled methanol at a ratio of 8:1 w/w of methanol to whole dried hibiscus flower powder, respectively [33]. All results were based on the weight of the dried samples.

**2.3. Scanning Electron Microscopy Analysis.** SEM images were taken at 60 and 120 minutes in MAHD and HD of untreated dried hibiscus flower powder, presoaked hibiscus flower powder, and hibiscus flower powder that has had its essential oils extracted. The TM3030 Plus tabletop microscope was used to examine the specimens. All samples were analyzed at an analytical working distance of 11.3 mm and an accelerating voltage of 5.0–15 kV (15–600,000 magnification). Electrical discharge was prevented by sputter coating prior to SEM observation. The powdered hibiscus flowers underwent SEM analysis to detect morphological alterations throughout the extraction process.

**2.4. Extraction of Hibiscus Oil via Microwave-Assisted Hydrodistillation.** The Clevenger-type equipment described in the literature [23] could be microwaved in a Milestone MWS Ethos E Solvent Extraction system (2.5 kW; 230 V-60 Hz; 2450 MHz). The microwave oven cavity was filled with a 1 L reactor (round bottom flask) holding 35 g of the powdered hibiscus flower matrix (which had been presoaked in methanol at a weight-to-weight ratio of 8:1 of methanol to dried hibiscus flower powder). The extracted oil was collected using the clenching equipment placed on the microwave outside. For a total of 120 minutes, the microwave was set to 400 W of power. When the oil in the flask evaporates, it leaves vapor behind. A condenser reduces the pressure of steam and the vapors of essential oils. Methanol and essential oil are combined in the condensate before being collected and separated using dichloromethane in a separating funnel.

**2.5. Extraction of Hibiscus Flower Oil through Hydrodistillation.** The essential oil of hibiscus flowers was extracted using a Clevenger machine and the HD method. The extraction was heated with an electromantle (Nahita model 655). HD extraction works similar to MAHD extraction. Nevertheless, what sets apart the various methods is the heat source used. To make a fair comparison between HD and MAHD, we used the same sample size (1.0 L) and concentration (35 g of powdered hibiscus flowers in the same volumes of methanol) for both extraction procedures. The conventional methods

require a longer extraction time to attain maximal oil recovery [23, 29], while this extraction was conducted at an operating power of 300 W for only 180 minutes. The collected condensate containing a mixture of methanol and essential oil is poured into the separating funnel with solvent dichloromethane.

**2.6. Gas Chromatography-Mass Spectrometry Analysis.** The analysis was carried out with a DB-WAX-fused silica column (30 m  $\times$  0.25 mm i.d. and film thickness 0.25  $\mu$ m) and Agilent 5975C Series GC/MS, Agilent, USA. The oven was set to 60°C for 10 minutes, then increased by 20°C every minute to 230°C, and finally maintained at 250°C for 10 minutes. In order to achieve a 30 cm/s linear velocity, helium was used as the carrier gas.

Both MAHD extract samples were diluted to a concentration of 3.0% in dichloromethane by mixing 1.0  $\mu$ L of pure essential oil of flowers with 10  $\mu$ L of dichloromethane. The split ratio was maintained when injecting the diluted samples into GC. Mass spectral data collected from the sample were compared to data obtained from pure, commercially available standards injected under identical conditions to identify the components.

Mass spectral data of essential oils from the National Institute of Standard and Technology (NIST MS collection) were compared to the components found in the extracted oil. The retention times of the chemicals found in the hibiscus flower oil were similar enough for a quantitative analysis to be performed using both techniques. The relative abundance of individual components in the purified hibiscus flower oil was determined by measuring the area under respective peaks. When estimating the size of each peak, the normalization approach was used, with 100% being the full extent of the peaks.

**2.7. Fourier Transform Infrared Spectroscopy.** Oil extracted using HD and MAHD was analyzed for functional groups. The purpose was to learn more about the bonding structures and alterations in the oil's chemical compositions that had taken place during the extraction process. For the analysis, we used a Thermo Scientific Nicolet iS5 FTIR spectrometer, made in USA by Thermo Scientific®. The FTIR spectrum was recorded between 4000 and 400  $\text{cm}^{-1}$ .

**2.8. Calculation of Energy Consumption.** The total amount of energy expended during MAHD and HD extraction of hibiscus flower oil was determined to assess its environmental effect. The formula for determining energy consumption for both approaches is shown in the following equation:

$$\text{Energy consumed (kWh)} = \frac{(\text{Power consumed for the extraction process} / \text{Extraction time})}{1000} \quad (2)$$

**2.9. Antioxidant (DPPH Radical Scavenging) Assay.** Methanol extract of hibiscus flower (Cass.) was tested for its ability to quench free radicals with 2,2-diphenyl-1-picrylhydrazyl (DPPH). 95% of methanol was used to make a DPPH solution (0.004% w/v). The investigated medication was prepared in test tubes with five distinct concentrations. We added 1.0 mL of newly produced DPPH reagent to the test tubes and left them to incubate overnight in the dark. The absorbance was checked at 517 nm after incubating the sample for 10 minutes (Systronics UV: Visible spectrophotometer, USA). The positive control used in this process was ascorbic acid.

Scavenging activity (%) of the DPPH free radical was measured using the following equation [34]:

$$\text{Inhibition (\%)} = \left( \frac{A_0 - A_1}{A_0} \right) * 100, \quad (3)$$

where  $A_0$  is the absorbance of the control and  $A_1$  is the absorbance of the test.

### 3. Results and Discussion

The yield of flower oil can be affected by variables in the extraction process [35]. These variables include the solvent ratio, extraction power, and extraction time. Previous studies have looked into how different extraction settings affect the effectiveness of HD and MAHD procedures. Results from those analyses provided insights into the selection of extraction settings for MAHD and HD that yielded the best overall performance.

#### 3.1. Scanning Electron Microscopy Analysis of Hibiscus Flower Powder

**3.1.1. Surface Morphology of Untreated and Pretreated Hibiscus Flower Powder.** Figures 1(a) and 1(b) illustrate the surface morphologies of raw powdered hibiscus flower (without soaking in methanol before extraction) and pretreated hibiscus flower (soaked in methanol for 60 min). The raw powdered flower shown in Figure 1(a) is slender and dry. Conversely, the powdered hibiscus flower undergoes discernible physical changes after being pretreated. As shown in Figure 1(b), when the hibiscus flower is dried and ground into a powder, it has an enlarged and puffy appearance. The hibiscus flower swelled up after soaking it in methanol, which suggests that the plant had taken in the solvent. This would increase tissue swelling and subsequent rupture, allowing the volatile oils to flow more easily into the methanol during extraction [32].

**3.1.2. Morphological Variations in Hibiscus Flowers following MAHD and HD Extraction.** As shown in Figure 2(a), the oil glands in the powder have a strong propensity to swell and expand upon soaking. This could aid in refining oil extraction by facilitating the clean and rapid release of oil from the substance. However, if the material is extracted without soaking, contractile oil glands will be subjected to greater stress, resulting in less oil being released. The internal

pressure of the oil glands would need to be raised to a higher level before rupturing for oil emancipation to be possible. Soaking plant materials before extracting essential oils is recommended so that the induction time and overall energy usage can be shortened.

Figure 2(b) shows the oil gland following MAHD extraction. In contrast, Figure 2(c) shows an SEM image of the oil gland after HD extraction. The oil gland appears to have experienced different types of disruption (highlighted in red) in both the images. The oil gland is damaged in MAHD, which may be related to how the oil is heated. Methanol's high dielectric qualities allow it to absorb and transform microwave radiation into heat which is then delivered to the plant material in microwave-assisted hydrodistillation (MAHD) [36]. Because of this, heat energy can be focused precisely where the oil glands are located, allowing for the most efficient and least invasive method of oil release possible (Figure 2(b)). In the case of HD, however, the gland shows signs of a particularly severe fracture (Figure 2(c)). The high mechanical strain on the oil glands may be a side effect of the HD technique, suggesting that an aggressive attitude accompanies it. This is because the heat energy from HD first reaches the solvent surface through conduction and convection before heating the intended plant material [36]. This would cause the glands to explode, allowing the oil to be extracted. Based on what is shown in the SEM image [37], the gland ruptures violently when HD is extracted could be explained.

**3.2. Analysis of the Chemical Structure of an Essential Oil.** Figure 3 shows the FTIR spectra of hibiscus flower oils acquired using MAHD and HD. These spectra show that the absorption spectra of various oil components overlap. This is because volatile oils are such a complicated mixture. Since the peaks for the distinctive fingerprint of hibiscus flower oil are located in the region from 4000 to 400  $\text{cm}^{-1}$ , their spectrum is highly instructive.

Essential oil functional groups were identified by comparing the FT-IR spectrophotometer's sample vibration frequencies in wave numbers to those listed on an IR correlation chart. The FT-IR spectrum of the essential oil from hibiscus flowers showed the presence of O-H stretch for alcohol and phenol in the absorption band of frequency at 3052  $\text{cm}^{-1}$ . Aldehyde was detected thanks to an absorbance band at 1629  $\text{cm}^{-1}$  (m). The aromatic components' C=C skeletal vibration gives rise to the peak at 1420  $\text{cm}^{-1}$ . Specifically, ring stretching is responsible for the 1264  $\text{cm}^{-1}$  peak. The peak at 1057  $\text{cm}^{-1}$  represents the C-H stretching vibration. Vibrational bending absorption of C-N groups is responsible for the 895  $\text{cm}^{-1}$  peak. The absorption peak at 736  $\text{cm}^{-1}$  reflects the nitro group's vibrations. Table 1 provides a visual summary of the significant peaks.

The spectra of oils extracted using these two processes are typically very close to one another. These oils' complexity and the similarity in their chemical fingerprints make it challenging to identify their distinguishing features. This demonstrates that MAHD may be used to safely extract essential oils from hibiscus flowers without altering the oil's chemical composition.

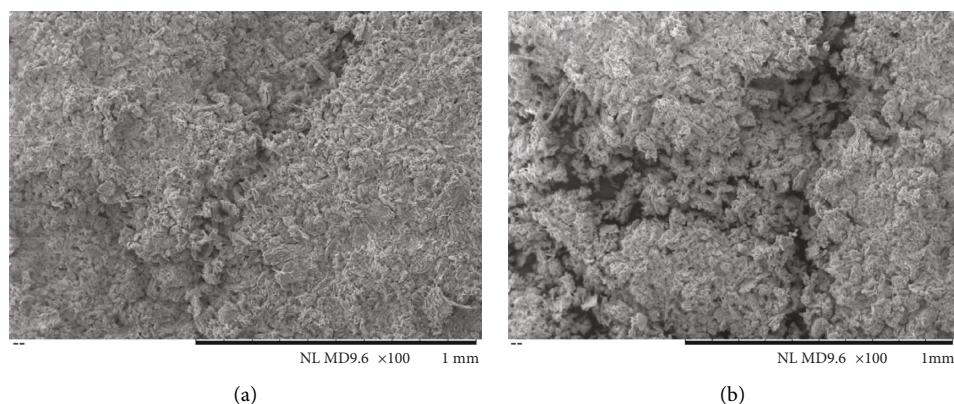


FIGURE 1: SEM images of (a) untreated raw hibiscus flower powder and (b) pretreated hibiscus flower powder after being soaked for 60 min.

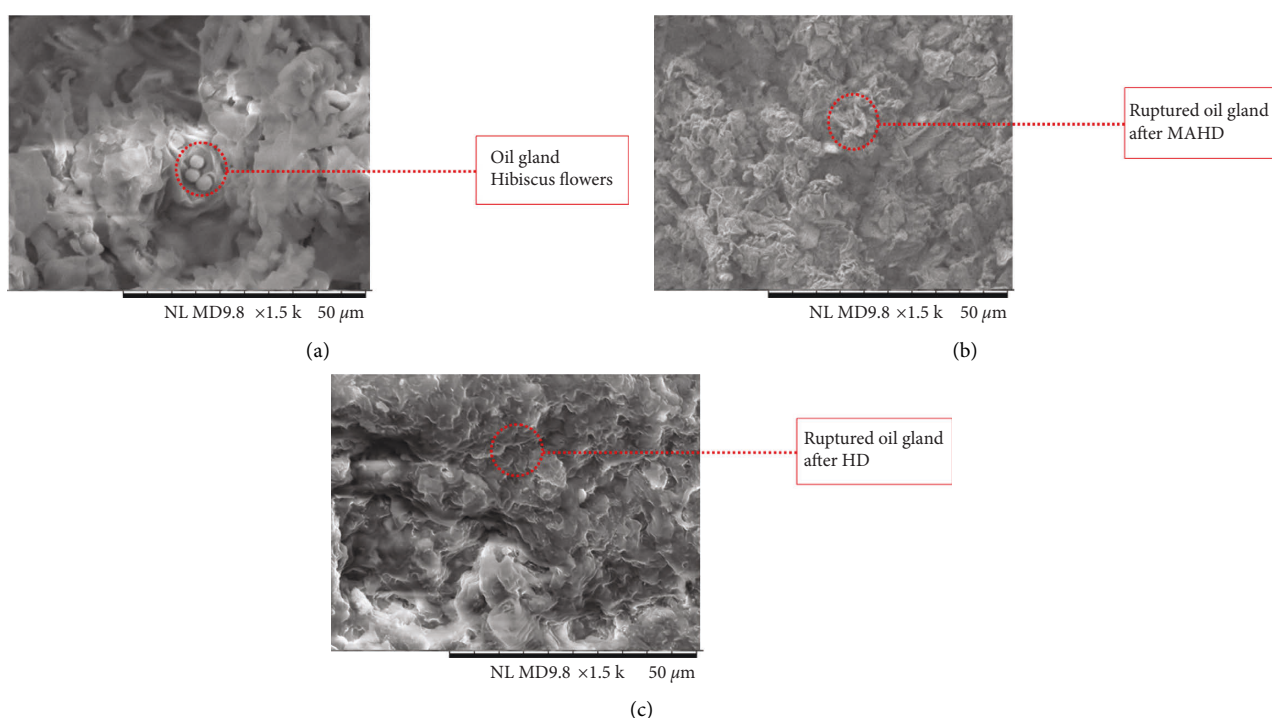


FIGURE 2: SEM images of oil cell glands of hibiscus flower (a) after pretreatment (soaking for 60 min), (b) after MAHD extraction (120 min), and (c) after HD extraction (160 min).

TABLE 1: Details of hibiscus flower oil functional groups derived from HD and MAHD.

Functional group representation	Vibration assignment ( $\text{cm}^{-1}$ )	
	HD	MAHD
O-H	3052.76	3053.10
C=O stretching	1629.29	1629.29
C=C stretching	1420.77	1420.94
Ring stretching	1264.18	1264.42
C-H wagging	1057.00	1019.85
C-N bending	895.36	895.56
NO <sub>2</sub> wagging	736.08	735.70

3.3. *Compositional Examination of Chemical Compounds in Hibiscus Flower Oil Obtained by MAHD and HD.* Essential oils are complex substances that may even contain oxygenated molecules. For hibiscus flowers, the essential oils

include only three elements: carbon, hydrogen, and oxygen. Terpenes (primarily monoterpenes and sesquiterpenes), phenolics, and alcohols are just a few of the volatile compounds found in these mixtures [29]. Aldehydes, ketones, acids, ethers, and esters are oxygenated derivatives of hydrocarbon terpenes [38]. In the treatment of cardiovascular illness [39], such as malaria [40] and cancer [41], several terpenes have proven to be highly effective medications.

The MAHD and HD extraction procedures obtained the crude extract of hibiscus flowers, and both were compared and evaluated for their chemical components and quality. The chemical components of MAHD and HD-obtained crude extract (%) of hibiscus flower oil are compared in Table 2. The table shows the results of a direct comparison between the percentage of chemical compounds in crude

TABLE 2: The average chemical composition of compounds in hibiscus flower oil.

Types	Compounds	Molecular formula	MW	Mass percentage of chemical compounds in crude extract (%)	
				MAHD	HD
Aldehyde	2,3-Dihydroxy propanal	C <sub>3</sub> H <sub>6</sub> O <sub>3</sub>	90.10	12.58 ± 0.1	10.34 ± 0.1
	Acetaldehyde	C <sub>2</sub> H <sub>4</sub> O	44.04	0.62 ± 0.1	0.53 ± 0.1
	1,4-Butanediol	C <sub>4</sub> H <sub>6</sub> O <sub>2</sub>	86.10	1.65 ± 0.2	1.43 ± 0.1
	Bicyclo[4.1.0]heptane-7-carbaldehyde	C <sub>8</sub> H <sub>12</sub> O	124.2	2.80 ± 0.1	3.50 ± 0.1
	1-Isopropyl diaziridine	C <sub>4</sub> H <sub>10</sub> N <sub>2</sub>	86.13	0.75 ± 0.1	0.92 ± 0.1
Amine	Ethylenediamine	C <sub>2</sub> H <sub>8</sub> N <sub>2</sub>	60.10	6.71 ± 0.1	5.42 ± 0.2
	1,3,5-Triazine-2,4,6-triamine	C <sub>3</sub> H <sub>6</sub> N <sub>6</sub>	126.12	2.48 ± 0.1	2.32 ± 0.1
	Methylguanidine	C <sub>2</sub> H <sub>7</sub> N <sub>3</sub>	73.10	0.73 ± 0.1	0.83 ± 0.3
	2-Butanamine, (S)-	C <sub>4</sub> H <sub>11</sub> N	73.13	2.72 ± 0.1	2.53 ± 0.2
	N-Formyl-β-alanine	C <sub>4</sub> H <sub>7</sub> NO <sub>3</sub>	117.10	2.36 ± 0.1	2.22 ± 0.1
	2-Methyl-piperazine	C <sub>5</sub> H <sub>12</sub> N <sub>2</sub>	100.20	0.80 ± 0.1	0.69 ± 0.1
	N-ethyl-propanamide	C <sub>5</sub> H <sub>11</sub> NO	101.20	10.69 ± 0.1	9.89 ± 0.1
Amide	2-Methyl- propanamide	C <sub>4</sub> H <sub>9</sub> NO	87.12	0.58 ± 0.1	0.74 ± 0.2
	1-(2-Adamantylidene)semicarbazide	C <sub>11</sub> H <sub>17</sub> N <sub>3</sub> O	207.30	0.68 ± 0.2	0.47 ± 0.2
Alcohol	2-Propen-1-ol	C <sub>3</sub> H <sub>6</sub> O	58.10	0.98 ± 0.1	1.00 ± 0.2
	Tetrahydro-, trans-3,4-furandiol	C <sub>4</sub> H <sub>8</sub> O <sub>3</sub>	104.10	0.91 ± 0.2	0.76 ± 0.1
	3-Piperidinol	C <sub>5</sub> H <sub>11</sub> NO	101.14	0.28 ± 0.1	0.45 ± 0.1
	1,5-Pentanediol	C <sub>5</sub> H <sub>12</sub> O <sub>2</sub>	104.15	0.48 ± 0.1	0.54 ± 0.2
	2-Methyl-1-propanol	C <sub>4</sub> H <sub>10</sub> O	74.12	1.57 ± 0.1	1.32 ± 0.1
Alkyne	(Z)6,(Z)9-pentadecadien-1-ol	C <sub>15</sub> H <sub>28</sub> O	224.40	1.70 ± 0.1	1.90 ± 0.1
	1-Octyne	C <sub>8</sub> H <sub>14</sub>	110.20	0.45 ± 0.1	0.62 ± 0.2
Acid	Succinamic acid	C <sub>4</sub> H <sub>7</sub> NO <sub>3</sub>	117.10	0.66 ± 0.1	0.58 ± 0.1
	Thioacetic acid	C <sub>2</sub> H <sub>4</sub> OS	76.12	1.08 ± 0.1	1.10 ± 0.2
	Citramalic acid	C <sub>5</sub> H <sub>8</sub> O <sub>5</sub>	148.10	0.36 ± 0.1	0.50 ± 0.2
Ester	Dodecahydropyrido[1,2-b]isoquinolin-6-one	C <sub>13</sub> H <sub>21</sub> NO	207.30	0.35 ± 0.1	0.23 ± 0.2
	Ethanimidic acid, ethyl ester	C <sub>4</sub> H <sub>9</sub> NO	87.12	31.43 ± 0.2	29.23 ± 0.2
	Carbamic acid, ethylnitroso-, ethyl ester	C <sub>5</sub> H <sub>10</sub> N <sub>2</sub> O <sub>3</sub>	146.14	0.82 ± 0.1	0.90 ± 0.1
	Propanedioic acid, oxo-, bis(2-methylpropyl) ester	C <sub>11</sub> H <sub>18</sub> O <sub>5</sub>	230.25	0.08 ± 0.1	—
	o-Methylisourea hydrogen sulfate	C <sub>2</sub> H <sub>8</sub> N <sub>2</sub> O <sub>5</sub> S	172.20	4.06 ± 0.1	3.90 ± 0.2
	6-Acetyl-β-d-mannose	C <sub>8</sub> H <sub>14</sub> O <sub>7</sub>	222.20	0.08 ± 0.1	—
	Hexadecanoic acid, methyl ester	C <sub>17</sub> H <sub>34</sub> O <sub>2</sub>	270.45	2.99 ± 0.1	3.01 ± 0.1
Ether	Decanoic acid, ethyl ester	C <sub>12</sub> H <sub>24</sub> O	200.32	0.77 ± 0.1	0.98 ± 0.1
	Trimethyl-oxirane	C <sub>5</sub> H <sub>10</sub> O	86.13	0.78 ± 0.1	0.90 ± 0.1
	1-(3-Methyloxiranyl)-ethanone	C <sub>5</sub> H <sub>8</sub> O <sub>2</sub>	100.11	0.18 ± 0.1	0.20 ± 0.1
Other	Ethoxy-ethene	C <sub>4</sub> H <sub>8</sub> O	72.10	3.63 ± 0.2	3.49 ± 0.1
	Isothiazole	C <sub>3</sub> H <sub>3</sub> NS	85.12	0.17 ± 0.1	0.21 ± 0.2
	2,3-Dioxabicyclo[2.2.1] heptane	C <sub>5</sub> H <sub>8</sub> O <sub>2</sub>	100.10	0.07 ± 0.1	—

(-) not detected, <sup>a</sup>mean, and ± standard deviation.

extractants under ideal conditions and the marker components, ethanimidic acid and ethyl ester. Under ideal conditions, the median concentrations of ethanimidic acid and ethyl ester in MAHD and HD were 31.43 and 29.23%, respectively. Certain chemical families were found in the crude extract of hibiscus flowers. These include oxygenated monoterpenes, sesquiterpene hydrocarbons, and other oxygenated chemicals. Hemiterpenes (C<sub>5</sub>) and sesquiterpenes (C<sub>15</sub>) are the most common chemicals in essential oils. Monoterpenes and sesquiterpenes are said to be “oxygenated” or “oxygenated” when an oxygen molecule is incorporated into their molecular structure. Our findings corroborate previous findings [42–46]. The crude extract percentages, however, were not the same between the two extraction strategies. MAHD could be suggested as a useful approach for extracting more oxygenated chemicals from hibiscus flowers. However, the total percentage of essential

oil acquired from MAHD and HD might be very close. MAHD resulted in a more significant proportion of oxygenated compounds and a smaller proportion of sesquiterpene hydrocarbon than HD.

As a result, MAHD-obtained compounds are pretty helpful. These chemicals’ high aromatherapy characteristics and high toxicity to insects and pests make them indispensable to pharmaceutical and other sectors [47]. As a result, the constituents are less likely to undergo partial degradation typical of HD extractions, which can be associated with a shorter extraction time [48]. This suggests that the essential oil extracted from hibiscus flowers using MAHD has excellent therapeutic potential.

**3.4. Energy, Economy, and Environmental Impact.** In light of the data presented here, it can be said that the MAHD method of hibiscus flower oil extraction is superior to the

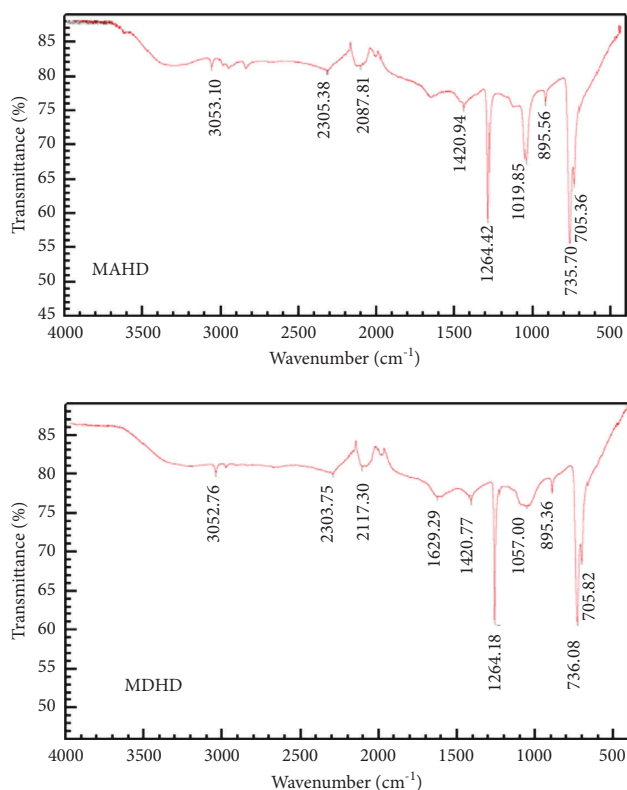


FIGURE 3: FTIR spectrum of essential oil for hibiscus flower through MAHD and HD.

HD method (conventional). This work aims to compare and contrast the two methods by considering their effects on the natural world, the economy, and the energy supply. This is essential for foreseeing the commercial viability of the MAHD extraction process.

In order to reach the boiling point of the plant matrix during HD extraction, 280 mL of methanol containing 35 g of hibiscus powder had to be heated for roughly 15 minutes [35]. The hibiscus oil was recovered entirely after 160 minutes of the HD procedure. Amazingly, the MAHD technique only needed 8.0 minutes to reach its induction time at 300 W and start making hibiscus oil. The oil from the hibiscus flowers was retrieved entirely in less than an hour. Table 3 shows how two extraction strategies stack up regarding energy use and CO<sub>2</sub> emissions. The energy consumption calculations show that 0.639 kWh of energy was used for MAHD extraction, and 1.087 kWh was needed for HD extraction. The wattmeter in the microwave and the power cord for the electric heater was used to calculate power consumption. As shown in Table 3, HD requires more energy to produce essential oil, and its energy requirements are more than MAHD. Noticeably, the yield produced by MAHD (1.25%) was higher than that obtained from HD (1.15%). In addition, MAHD consumed less energy than HD. These results were better than those obtained for essential oil yield from *Sideritis raeseri*, where the product was found to be 0.61–0.67% using microwave-assisted distillation [49]. In addition, the current work yield was higher than that obtained by *Hibiscus sabdariffa* L. using different methods,

TABLE 3: Summary of energy consumption and CO<sub>2</sub> emission of MAHD and HD methods.

Parameters	MAHD	HD
Total operating time (min)	128	181
Electricity consumption (kWh)	0.639	1.087
CO <sub>2</sub> released (g)	511.2	869.6
Yield (%)	1.25	1.15

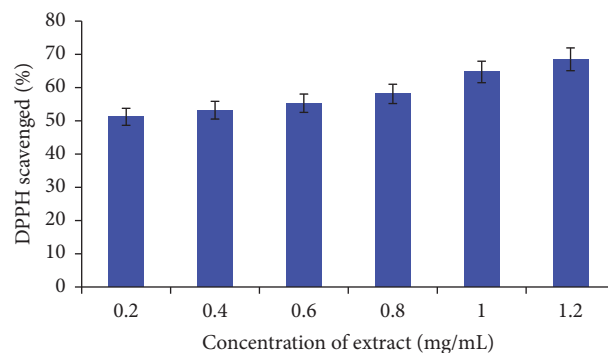


FIGURE 4: DPPH radical scavenging activity of essential oil and the methanol extract of hibiscus flower and the IC<sub>50</sub> of essential oil 0.7 mg/mL.

where it was 0.86, 0.54, and 1.10% using HD, steam distillation, and solvent-free microwave-assisted extraction [50]. Concerning the environmental impact, the amount of carbon dioxide released to the environment is higher in HD (869.6 g CO<sub>2</sub>/g of hibiscus flower oil) than that in MAHD (511.2 g CO<sub>2</sub>/g of hibiscus flower oil). According to [37], 1 kWh energy consumption from coal or fuel releases almost 800 g of carbon dioxide into the environment during combustion. The calculation of energy consumption was carried out using equation (2).

From these findings, we deduce that the MAHD method is a green extraction process that benefits the natural world. MAHD is a solvent- and energy-efficient method that does not require chemicals to extract high-quality essential oil.

### 3.5. In Vitro Antioxidant Assay

**3.5.1. DPPH Radical Scavenging Assay.** Antioxidants are expected to neutralize DPPH radicals because of their hydrogen-donating capacity. Radical scavenging actions are essential to prevent many illnesses, including cancer [51]. Extracts of hibiscus flowers are commonly tested for their antioxidant capacity using the DPPH assay. Figure 4, shows the DPPH antioxidant capability of a hibiscus flower extract. The DPPH free radical scavenging activity of a hibiscus flower extract was 51.2%, 53.2%, 55.3%, 58.1%, 64.7%, and 68.5% at concentrations of 0.2, 0.4, 0.6, 0.8, 1.0, and 1.2 mg/mL, respectively. Scavenging activity for DPPH is shown in Figure 4 and Table 4. These results demonstrate that higher quantities of the extract have higher radical scavenging activity (see Figure 4). The IC<sub>50</sub> for essential oil in the DPPH assay method was 0.7 mg/ml (Table 4). Methanol extract of hibiscus flower has a scavenging action comparable to ascorbic acid [52].

TABLE 4: DPPH assay of methanol extract of hibiscus flower.

No.	Concentration of extract (mg/mL)	DPPH scavenged (%)	IC <sub>50</sub> (mg/mL)
1	0.2	51.2	
2	0.4	53.2	
3	0.6	55.3	
4	0.8	58.1	0.7
5	1.0	64.7	
6	1.2	68.5	
7	Ascorbic acid (5 mg/mL)	81.85	

#### 4. Conclusion

Hibiscus flowers were used to create an eco-friendly essential oil using the modern green extraction methodology MAHD and the traditional HD method. The characteristics of hibiscus flower essential oils extracted using MAHD and HD techniques were compared. A scanning electron microscopy (SEM) study of powdered hibiscus flowers that had previously undergone oil extraction found that MAHD yielded the purest oil with minimally damaged sebaceous glands. FTIR testing confirmed that MAHD contained the same chemical constituents as that of the oil recovered from hibiscus flowers using HD. Based on the data, it is clear that the MAHD method is superior in terms of cost-effectiveness, environmental friendliness, and the number of oxygenated compounds it generates. The MAHD-obtained hibiscus flower raw material was put through the DPPH radical scavenging assay to determine its antioxidant quality. The MAHD crude extract has an intriguing IC<sub>50</sub> value of 0.7 ppm. These findings demonstrate that MAHD crude extracts have more significant therapeutic potential. The MAHD procedure yielded higher quality hibiscus flower oil than the conventional HD method. This study reveals that MAHD may be used to successfully extract volatile oils from hibiscus flowers without disrupting their original chemical structures. Due to its high yield and low energy consumption, MAHD proved to be an eco-friendly option for separating essential oils compared to HD.

#### Data Availability

The data used to support the findings of this study are included within the article.

#### Conflicts of Interest

The authors declare that they have no conflicts of interest.

#### Acknowledgments

The authors would like to thank Universiti Malaysia Pahang for assisting with this study under the research (UIC 190806). In addition, M.A. Assiri extends his appreciation to the Deanship of Scientific Research at King Khalid University for funding this work through the research group project under grant number (KKU/RCAMS/G015-21).

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