Original Artikel
Received Agustus 2022 / Revised September 2022 / Accepted September 2022
Spizaetus: Jurnal Biologi dan Pendidikan Biologi
p-ISSN: 2716-151X e-ISSN: 2722-869X

Metode Ekstraksi dan Bioaktivitas Minyak Atsiri dari Daun Kesum (*Persicaria odorata*): Sebuah Tinjauan Singkat

Extraction Methods and Bioactivity of Essential Oils from *Kesum* Leaves (*Persicaria odorata*): A Short Review

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Abstrak. *Persicaria odorata* (Synonym of *Polygonatum odoratum* and *Polygonum minus*) leaves, locally known as *kesum* leaves, is one of Indonesia's biodiversity species, particularly on Kalimantan Island, and it is contained a high concentration of essential oils. The extraction of essential oil from *kesum* leaves commonly uses various methods, including solvent extraction, steam distillation, hydro-distillation, supercritical fluid extraction, microwave-assisted extraction, ultrasonic-assisted extraction, and so on, with various extract characteristics produced. Several studies reported that *kesum* leaves essential oil has numerous advantages, including anti-bacterial, hepatoprotective, anti-tyrosinase, antioxidant properties, and so on. Future research will require additional and new techniques, particularly non-thermal extraction technology and other bioactivity tests, to improve yield and maintain the essential oil composition of *kesum* leaves.

Kata Kunci: bioactivity; essential oil; extraction; *kesum* leaves

DOI: http://dx.doi.org/10.55241/spibio.v3i3.82

1. Introduction

Kesum plant or *Persicaria odorata* (Synonym of *Polygonatum odoratum* and *Polygonum minus*) is the native plant in South East Asia [1]. This plant is widely growing in Kalimantan Island, including Indonesia and Malaysia. The *kesum* leaves are an essential part of this plant. Commonly, it is used as a herb or seasoning in various unique dishes, especially in "*bubur padas*", a famous dish from West Kalimantan Indonesia [2]. *kesum* leaves are rich in essential oil, such as decodecanal, decanal, 1-decanol, (E)-Caryophyllene, Isobornyl acetate, 1-Dodecanol, α-Caryophyllene, and other minor

essential oil [3]–[8]. Several previous studies reported that the essential oil in *kesum* leaves is effective as an anti-bacterial [4], [9], [10], hepatoprotector [11], [12], anti-tyrosinase [13], [14], and antioxidant [15], [16], both in *vivo* or in *vitro* studies.

The extraction of essential oil from *kesum* leaves can be done by several methods, from simple to advanced methods, such as hydro-distillation [3], [4], [9], [12], [17], [18], steam distillation [5], [6], [19], supercritical fluid extraction (SFE) [7], [20], solvent extraction ,microwave-assisted extraction (MAE), and ultrasonic-assisted extraction (UAE) [8]. Each method had different factor parameters, advantages, and limitations in the extraction of essential oil of *kesum* leaves and also affected the final product, namely yield and essential oil composition. However, until now, the author has not found a review that discusses the method of extracting essential oils from *kesum* leaves and their bioactivity properties. So, on this occasion, the author briefly reviews several methods used to extract essential oils from *kesum* leaves and their bioactivity properties *in vivo* and *in vitro* through secondary data.

2. Method

This type of research is a literature study [21]. The material used in this review paper comes from research papers that have been reported by previous researchers from around the world, which is obtained from open access sources, such as Google Scholar (scholar.google.com) and Crossref (https://www.crossref.org/), related to the topics discussed, which are the extraction methods of *kesum* leaves essential oil [3]–[6], [8], [9], [12], [17]–[19] and their bioactivity [4], [9]–[16].

3. Results and Discussion

3.1. *Kesum* leaves and Its Essential Oil Composition

The *kesum plant* or its scientific name is *Persicaria odorata* (Synonym of *Polygonatum odoratum* and *Polygonum minus*), is the native plant in South East Asia [1]. *Kesum* plant widely used is the leaf known as the *kesum* leaves. In Malaysia and Indonesia, *kesum* leaves are used as herbs and seasoning for various unique dishes because it gives food a delicious aroma and taste [3], [19], [22]. The main components of *kesum* leaves are essential oil (72.54%)[3], a secondary metabolite that functions as a bioactive compound in the medical field [15]. Commonly, the main active components of *kesum* leaves essential oil are dominated by decodecanal, decanal, 1-decanol, (E)-Caryophyllene, Isobornyl acetate, 1-Dodecanol, α-Caryophyllene, and trace compounds [3]–[8], and its influenced by the extraction method. The comparison of *kesum* leaves essential oil composition influenced by the extraction method is summarized in Table 1.

Table 1. The *kesum* leaves essential oil composition is affected by extraction methods

Bioactive Compound	Extraction Methods and its Essential Composition Content (%)				
	HD [3], [4]	**SD** [5], [6]	**MAE** [8]	**UAE** [8]	**HRE** [8]
Hexanal	0.05	n.d		n.d	n.d
1-Hexanol	0.09	n.d	1.61	n.d	n.d
α-Pinene	0.39	0.61	1.16	n.d	n.d
Undecane	0.41 -2.52	0.82	2.46	2.46	n.d
Nonanal	0.15 -0.26	0.30-0.86	1.87	1.87	n.d
1-Nonanol	0.05 -0.35	0.76-1.40	1.02	n.d	n.d
1-Nonene	n.d	tr	n.d	n.d	n.d
Nonane	n.d	0.32	n.d	n.d	n.d
Decanal	16.263 -18.40	24.36-26.60	2.01	n.d	n.d
Undecanal	0.14 -1.37	0.75- 1.77	1.01	1.01	n.d
1-Decanol	5.37-12.68	2.49- 2.58	16.29	16.29	n.d
1-undecanol	1.16	1.41	n.d	n.d	n.d
1-decene	n.d	tr	n.d	n.d	n.d
Decane	n.d	tr	n.d	n.d	n.d
Isobornyl acetate	2.39	n.d	15.13	15.13	n.d
n-Decanoic acid	0.52	n.d	n.d	n.d	n.d
α-Cubebene	0.37	n.d	3.72	n.d	n.d
Xanthorrhizol	0.10	n.d	2.53	n.d	n.d
(-)-α -Panasinsene	0.27	n.d	1.74	n.d	n.d
Dodecanal	37.08 -43.47	33.60-48.18	1.36	1.36	n.d
(*E*)-Caryophyllene	3.83	n.d	43.29	43.29	n.d
α-Bisabolol	0.05 -0.06	n.d	n.d	n.d	n.d
Farnesene	0.18	n.d	0.06	n.d	n.d
α-Caryophyllene	1.02	n.d	0.57	n.d	0.57
1-Dodecanol	1.19 -4.81	2.44-4.00	n.d	n.d	n.d
β-Himachalene	0.48	n.d	0.58	0.58	n.d
α-Selinene	0.15	n.d	0.51	n.d	0.51
7-*epi*- α-Selinene	0.54	n.d	n.d	n.d	n.d
Selina-4.11-diene	0.23	n.d	n.d	n.d	n.d
Valencene	0.04 -0.32	n.d	0.63	n.d	n.d

Table 1. *Continued*

Bioactive Compound	Extraction Methods and its Essential Composition Content (%)				
	HD [3], [4]	**SD** [5], [6]	**MAE** [8]	**UAE** [8]	**HRE** [8]
Phylloquinone	n.d	n.d	3.78	n.d	n.d
δ -Cadinine	0.19	n.d	1.08	n.d	n.d
Alloaromadendrene	0.06	n.d	0.71	n.d	0.71
α-Curcumene	0.18 -0.23	1.46	2.56	n.d	2.56
cis -Lanceol	0.27	n.d	2.02	n.d	n.d
Farnesol	0.14	n.d	0.90	n.d	n.d
Humulene	0.13 -4.50	tr	n.d	n.d	n.d
Nerolidol	0.24	1.76	1.02-5.40	n.d	n.d
Dodecanoic acid	0.23		1.27	n.d	n.d
Decanoic acid	n.d	0.98	0.70-2.02	n.d	n.d
Hexadecanoic acid	0.44	n.d	n.d	n.d	n.d
β-Bisabolol	0.34	n.d	0.70	n.d	n.d
β-bisabolene	0.07	n.d	n.d	n.d	n.d
β-Caryophyllene oxide	0.35	1.80	1.02	n.d	n.d
β-Curcumene	0.08	n.d	n.d	n.d	n.d
β-Caryophyllene	n.d	0.18 -2.33	n.d	n.d	n.d
β-Cyclocitral	0.04	n.d	n.d	n.d	n.d
β-Selinene	0.40	n.d	n.d	n.d	n.d
β-Nerolidol	0.53	n.d	n.d	n.d	n.d
Caryophyllene oxide	1.42	n.d	n.d	n.d	n.d
Caryophylla-4(12).8(13)-dien-5-ol	0.69	n.d	n.d	n.d	n.d
Citronellol	n.d	n.d	n.d	n.d	n.d
trans-α- (*Z*)-Bergamotol	0.05 -0.13	n.d	2.00	n.d	n.d
Trans- α- (*Z*)-Bergamotene	0.25 -0.49	1.61	n.d	n.d	n.d
Isogermacrene D	0.08	n.d	n.d	n.d	n.d
Tetradecanal	0.10 -0.26	1.42 - 1.56	1.50 -2.35	1.50	1.50
Alloaromadendrene oxide-(1)	0.31	n.d	1.09	n.d	n.d
trans- Longipinocarveol	0.28	n.d	0.80	n.d	n.d
Neoisolongifolene, 8-bromo-	3.09	n.d	1.57	n.d	n.d
iso-Caryophyllene	0.08 -3.88	n.d	3.02	n.d	n.d

Table 1. *Continued*

Bioactive Compound	Extraction Methods and its Essential Composition Content (%)				
	HD [3], [4]	**SD** [5], [6]	**MAE** [8]	**UAE** [8]	**HRE** [8]
Drimenol	1.24-2.01	n.d	0.69	n.d	n.d
Drimenin	0.28 -0.30	n.d	1.14	n.d	n.d
Phytol	0.13	n.d	n.d	n.d	n.d
Limonene	n.d	tr	n.d	n.d	n.d
Trans-Ocimene	n.d	tr	n.d	n.d	n.d
7-*epi*-sesqiuthujene	0.52	n.d	n.d	n.d	n.d
γ-gurjunene	0.30	n.d	n.d	n.d	n.d
cis-sesquisabinene hydrate	0.96	n.d	n.d	n.d	n.d
Humulene epoxide I	1.09	n.d	n.d	n.d	n.d
Phytone	0.38	n.d	n.d	n.d	n.d
2-pentyl-furan	0.06	n.d	n.d	n.d	n.d
6-methyl-Hept-5-en-2-ol	0.03	n.d	n.d	n.d	n.d
Intermedeol	0.13	n.d	n.d	n.d	n.d
Linoleoyl chloride	0.16	n.d	n.d	n.d	n.d
n-dodecenylsuccinic anhydride	0.29	n.d	n.d	n.d	n.d

Notes: HD: Hydrodistillation, SD: Steam Distillation, MAE: Microwave Assisted Extraction, UAE: Ultrasonic Assisted Extraction, HRE: heat reflux extraction, n.d: not detected, tr: trace < 0.1%

3.2. Extraction Methods of Essential Oil from *Kesum* Leaves

The extraction of essential oil from *kesum* leaves can be carried out using several methods: hydro-distillation, steam distillation, supercritical fluid extraction (SFE), microwave-assisted extraction (MAE), ultrasonic-assisted extraction (UAE), and solvent extraction. Further description of each method is as follows:

1. Hydrodistillation

Hydrodistillation is the oldest and easiest method for extracting essential oils from medicinal and aromatic plants. The principle of this method is isotropic distillation. Extraction of essential oils with this method is done by heating a mixture of solvents (usually water or other solvents) and plant materials to evaporate and continue by liquefaction using a condenser. The setup also comprises a condenser and a decanter to collect the condensate and separate essential oils from water. The extraction method can be used for small and large-scale production [23]. Extraction of essential oils from *kesum* leaves, fresh and dry leaves, usually takes a certain time (4 -12 hours) using a Clevenger-type apparatus. The essential oils were collected over water, separated, dried over anhydrous sodium sulfate or nitrogen, and stored in the dark at 4 C for prior analysis [3], [4], [9], [12], [17], [18]. The yield of

essential oil from fresh *kesum* leaves is 0.274% - 0.475% [9], [18], and the yield of dried *kesum* leaves ranges from 0.12% - 0.41% [4], [9].

2. Steam Distillation

Steam distillation is the most widely used method for extracting essential oils from plants. The distillation system is a process for plant materials generated outside the still in a stand-alone boiler. The plant material is placed on top of a perforated gird above the steam inlet in the steam-water distillation system [13], [19]. Water and steam are used in this method, but the plant material is not in direct contact with either. Steam is generated outside the still in a boiler and flows through a pipe into the steel's bottom. Water and oil are vaporized and then condensed. Finally, a separator separates the oil from the water. In this method, steam is always fully saturated, wet, and never overheated. Also, there is no thermal degradation of the components, and the amount of steam is adjustable [24]. The basic idea behind this technique is that the combined vapor pressure equals the ambient pressure at about 100 °C, allowing volatile components with boiling points ranging from 150 to 300 ^{0}C to be evaporated at a temperature close to that of water [23], [25]. Extraction of essential oil from *kesum* leaves using this method begins with reducing the size of the fresh leaves and continues with a distillation process using conventional steam distillation for 1.5 hours to obtain essential oils, and the fractionation process is carried out through a silica gel column, and those used as eluents are hexane, benzene, chloroform, and ethanol, and continued with the GC-MS test. The yield of essential oils from this process ranges from 0.3 – 0.4% with a density value of 0.831 gms/ml and a refractive index of 1.4816 at a temperature of 27 ^{0}C [5] [6]. Syaiful *et al.* [19] reported that *kesum* leaves essential oil yield from West Kalimantan ranged from 0.08 to 0.10%, depending on the extraction time applied. Yaacob [6] developed another method for extracting *kesum* leaves essential oil by combining the method described by Yaacob [5], followed by exhaustive extraction of the steam distillate with dichloromethane evaporated the solvent to obtain the *kesum* oil with a yield of 0.3%.

3. Supercritical Fluid Extraction (SFE)

Supercritical Fluid Extraction (SFE) is an alternative method for general-purpose sample preparation to reduce organic samples and increase sample throughput [26]. This method separates one component (extractant) from the matrix using a supercritical fluid as the extraction solvent. This type of extraction is used to extract solid matrices but can also be derived from solutions [23]. The supercritical fluid which is generally used for supercritical extraction is CO_2. The advantages of CO_2 as a supercritical fluid are low critical pressure (7.4 MPa), low critical temperature (32 ^{0}C), cheap, safe, abundant, non-toxic, non-flammable, non-corrosive, and easily separated from the extract. The disadvantage of CO_2 is that its low polarity is not suitable for the extraction of polar analytes [20], [23], [27]–[29]. Commonly, CO_2 was combined with modified co-solvent to improve extraction efficiency. Usually, the co-solvent used is methanol, ethanol, and water [20], [30]. This extraction method has a higher yield, diffusion coefficient, and viscosity. Many essential oils that cannot be extracted by steam distillation can be extracted using carbon dioxide. Nonetheless, this technique is costly due to the high cost of the equipment used in this process, and it is not easy to handle [23], [30]. Supercritical fluid extraction is widely used to extract

flavors and fragrances [20], [26], [27]. Extracting essential oils using SFE CO_2 begins with drying *Polygonatum* chips in an oven at 45 ^{0}C, milled, and sieved through a particle size of 0.63 mm, to obtain *Polygonatum* powder. Three hundred grams of *Polygonatum* powder were put in an extraction kettle and heated to a specific temperature (35-50 ^{0}C). Then, the gas inlet valve is opened, a high-pressure pump is applied, and a valve is determined for the extraction process (15 – 30 MPa) at a specific time (30-150 minutes). After that, the essential oil extract was collected, and the gas inlet valve was closed. After all, processes are completed, the outlet valve is opened and cooled. The last process was turning off the equipment used. These optimum conditions for essential oil extraction using these methods are at an extraction pressure of 27 mPa, an extraction temperature of 50 ^{0}C, and an extraction time of 97.10 minutes with a yield of 2.02% with a pale yellow color and an intense aroma [7]. Markom *et al.* [20] extracted *kesum* leaves using the Supercritical CO_2 extraction method. They combined it with different co-solvents such as water, methanol (absolute methanol, 50%, and 70% concentration), and ethanol (absolute ethanol, 50%, and 70%) with yield in the range of 5.8% - 33.1%. The highest yield was found in co-solvent of 70% methanol, with a yield of 33.1%, and the lowest was 5.8% in absolute ethanol. The results of *kesum* leaves extract with various co-solvents had TP values of 1.2 – 11.2 mg GAE/g, TF values of 3.7 – 11.9 mg CAE/g, FRAP was 89 -346.7 mol Fe (II)/g, and DPPH was 39.8 – 88.7%. The highest and lowest values of antioxidant activity in *kesum* leaves extract processed with various co-solvents had the same trend data as the yield.

4. Microwave Assisted Extraction

MAE is a current technology for extracting biological materials that have been hailed as a viable alternative to traditional extraction methods due to its numerous benefits, including reduced extraction time and solvent consumption, selectivity, volumetric heating, and a controllable heating process. In various studies, MAE effectively extracts essential oils, fragrances, pigments, antioxidants, and other organic compounds found in animal tissues, food, and plants. This process has additional advantages, such as more effective heating, faster energy transfer, smaller equipment, faster warming onset, and increased yields, in addition to the time, solvent, and energy savings [31]. The essential oil extraction using this technique has been reported by Ullah *et al.* [8] using organic solvents (toluene, hexane, and pentane) and Ionic liquids (ILs), either with or without a Clevenger apparatus. Briefly, *kesum* leaves were washed with distilled water three times to remove dirt and impurities and dried for 12 days at 45 °C in the oven, and then the size was reduced to 60 -80 mesh. For MAE with an organic solvent, dried *kesum* leaves powder was mixed with organic solvent (toluene, hexane, and pentane) in a specific solid-liquid ratio in a microwave vessel and heated for a specific time (30 -60 min) at 60 °C, and microwave power of 400 watts with or without a Clevenger apparatus. Filtering was performed to separate the filtrate and residue using nylon membrane filter paper (0.02 mm). The essential oil was stored at 4 °C for further analysis. Meanwhile, for Ionic Liquid (ILs) based microwave-assisted extraction (ILMAE), the dried *kesum* leaves were mixed with aqueous ionic liquids with different concentrations (0.1 – 0.6 mol/L), solid-liquid ratio (5 mL/g – 15 mL/g), and heated for a specific time

(15-25 min with Clevenger apparatus, and 5-8 min for without Clevenger apparatus) at 60 °C, and microwave power of 400 watts. After that, the following step is the extraction using the MAE-organic solvent, as described before. The high yield of essential oil of *kesum* leaves was obtained using the ionic liquid-based microwave-assisted extraction (ILMAE) with Clevenger apparatus of 9.61%.

5. Ultrasonic Assisted Extraction

Ultrasonic Assisted Extraction is an extraction method that utilizes ultrasonic wave energy. This method can be referred to as ultrasonic extraction or sonication. The frequency used ranges from 20 kHz to 2000 kHz. The principle of this extraction method, ultrasound in the solvent, produces cavitation bubbles that can accelerate the dissolution and diffusion of the solute and heat transfer, thereby increasing the extraction efficiency. The advantages of this method are low solvent and energy consumption, low extraction temperature, and fast extraction time, making it suitable for the extraction of hazardous and thermolabile materials [26], [28]. However, the drawback of this method is that ultrasound energy (greater than 20 kHz) harms the active constituents of medicinal plants, resulting in the formation of free radicals and, as a result, undesirable changes in drug molecules [26]. While undergoing ultrasound work, the plant's raw material is immersed in water or another solvent (Methanol, ethanol, or other solvents). Many essential oils have been extracted using this method, particularly from flowers, leaves, and seeds [23]. Ullah *et al.* [8] have reported the essential oil extraction by Sonic Vibra cell (T910 DH) using organic solvents (toluene, hexane, and pentane) and Ionic liquids (ILs), either with or without a Clevenger apparatus. Briefly, *kesum* leaves were washed with distilled water three times to remove dirt and impurities and dried for 12 days at 45 °C in the oven, and then the size was reduced to 60 -80 mesh. For UAE with an organic solvent, dried *kesum* leaves powder was mixed with organic solvent (toluene, hexane, and pentane) in a specific solid-liquid ratio in the flask and sonication for a particular time (60-90 min) at 60 °C, and amplitude of 70 watts, with or without a Clevenger apparatus. Filtering was performed to separate the filtrate and residue using nylon membrane filter paper (0.02 mm). The essential oil was stored at 4 °C for further analysis. Meanwhile, for Ionic Liquid (ILs) based ultrasonic-assisted extraction (ILUAE), the dried *kesum* leaves were mixed with aqueous ionic liquids with different concentrations (0.1 – 0.6 mol/L), solid-liquid ratio (5 mL/g – 15 mL/g), and heated for a specific time (60-90 min) with or without Clevenger apparatus at 60 °C and amplitude of 70 watts. After that, the following step is the extraction using the UAE-organic solvent, as described before. The high yield of essential oil of *kesum* leaves was obtained using the ionic liquid-based ultrasonic-assisted extraction (ILUAE) with Clevenger apparatus of 9.58%.

6. Solvent Extraction

Solvent extraction, also known as solid-liquid extraction, is based on a transfer of matter that intends to separate the soluble to a solid substrate by diffusion in a solvent [32]. This extraction is generally used for processing the cosmetics, perfume, vegetable oil, and biodiesel industries. This method is suitable for fragile plants or soft textures, sensitive to heat, and has a low cost with large-scale essential oil quantity [32]. *Kesum* leaves were washed with distilled water three times

to remove dirt and impurities and dried for 12 days at 45 °C in the oven, and then the size was reduced to 60 -80 mesh. Dried *kesum* leaves powder was mixed with 40 ml of solvent (toluene, hexane, and pentane) with a specific ratio and stirred for 1 hour at room temperature. Filtering was performed to separate the filtrate and residue using nylon membrane filter paper (0.02 mm). The resulting filtrate is stored and further tested [8].

3.3. Factors Influencing the Extraction of Essential Oil From *Kesum* Leaves

The quantity and quality of essential oil from *kesum* leaves were affected by several factors, such as raw materials, different methods, and extraction conditions. Further description of each factor is as follows:

a. Raw material

The age of the *kesum* leaves affects the yield and quality of the essential oil. *Kesum* leaves harvested at 6 months have a higher yield and better quality of essential oil than the leaves harvested at 3-5 months [6]. In addition, the particle size and physical condition of the *Kesum* leaves (wet leaves and dry leaves) are other factors that affect the yield and quality of the essential oil produced [4], [9], [18].

b. Extraction Method

The extraction method significantly affects the yield value of essential oil from *kesum* leaves. Ullah *et al.* [8] reported that MAE extraction is the best method and has high efficiency in extracting *kesum* leaves essential oils compared to UAE, and conventional extraction methods, such as extraction with manual stirring and heat reflux. The extraction method using ionic liquid-based microwave-assisted extraction (ILMAE) with Clevenger apparatus has a yield value of 9.61% compared to the ionic liquid-based ultrasonic-assisted extraction (ILUAE) method (9.58%).

c. Extraction Time

Time has a significant effect when extracting the essential oil of *kesum* leaves. The increase in extraction time in steam distillation tends to increase the essential oil yield from 0.08% at 4 hours of distillation time to 0.11% at 8 hours. Furthermore, steam distillation for 8 hours reduces essential oil components from *kesum* leaves such as dodecanal, decanal, 1-dodecanal, 8-bromoneoisolongifoline 1-decanol, and 1-undecanol, but increases other components such as drimenol, trans-caryophyllene, octadecanal, and borane [19]. However, *kesum* leaves essential oil extraction time using the SFE-CO_2 method has a quadratic trend. Increasing the extraction time of *kesum* leaves by up to 90 minutes increases the yield of essential oils, but additional time tends to decrease [7]. In the hydro-distillation method, the time has a similar effect on the extraction in the steam distillation method on yield parameters [9], [18], [33]. Extraction of the essential oil of *kesum* leaves using an ionic liquid with various extraction techniques increased the essential oil yield from 15 minutes to 21 minutes with the Clevenger apparatus, and extraction time above 21 minutes did not increase the yield of the essential oil of *kesum* leaves. The highest yield value of *kesum* leaves essential oil with Clevenger apparatus was obtained at 21 and 25 minutes of extraction with a value of 9.61%. The different yield trend was shown in the extraction of essential oil of *kesum* leaves without the Clevenger apparatus. The yield value increased with extraction time from 15 minutes to 25 minutes. The highest yield value was obtained at an

extraction time of 25 minutes with a yield of 5.5% [8].

d. Temperature

The extraction temperature influences the yield value of the essential oil of *kesum* leaves. The higher the extraction temperature, the higher the yield value. Using high temperature (500 °C) in essential oil extraction using the SFE-CO_2 method resulted in the highest yield compared to temperatures of 40 and 45 °C [7].

e. Pressure

In specific extraction methods such as SFE, pressure influences the yield of the essential oil produced. The use of high pressure produces a high yield value because the increase in pressure causes an increase in the density of supercritical CO_2 so that its solubility also increases. In the extraction of essential oil from *kesum* leaves, the use of a pressure of 15 – 20 mPa increased the yield value, which was more tempestuous than the use of a pressure of 20 – 30 mPa [7].

f. Co-Solvent Concentration

The concentration of co-solvent used in the extraction of SFE-CO_2 has a major contribution to the yield of essential oil from *kesum* leaves at the same temperature (40 ^{0}C) and pressure (150 bar). The use of 70% methanol resulted in essential oil yields from the extraction of *kesum* leaves (33.1%), followed by 70% ethanol (31.5%), 50% methanol (27.9%), 50% ethanol (25.4%), water (20 .6%), absolute methanol (8.7%), and absolute ethanol (5.8%). Alcohol-water mixture produces a higher volatile oil yield value than water and alcohol. It caused the use of the water-alcohol mixture as co-solvent increases the sample solubility in the supercritical phase. Polarity changes of supercritical fluid increased the solvation power of solvents toward analytes, especially polar analytes [20].

2.3.7 Anions and Cations

The use of cation 1-allyl-3-methylimidazolium acetate [AMIM] - anion [Ac] and Clevenger apparatus was the best condition for the extraction process of essential oil of *kesum* leaves, with a yield value of 9.61%, compared to 1-butyl-3-methylimidazolium acetate [BMIM][Ac] (9.58%), 1-hexyl-3- methylimidazolium acetate [HMIM][Ac] (9.56%), 1-butyl-3-methylidazolum bis (trifluoromethyl sulfonyl) [BMIM][NTf_2] (9.56%), and 1-butyl-3-methylimidazolium methylimidazolium chloride [BMIM] [Cl] (9.5%). In general, the anion variation, the [Ac] anion, produced a higher yield value than Cl and NTf_2. Ac anions with [BMIM] cation have a lower viscosity than Cl and NTf_2 anions. The yield value of *kesum* leaves essential oil decreased without using a Clevenger apparatus because the volatile compounds evaporated due to the open system. In the condition of essential oil extraction without Clevenger apparatus, it was seen that [AMIIM][Ac] had the highest yield value (6.58%) compared to [BMIM] [Ac] (5.45%), [HMIM][Ac] (4.90%), [BMIM][NTf2] (6.40%), and [BMIM] [Cl] (5.25%). In general, variations in Ac anions have higher yield values than Cl and [NTf_2] (6.40%) with cation [BMIM] [8].

g. Ionic Liquid Concentration

Ullah *et al.* [8] investigated the use of concentrations of ILs (0.1 -0.6 mol/L) in the extraction of *kesum* leaves essential oil. The results showed that the extraction of *kesum* leaves essential oil, both using and without the Clevenger apparatus, had a significant increase in yield up to 0.5 mol/L and tended to be stable with higher

concentrations. The highest yield values extracted from the essential oil of *kesum* leaves with or without the Clevenger apparatus were 9.61% and 6.58%, respectively.

h. Solid to Solvent Ratio

The ratio of solids and solvents significantly affects the extraction efficiency of bioactive compounds, including essential oils. Ullah *et al.* [8] reported that increasing the ratio from 5 g/ml to 20 g/ml in the extraction with or without a Clevenger apparatus increased the yield of *kesum* leaves essential oil, and the use of a ratio of 1:20 g/ml tended not to increase the yield of essential oil of *kesum* leaves. In extraction using the Clevenger apparatus, the yield increased from 7.3% at a 5 g/ml ratio to 9.61% at 15 g/ml and 20 g/ml. Almost the same trend was shown in the extraction without using a Clevenger apparatus, and the yield increased from 3.7% at a ratio of 5 g/ml to 5.62% at 15 g/ml and 20 g/ml.

3.4. Bioactivity of *Kesum* Leaves Essential Oil

Kesum leaves essential oil has a benefit in the medical field, such as anti-bacterial [4], [9], [10], hepatoprotector [11], [12], anti-tyrosinase [13], [14], and antioxidant [15], [16].

3.4.1. Anti-bacterial

Kesum leaves *e*ssential oil has a robust anti-bacterial activity for all bacteria (gram-positive and gram-negative). In sequence, the lowest minimum inhibitory concentrations (MIC) that can inhibit the growth of bacteria at 6.25 µl/mL and 12.5 µl/mL for *S. aureus* and *E. coli*, respectively. The average zone inhibition of *kesum* leaves essential oil from fresh or dried *kesum* leaves in *E. coli* was lower than in *S. aureus*. The inhibition zone of *Kesum* leaves essential oil from fresh and dry leaves was 13 mm, and 19 mm, respectively. However, *S. aureus* had an inhibition zone of 21 mm for fresh leaves and 26 for dry leaves [9]. Rebickova *et al.* [4] reported that the *kesum* leaves essential oil has antimicrobial activity in a concentration of 512–1024 µg/ml in broth or agar medium. Fujita *et al.* [10] reported that the *kesum* leaves essential oil showed anti-bacterial activity against *S. choleraesuis* at 200 µg/ml concentration. The aldehyde and terpene compounds are possessed anti-bacterial properties in *kesum* leaves essential oil [4]–[6], [10], [34].

3.4.2. Hepatoprotector

Kesum leaves essential oil has a hepatoprotective effect in in-vivo animal studies. Rashid *et al.* [12] reported that *kesum* leaves essential oil at a dose of 100 mg/kg in mice protected against cisplatin-induced hepatotoxicity by decreasing CYP2E1 and oxidative stress indicators such as malondialdehyde, 8-OHdG, and protein carbonyl, as well as increasing antioxidant status (glutathione, glutathione peroxidase, superoxide dismutase, and catalase) compared to the cisplatin group. Moreover, these doses also reduced cisplatin-induced apoptosis compared to the cisplatin group and modulated changes in liver inflammatory markers (TNF-α, IL-1α, IL-1β, IL-6, and IL-10). Rashid *et al.* [11] also reported that the supplementation of *kesum* leaves essential oil at a dose of 100 mg/kg in mice reduced the levels of transaminase enzymes (ALT (alanine aminotransferase), AST (aspartate aminotransferase), dan ALP (alkaline phosphatase) enzymes) serum bilirubin and oxidative stress (glutathione, glutathione peroxidase, catalase, superoxide dismutase, and malondialdehyde) compared to the Cisplatin group. Moreover, these doses

reduce the hepatotoxicity effect, exhibited by minimal changes at cytoplasmic vacuolation, congested blood sinusoids, and the number of Kupffer cells based on light microscopic and ultrastructural examination. Dodecanal and decanal, the two main aldehydes of main active aromatic compound and concentrated in the *kesum* leaves essential oil and act as hepatoprotector [11], [12].

3.4.3. Anti-Tyrosinase

Murray *et al.* [13] reported that essential oil from fresh *kesum* leaves effectively inhibits the tyrosinase catalyzed oxidation of L-3,4-dihydroxyphenylalanine (L-DOPA). Dodecanal, decanal, and anisaldehyde were the *kesum* leaves essential oil components that function as anti-tyrosinase. Saeio *et al.* [14] also reported that the essential oil of *Polygonum odoratum* Lour whole plant (including leaves) had an inhibitory tyrosinase activity of approximately 40%.

3.4.4. Antioxidant

Woraratphoka *et al.* [15] reported that the main active components of *kesum* leaves extracts were essential oil and tannin. Ethanol extract and the essential oil of *kesum* leaves had higher free radical-scavenging activity (DPPH assay) than *C. caudatus, C. asiatica,* and *A. argyi.* However, in Ferric-reducing antioxidant power (FRAP Assay), ethanol extract and essential oil of *kesum* leaves were lower than *C. caudatus* but still high compared with the C*.asiatica* and *A. argyi* [16]. The higher antioxidant activity in *kesum* leaves essential oil was correlated with their active constituents. Dodecanal, decanal, 1-decanol, and sesquiterpenes were the main active compounds in *kesum* leaves essential oil [4], [6], [9], [13], [35], [36], and are dependent on the extraction methods used.

4. Conclusion

Kesum leaves are a type of biodiversity found in Indonesia, particularly on Kalimantan Island, which possesses many essential oils. Different extraction methods are used in order to increase the yield and quality of *kesum* leaves essential oil, such as solvent extraction, supercritical fluid extraction (SFE), microwave-assisted extraction (MAE), ultrasonic-assisted extraction (UAE), steam distillation, and hydro-distillation. Additionally, *Kesum* leaves essential oil provides numerous health advantages, such as antioxidant, anti-tyrosinase, hepatoprotecor, and anti-bacterial properties. In the future, new extraction techniques, particularly non-thermal extraction technology and other bioactivity tests, will be required for further research and verification.

Daftar Pustaka

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