2ND Makassar International Conference on Pharmaceutical Sciences (MICPS) 2023
Jurnal Fitofarmaka Indonesia, 2023; 10(3) 101-106
http://jurnal.farmasi.umi.ac.id/index.php/fitofarmakaindo/index

GC-MS Analysis of Lemongrass with Various Extraction Methods

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Article info
History
Submission: 26-10-2023
Review: 29-11-2023
Accepted: 05-12-2023

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DOI: 10.33096/jffi.v10i3.1108

Keywords:
extraction; GC-MS; lemongrass (Cymbopogon citratus)

Abstract
Lemongrass (Cymbopogon citratus) is one plant producing bioactive essential oil. This research aims to determine the percentage yield of extracts and GC-MS analysis of chemical compound profiles of lemon grass with various extraction methods. Lemongrass bioactive compounds are extracted using maceration, ultrasonic, and distillation. The highest percentage extract yield was from maceration extraction, namely 14.83%, ultrasonic extraction was 4.68%, and distillation extraction was 6.00%. The GC-MS results showed that the secondary metabolite compounds resulting from maceration, ultrasonic, and distillation contained geraniol, citronellal, and citronellol. The citronella compound group was obtained with the highest percentage from distillation extraction at 14.83%, followed by ultrasonic extraction at 2.23% and maceration extraction at 0.37%.

I. Introduction

Lemongrass (*Cymbopogon citratus*) is a spice that is very abundant in Indonesia. Kitchen lemongrass is also one of the commodities that have the potential to be developed for its use, both as a food ingredient and as an industrial raw material, kitchen Lemongrass is widely used as a spice in some processed foods, while as an industrial raw material kitchen, Lemongrass can be processed into essential oil. Essential oil is obtained from Lemongrass plants where the essential oil itself is volatile at room temperature and smells fragrant. according to the smell of the producing plant, essential oils contain resin, and wax in small enough quantities which is a non-volatile component (Muslida, Norfai and Rahman, 2018).

Lemongrass (*Cymbopogon citratus*) can be empirically used as a medicine for headaches, coughs, stomach pain, diarrhea, body warmers, heat reducers, and mosquito repellents. Lemongrass stem extract contains several vegetable constituents, which are essential oils. The stem of kitchen Lemongrass (*Cymbopogon citratus*) contains saponins, tannins, alkaloids, and flavonoids (Nuryadin et al., 2018).

The content of secondary metabolite compounds in Lemongrass plants (*Cymbopogon citratus*) in the form of saponins, and tannins have mucolytic activity. Saponins can play a role in the discharge of bronchial secretions and increase the activity of a cell that has cilia so that it can remove phlegm. Secondary metabolites in the form of tannins can shrink the mucous membrane of the intestine (Clara, Arifuddin and Rusli, 2022).

Lemongrass essential oil can be obtained by extraction or distillation of other methods that can be used to take Lemongrass essential oil is the extraction method. The extraction methods used are maceration and ultrasonic, where maceration is used because it is suitable to extract compounds that are not heat-resistant such as essential oils. Ultrasonics can shorten the extraction time and increase the extraction yield (Wen *et al.*, 2018).

In addition to using water distillation and ultrasonic maceration extraction methods, to identify the type and percentage of content of essential oil chemical compounds, there are several studies that use other extraction methods, including extraction using the reflux method on iler leaves with the solvent used, namely 70% ethanol, getting a percent yield of 26.31% (Utami *et al.*, 2020).

Some researchers suggest that there is an influence on the type and percentage of citronella (*Cymbopogon citratus*) essential oil chemical compound content by maceration, water distillation, and ultrasonic extraction methods.). The UAE lemongrass essential oil yield is higher than that of other extraction methods, such as microwave-assisted hydrodistillation (MAHD), which produced 0.35% for 90 min of extraction, and hydrodistillation, which produced 0.2% for 360 min (Yuniarto *et al.*, 2022) while extraction by using different solvents namely methanol, acetone, and ethyl acetate the percent yield is around 6.73%, 0,44%, and 3.15% respectively (Ariyani, Eka Setiawan and Edi Soetaredjo, 2008). Maceration extraction using a solvent ratio of 95% methanol and 70% *n*-hexane resulted in yields of 11.64 and 5.08% (Evama, Ishak and Sylvia, 2021).

II. Research Method

II.1 Sample Collection and Processing

Lemongrass (*Cymbopogon citratus*) samples were taken from those growing in the South Sulawesi region. 11 kg of Lemongrass that had been collected was then cleaned of dirt, chopped, and dried in the drying room. the dried Lemongrass was ground to obtain a dry powder weight of 776 g and then stored in a clean place and free from water.

II.2 Sample Extraction

II.2.1 Maceration

200 grams of sample was put into a maceration container then the sample was soaked with 70% ethanol (1:7) for 3 days while occasionally stirring. The macerate was collected and evaporated with a vacuum rotary evaporator at 60°C and concentrated on a water bath at 60-70°C until a thick extract was obtained (Bachri, Nursalma and Nora, 2015).

II.2.2 Ultrasonic

50 grams of Lemongrass powder was weighed and put into an eluent bottle and 350 mL of ethanol solvent was added. Extraction was carried out ultrasonically at 50°C for 2 hours. The extract was filtered with filter paper, then evaporated using a rotary vacuum evaporator until a thick extract was obtained (Maharani, Bintari and Wulandari, 2022).

II.2.3 Distillation

A total of 50 grams of Lemongrass powder was put into a distillation flask, then 350 mL of distilled water was added, after which the distillation process was carried out for 3 hours with temperatures ranging from 100°C-105°C (Bachri, Nursalma and Nora, 2015).

II.3 Identification of compound components using Gas Chromatography – Mass Spectrometry (GC-MS)

II.3.1 Sample preparation

0.1 mL of sample was added to a mixture of chloroform and methanol (1:1) as much as 5 mL. Extracted using a sonicator for 20 minutes at 40°C. The top layer formed was pipetted into a vial and GC-MS tested.

II.3.2 Operating GC-MS Ultra QP 2010 Shimadzu

0.5 mL of isolate into a 50 mL volumetric flask and dilute with acetone and dilute to the limit. Pipette as much as 3 mL and put it into a vial. GC-MS instrument conditions Injector temperature 250°C with Splitless mode, 76.9 kPa pressure and 14 mL/min flow rate and 1:10 ratio and a ratio of 1:10. Ion source and interface temperatures 200°C and 280°C, solvent cut time 3 minutes, 400-700 m/z. Column type SH-Rxi-5Sil MS column length 30 m with an inner diameter of 0.25 mm. The initial column temperature was 700°C with a holding time of 2 minutes and the temperature was increased to 200°C at a rate of 100°C/min and a final temperature of 280°C with a holding time of 9 minutes at a rate of 50°C/min for a total analysis time of 36 minutes. The chromatogram data obtained was read using NIST and Wiley 9 libraries.

III.4 Data analisys

The percentage yield value of the extract can be calculated by the formula (1).

$$\text{Percentage Yield} = \frac{\text{Lemongrass extract weight}}{\text{Lemongrass sample weight}} \times 100\% \quad (1)$$

III. Results and Discussion

Samples used in this study were Lemongrass stems (*Cymbopogon citratus*) from South Sulawesi. Sample handling was carried out by cleaning Lemongrass stems first, then dried in a drying cabinet. The sample was then pulverized to reduce its size and expand the contact area between the sample powder and the distillation liquid so that the active substance content is much extracted. Samples were then weighed as much as 200 grams for maceration, 50 grams for water, and ultrasonic distillation. The three methods were used to determine which method produced the highest percentage of extract yield.

Ethanol 70% solvent is used in maceration and ultrasonic extraction methods, aiming to attract chemical components in Lemongrass, because ethanol is a universal solvent that can attract compounds that are soluble in non-polar to polar solvents (Astarina, Astuti and Warditiani, 2013). The Lemongrass essential oil component contained in it is a terpenoid group. Essential oils are polar due to the presence of polar components such as geraniol, which will dissolve in polar solvents, while non-polar essential oil components such as citronellol will also be partially extracted (Evama, Ishak and Sylvia, 2021).

The yield value of Lemongrass extract obtained from maceration, ultrasonic, and water distillation extraction can be seen in Table 1.

Table 1. Effect of extraction method comparison on Lemongrass stem extract yield (*Cymbopogon citratus*)

Extraction	Solvent	Fresh sample weight (kg)	Dried sample weight (g)	Total solvent (mL)	Extract weight (g)	Yield value (%)
Maceration	Ethanol 70%	11	200	2800	28,45	14,23
Ultrasonic	Ethanol 70%		50	350	2,34	4,48
Destillate	Aquadest		50	350	3.00	6.00

The way to obtain the desired compound is by extraction. The extraction methods used are maceration, distillation, and ultrasonic. According to (Hasnaeni, Wisdawati and Usman, 2019) the maceration method is a cold extraction method that has the advantage of using simple equipment such as maceration vessels and glass bottles, easily carried out without special treatment, namely only by immersing the sample in the extracting solvent while occasionally stirring. Distillation extraction can be defined as the process of separating the components of a mixture consisting of two or more liquids based on differences in vapour pressure or based on differences in the boiling points of the compound components. Ultrasonic extraction is an extraction method that uses ultrasonic waves, namely acoustic waves with a frequency range of 16-20 kHz. The use of ultrasonic in the extraction process of organic compounds in a plant using organic solvents can take place more quickly, where the cell walls of the material will break with ultrasonic vibrations so that the content inside can come out easily (Yuniarto *et al.*, 2022).

The results of the identification of essential oil compounds of Lemongrass extract (*Cymbopogon citratus*) can be seen in Table 2.

Table 2. Average AUC and percent anti-inflammatory power in rat paw edema thickness

Essential Oil	No	Peak	RT	Chemical Compound	% Area	Boiling Point
Water extract from Destillation	1	14	7.867	2,6-Octadional acid, 3,7-dimethyl-, (Z)- **Citronellal**	14.83	204ºC-208 ºC
	2	16	8.309	E-Citral **Citronellal**	14.64	204ºC-208ºC
	3	15	8.041	GERANIOL **Graniol**	9.58	125ºC-150ºC
Ethanol 70% extract from Ultrasonic	1	5	9.815	2,6-Octadienoic acid, 3,7-dimethyl-, (E)- **Citronellal**	2.23	204ºC-208ºC
	2	4	9.258	Neric acid **Citronellal**	0.29	204ºC-208ºC
	3	7	10,296	CITRONELLA **Graniol**	0.16	125ºC-150ºC
Ethanol 70% extract from Maceration	1	15	9.875	2,6-Octadienoic acid, 3,7-dimethyl-, (E)- **Citronellal**	0.37	204ºC-208ºC
	2	36	12.669	Pulgeone **Citronellal**	0.16	204ºC-208ºC
	3	95	35.857	10-PINANOL **Graniol**	0.08	125ºC-150ºC

The results of the yield value of extracts obtained using maceration, ultrasonic, and water distillation extraction methods can be seen in Table 2 where the yield value of extracts with maceration extraction is higher than ultrasonic extraction and water distillation, using maceration extraction obtained a yield value of 14.23% extract while in ultrasonic extraction and water distillation methods obtained a yield value of 14.23% and 6% extract.

The highest yield of extracts found in maceration extraction is thought to be due to the extraction time carried out and the presence of re-maceration, so it is likely that the compound withdrawal process is maximized compared to other methods. The yield of a sample is very necessary because it is to know the amount of extract obtained during the extraction process, besides that the yield data has a relationship with the compounds of a sample so that if the amount of yield is more, the number of compounds contained in the sample is also more. As Hasnaeni has reported that the high compounds contained in a sample are indicated by the high amount of yield produced (Hasnaeni, Wisdawati and Usman, 2019).

Measurement of Lemongrass stem extract was then analyzed using a GC-MS Spectrophotometer which showed the presence of several chemical compounds contained in Lemongrass stem extract seen based on peak height (peak), retention time (RT) and percent area.

The water distillation extract of Lemongrass stems contains various types of essential oil chemical compounds, such as citronellal, citronellol and graniol, where based on the results of the analysis that have been analysed from the GC results, 13 peaks were detected as chemical compounds of essential oil groups after being analysed into the form of essential oil grouping based on GC and MS information, 3 peaks of chemical compounds were obtained which had the highest percentage area where the essential oil compounds 2,6-Octadional acid, 3,7-dimethyl-, (z)-

citronellal group had the highest peak percentage area of 14. 83% retention time (7.86 min).

Ultrasonic extracts of Lemongrass stem contain various types of essential oil chemical compounds, namely citronellal, citronellol, and graniol, which based on the results of the analysis that have been analyzed from the GC results detected there are 4 peaks as chemical compounds of essential oil groups after being analyzed into the form of essential oil grouping based on GC and MS information obtained 3 peaks of chemical compounds that have the highest percentage area where essential oil compounds 2,6-Octadienoic acid, 3,7-dimethyl-, (E)- citronellal group which has the highest peak percentage area of 2. 23% at retention time (9.81 minutes).

The macerated extract of Lemongrass stems contains various types of essential oil chemical compounds, these consist of citronellal, citronellol, and graniol, where based on the results of the analysis that have been analyzed from the GC results, 3 peaks were detected as chemical compounds of essential oil groups after being analyzed into the form of essential oil grouping based on GC and MS information, 3 peaks of chemical compounds were obtained which had the highest percentage area where the essential oil compounds 2,6-Octadienoic acid, 3,7-dimethyl-, (E)-citronellal group which had the highest peak percentage area of 0. 37% at retention time (9.87 minutes).

Based on the data obtained, it can be seen that to extract Lemongrass essential oil with the target chemical compounds citronellal, citronellol and graniol should use the water distillation extraction method.

IV. Conclusions

Based on the results of the research that has been carried out, it can be concluded that the yield value of Lemongrass (*Cymbopogon citratus*) extract from three extraction methods which are maceration, ultrasonic, and water distillation where the maceration extraction method obtained a high percentage of extract yield of 14.23% compared to ultrasonic extraction of 4.68% and water distillation of 6.00%.

The results of identification of essential oil compound extracts using GC-MS spectrophotometry of Lemongrass extracts by maceration, water distillation, and ultrasonic methods obtained several types of essential oil chemical compounds namely graniol, citronellal, and citronellol. The citronellal compound has the highest peak compared to the types of graniol and citronellol compounds, from the three extraction methods the highest citronellal compound is found in distillation extracts with the chemical compound 2,6-Octadional acid, 3,7-dimethyl-, (Z)- with a percent area of 14. 83% at a retention time of 7.86 minutes compared to the ultrasonic extraction method with the chemical compound 2,6-Octadienoic acid, 3,7-dimethyl-, (E)- with a percent area of 2.23% and a retention time of 9.81 minutes and maceration with the chemical compound 2,6-Octadienoic acid, 3,7-dimethyl-, (E)- with a percent area of 0.37% and a retention time of 9.87 minutes.

V. Acknowledgment

The authors express their sincere thanks to the Chancellor of the Universitas Muslim Indonesia (UMI), Research and Development Institute (LP2S) of UMI, and the Dean of the Faculty of Pharmacy UMI, for supporting and providing funding for this research.

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