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Ultrasound Microwave Assisted Extraction on Citronella Leaves Using Ionic Solvent

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Abstract

Leaves of citronella (Cymbopogon nardus L.) have pretty much essential oil. Essential oil from citronella leaves is generally extracted by conventional methods. Conventional method is still less effective. The problem can be overcome by using non-conventional methods in the form of UMAE (Ultrasound-Microwave Assisted Extraction). UMAE is an extraction process assisted by ultrasound and microwaves. NaCl solution can be used as a solvent in the UMAE method because NaCl has a large dielectric constant. The aim of this study was to examine the effect of extraction time and the addition of salt on the yield and color of essential oils produced using the UMAE. The extraction process begins with the raw material preparation stage, such as harvesting, size reduction, and drying. Then, pre-treatment was carried out using ultrasound for 5 minutes at 35°C with the addition of 2, 4, 6, 8, and 10% (w/v) salt. Pre-treatment results were followed by the extraction process using microwaves for 30, 45, 60, 75, and 90 minutes. The best results were obtained with the addition of 6% salt and 60 minutes of extraction time with a feed to solvent ratio of 0.1 with a yield of 1.975% and the color is yellow.

Keywords: *assisted extraction, cymbopogon nardus l, essential oils, ionic liquid, ultrasound-microwave assisted extraction, yield*

Abstrak

Ultrasound Microwave Assisted Extraction Pada Daun Serai Wangi Menggunakan Pelarut Ionik. Daun serai wangi (*Cymbopogon nardus L.*) memiliki kandungan minyak atsiri yang cukup banyak. Minyak atsiri dari daun serai wangi umumnya diekstrak dengan metode konvensional. Metode ini masih kurang efektif. Permasalahan ini dapat diatasi dengan menggunakan metode nonkonvensional berupa UMAE (*Ultrasound-Microwave Assisted Extraction*). UMAE merupakan proses ekstraksi menggunakan gelombang ultrasonik dan gelombang mikro. Larutan garam NaCl dapat digunakan sebagai pelarut pada metode UMAE karena NaCl mempunyai nilai konstanta dielektriknya yang besar. Tujuan penelitian ini untuk mengkaji pengaruh waktu ekstraksi dan penambahan garam terhadap yield dan warna minyak atsiri yang dihasilkan menggunakan metode UMAE. Proses ekstraksi diawali dengan tahap persiapan bahan baku yaitu, pemanenan, pengecilan ukuran daun, dan pengeringan. Kemudian dilakukan *pre-treatment* menggunakan gelombang ultrasonik selama 5 menit pada suhu 35°C dengan penambahan garam 2, 4, 6, 8, dan 10% (b/v). Hasil *pre-treatment* dilanjutkan dengan proses ekstraksi menggunakan bantuan gelombang mikro selama 30, 45, 60, 75 dan 90 menit. Hasil terbaik didapatkan pada variabel penambahan garam 6% dan 60 menit waktu ekstraksi dengan rasio *feed to solvent* 0,1 dengan yield 1,975% dan warna minyak atsiri berupa kuning.

Kata kunci: assisted extraction, cymbopogon nardus l, minyak atsiri, pelarut garam, ultrasound-microwave, yield.

INTRODUCTION

Essential oils or called aesthetic oils are a large group of vegetable oils that are viscous liquids at room temperature but evaporate easily, giving them a distinctive aroma [1]. One of the plants that have high prospects for producing essential oils is (*Cymbopogon nardus* L). In general, citronella plants are only used as a spice in the kitchen and not much further processing is carried out. This plant contains essential oils which can increase the selling price to be higher [2]. Citronella essential oil can be used as a basic ingredient for cosmetics, perfumes, medicines, and fragrances [3]. Citronella leaves contains citronella essential oil with the largest components, namely citronellol 32-45%, geraniol 12-18%, and citronellal 12-15%. These components are very important for use in various industries and determine the intensity of the smell, fragrance, and the price value of citronella essential oil [4]. The part of citronella plant that is commonly extracted is the leaves. This is because this section has the highest essential oil content and high commercial value in the global market [5]. Generally, the parts of the citronella plant can be divided into leaves, pseudo stems, and stems. The leaf part produces a higher citronella essential oil yield of around 0.88% than the pseudo stem part of around 0.59% [2].

Many studies have been carried out in extracting essential oils from citronella plants, generally in the form of conventional methods such as maceration, soxhletation, and water distillation. Research on extracting essential oil from citronella leaves using the Soxhlet method was carried out by [4] to obtain a yield of 1.8% (v/w) with an extraction time of 90 minutes. In the maceration method for extracting citronella essential oil from citronella leaves carried out by [6], a yield of 11.64% was obtained within 3 days. In a study conducted by [7] regarding the extraction of citronella essential oil using the distillation method, the highest yield of citronella leaf essential oil was 1.22% in 4.5 hours at 130°C. The disadvantages of these methods are they have low extraction efficiency, the energy used is quite large, it takes too long, and allows the degradation of thermolabile compounds [8].

Based on several studies of essential oil extraction using conventional methods, the results are less than optimal, it is necessary to have a breakthrough in the latest extraction methods with non-conventional methods. Research conducted by [9] compared the use of conventional and non-conventional methods. The research showed that the extraction of kaffir leam essential oil using the hydrodistillation method produced a yield of 0.33%, the Microwave Assisted Extraction (MAE) method produced a yield of 1.045% and the Ultrasound Microwave Assisted Extraction (UMAE) method produced a yield of 1.684%. with 60 minutes. Based on this research, the UMAE method can be used as an alternative extraction method to increase the yield obtained. Research conducted using a microwave is often found using a solvent in the form of aquadest. This is because aquadest has a high dielectric constant value of around 80. The addition of salt to aquadest is an alternative to improve the ability to distribute heat better [10]. From the research on cumin essential oil extraction, it was shown that variations in salt solvents had an effect on the composition and yield of the essential oil. The use of NaCl salt showed better results than $BaCl_2$ salt on the overall quality of the essential oil [11].

Based on the description above, research was carried out on the extraction of citronella essential oil from citronella leaves using ultrasonic waves as pre-treatment and followed by microwaves from the microwave to study the effect of variations in the concentration of NaCl salt solution and extraction time on the essential oil. Pre-treatment by using ultrasound waves will help penetrating NaCl solution to wall cell of citronella leaves by cavitation bubbles effect. Afterwards, extracting by using micro wave will generate much more citronella essential oil by using solution which can adsorb electromagnetic waves of microwaves devices and turn it into heat.

MATERIAL AND METHODE

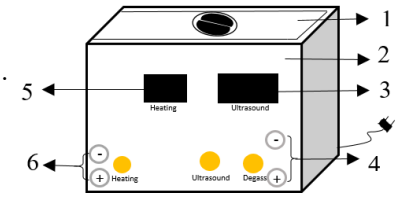
The material used is citronella leaves (*Cymbopogon nardus* L.) that are harvested from the citronella cultivation area “Kosagraha Lestari Farmers Group” in the Surabaya area, as like **Figure 1.(a)**. Other supporting materials are Pro Analysis NaCl salt (Merck) that was purchased at the chemical shop “UD. Nirwana Abadi”, as like **Figure 1.(b)**, and aquadest as solvent. NaCl was diluted by aquadest to make salt solution with a various concentration.

(a) (b)

Figure 1. Citronella Plants (*Cymbopogon nardus* L.) from Kosagrha Lestari Farmers Group (a) and NaCl Component (b)

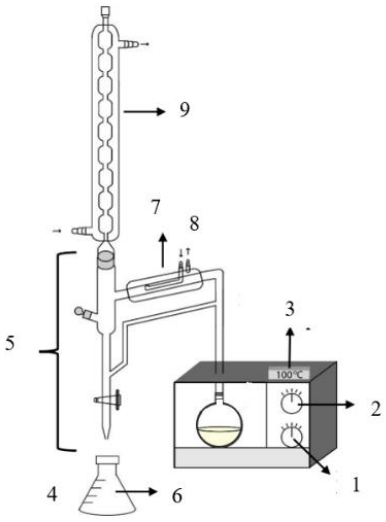
Equipment

The tools that is used consist of a series of pre-treatment tools using a cleaning bath-type sonicator and a series of extraction tools in the form of a modified microwave. Sonicator tools that is used for pre-treatment has specification such as Ultrasonic Batch BK-1200, ultrasonic power 60 Watt, heating power 100 Watt, frequency ultrasonic 40kHz, capacity 1600 ml, is depicted as **Figure 2.** Meanwhile, modified microwave devices that is used for extracting citronella essential oil has specification such as Electrolux EMM2308X, magnetron 2450 MHz, is depicted as **Figure 3.**



1. Cap of sonicator
2. Body of sonicator
3. Timer displayed
4. Timer buttons
5. Temperature displayed
6. Temperature buttons

Figure 2. A series of pre-treatment tools using a sonicator



1. Timer
2. Power Button
3. Temperatur Indicator
4. Erlenmeyer
5. Clavenger
6. Citronella Oils
7. Liebig Condensor
8. Water Flow
9. Refluks Condensor

Figure 3. A series of extraction tools using a microwave (adopted from [12])

Extraction of Essential Oil from Citronella Leaves

1. Preparation Stage for Materials

Citronella leaves were reduced in size to ± 1 cm. Citronella leaves were dried using an oven at 100°C for 90 minutes to a constant weight, then analysed for water content using equation (1). The solvent used was a solution of NaCl salt with various concentrations of 2%, 4%, 6%, 8%, and 10% (w/v).

2. Pre-treatment Stage Using Sonicator

The pre-treatment process of extracting citronella essential oils from citronella leaves was carried out using a sonicator. Citronella leaves that have been reduced in size and dried, are put in a closed container. The solvent used in this study was 400 ml of NaCl salt solution according. The pre-treatment process was carried out for 5 minutes at 35°C.

3. Extraction Stage Using Microwave

The results of the pre-treatment process obtained from the sonicator were then carried out with a further extraction process using a microwave based on the time variable (minutes) for 30, 45, 60, 75, and 90. In the clavenger section, a salt solution will be added according to the predetermined concentration variable. The purpose of adding a salt solution to the clavenger is to maintain the concentration of the salt solvent in the flask during the extraction process. Flask 1000 ml was chosen to increase quantity of citronellal essential oil [13]. The extraction process will produce two layers in the form of citronella essential oil and a salt solution. The citronella leaf essential oil is separated manually by controlling valve of clavenger until all of salt water come out and the results are collected in a glass vial as citronella essential oil. The results will be analysed for its yield using equation (2).

4. Analysis Stages

a. Analysis of Moisture Content of Citronella Leaves

Analysis of the lost water content in the leaves can be measured based on the difference in weight that is obtained before and after drying using the oven. According to [14], the lost water content can be calculated with the following equation:

$$Water\ Content == \frac{W_1 - W_2}{W_1} x\ 100\% \quad (1)$$

W_1 = Weight of Fresh Citronella Leaves

W_2 = Weight of Dry Citronella Leaves

b. Analysis of Citronella Essential Oil Yield

Yield indicates the amount of citronella essential oil obtained from each gram of citronella leaves sample extracted (% w/w). According to [15], yield is calculated by the equation:

$$Yield\ (\%) = \frac{W_3}{W_2} x\ 100\%\ (2)$$

W_2 = Weight of Dry Citronella Leaves
W_3 = Weight of Citronella Essential Oil

c. Analysis of Citronella Essential Oil Color

Color indicates quality of citronella essential oil. There are two standard for citronella essential oil, such as SNI 06-3953 1995 and ISO 3848: 2016 (E). ISO 3848 : 2016 (E) was chosen because it is avowed internationally as standard of citronella essential oil. Result of analysing color will be compared to ISO 3848 : 2016 (E).

RESULT AND DISCUSSION

Presenting the Results

This study of citronella essential oil extraction of citronella leaves aims to examine the effect of extraction time and concentration of the salt solution on the yield of essential oil of citronella leaves. In carrying out this research several other conditions could affect the levels of essential oil of citronella leaves obtained, such as harvesting time and treatment of ingredients from citronella leaves. Harvesting is done in the morning around 08.00-09.00 WIB with the condition that the citronella leaves that can be harvested are dark green and wide in size. Citronella leaves was harvested by using big scissor especially for grass. These conditions were chosen to obtain optimal essential oil quantity and quality [3]. Material treatment was carried out in the form of cutting the size of citronella leaves to ± 1 cm to accelerate the rate of mass transfer of citronella essential oils. Meanwhile, drying can affects the yield obtained. The less the water content of the ingredients, the more citronella essential oils will get. The water content of citronella leaves that have been oven has decreased by 65% so only 35% of their initial weight remains. However, if the water content is too low with drying conditions applied at high temperatures for a long time, the yield of citronella essential oil will be low [16].

A. Analysis Yield of Essential Oil from Citronella

This study of essential oil extraction of citronella leaves aims to examine the effect of extraction time and concentration of the salt solution on the yield of citronella leaf essential oil produced. The relationship between variations in the concentration of the salt solution and the extraction time used for obtaining the yield of citronella leave essential oil, is presented in **Figure 4.**

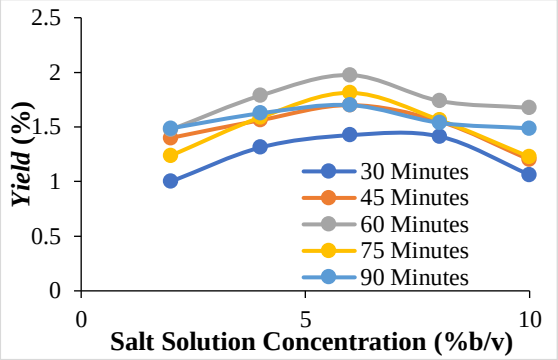


Figure 4. Effect of Salt Solution Concentration on Essential Oil Yield at Various Extraction Times

The effect of variations in salt solution concentration on the yield of citronella essential oil is shown in **Figure 3.** Based on this figure, it can be seen that there is an increase in yield that occurs at a salt solvent concentration of 2% to 6%. This shows that the concentration of the salt solution affects the yield of citronella essential oil because the salt solution as a solvent has a high dielectric constant of 150 in solid form [10]. The dielectric constant is a measure that indicates the ability of a solvent to absorb microwaves [17]. The water and salt molecules in the solvent will absorb electromagnetic energy which functions as a heating agent in the microwave so that it can assist the extraction process [18]. The research results of [19] stated that variations in the concentration of NaCl salt solutions can increase the citronella essential oil. This study stated that the salt solution will provide a greater heat effect than pure solvents so that the matrix walls of citronella leaves can be damaged more quickly and the oil content can be extracted quickly. The salt solvent used in this study is polar, while the citronella essential oil is non-polar. The NaCl salt solvent used acts as a carrier so that the citronella essential oil will not dissolve and an citronella essential oil emulsion will form in water at the beginning of the extraction process. The use of a greater concentration of salt solution can also stabilize the surface tension between citronella essential oil and water (salt). This phenomenon can be observed when the extraction process in the clavenger section forms an oil emulsion in the aquadest which can be identified by the cloudy color of the aquadest in the clavenger. These citronellas essential oil and aquadest emulsion will be visible for up to 60 minutes. After 60 minutes, the oil-in-aquadest emulsion which is characterized by a cloudy color will slowly fade and disappear and a

clear layer begins to form between the citronella essential oil and the aquadest. Based on this, the extraction of citronella essential oil can be given a certain time to separate the citronella essential oil and water emulsion so that a large amount of citronella essential oil. At a salt concentration of more than 6%, the average yield decrease is 6.4% - 17.69%. This phenomenon caused by the recrystallization phenomenon that has occurred in the solution so that many salt crystals are formed and cover the surface of the pore matrix of citronella leaves. Barriers from the presence of salt crystals that are formed resulted in the essential oil being blocked from exiting so that the yield decreased. The use of a concentrated salt solution can increase the extraction results. Recrystallization happened due to connection between flask and clavenger do not fit each other. The bigger concentration of salt solution and the bigger lacking will have bigger possibility to create recrystallization phenomenon. Extraction used microwave will accelerate mass transfer between each component involved. Lengthen extraction time will not generate much more citronella essential oil, because extraction system has reached equilibrium stage [13].

However, if the concentration is enlarged, it will make the solution more saturated so that it can reduce the ability of the solvent in the extraction process [20]. The addition of salt will make the solution easier to distribute heat. The heat that is delivered with the help of the addition of salt will accelerate the mass transfer of citronella essential oil from the leaves to the solvent. The bigger addition of salt, the smaller the resistance that arises during the mass transfer process. However, this is only up to 6% salt concentration. A salt concentration greater than 6% will make the solution more saturated so that the resistance that arises during the mass transfer process will be greater. The existence of a large resistance will make the mass transfer process of citronella essential oil from the leaves to the solvent smaller. Decreasing the mass transfer of citronella essential oil from the leaves to the solvent will make the yield of citronella essential oil smaller.

Figure 4. also shows that the extraction time affects the yield of citronella essential oil. In the process before extraction with the help of microwaves, citronella leaves were pre-treated with ultrasonic waves. Pre-treatment aims to increase the penetration of the solvent in the plant matrix so that the cell walls of citronella leaves are damaged more quickly by the presence of ultrasonic waves [21]. The combined effects of ultrasonic waves and microwaves will speed up the extraction process of citronella essential oil. When compared with the research of [22] that the extraction of citronella essential oil using steam and water distillation with the help of microwave heating produced a yield of 1.52% which was lower than that obtained in this study of 1.975%. The same thing was also conveyed by [9] that the results of extraction with the UMAE method have higher yields than the MAE (Microwave Assisted Extraction) method and water distillation. The longer the extraction time, the longer the contact time between the raw material and the solvent so the amount of citronella essential oil will increase. However, too long a time can also cause some of the thermolabile compounds contained in citronella essential oil to degrade [23] . At first, variations in the extraction time of citronella essential oil will increase the yield obtained. However, the extraction time that is too long can reduce the yield due to the long heating duration of the microwave device. The longer the microwave extraction time, the higher the temperature of the solvent and some of the solvents evaporate. This can reduce the volume of the solvent so that it can reduce the ability of the solvent to extract essential oils from citronella leaves. An extraction process that is too long will also make the solution more saturated because some of the solvents evaporate. The more saturated the solution, the lower its ability to extract essential oils so that a smaller essential oil yield will be obtained. Extraction time that is too long is indicated by a decrease in the amount of yield in the 75^{th} to 90^{th} minutes, which is 4.1% -14.43%. At the final time variable of 90 minutes will generate low quantity of citronella essential oil. It is because of losing thermolabile compound and solution become more saturated. Based on **Figure 4.** the information obtained that the highest yield was obtained at the variable extraction time of 60 minutes with a 6% salt solution concentration of 1.975%.

B. Color Analysis of Essential Oil from Citronella

The color of the citronella essential oil from citronella leave obtained through an extraction process using the Ultrasound Microwave Assisted Extraction (UMAE) method using ionic solvent (NaCl) has various colors. The representative color range of citronella essential oil is shown in Error! R eference source not found..

Figure 5. Range Color of Essential Citronella Oil (Pale Yellow – Yellow – Thick Yellow)

One of many parameter of citronella essential oil is color. Citronella essential oil color can be used to determine the quality of the essential oil of a material. Intensity of the color is determined by the amount of certain color pigments in the essential oil. Newly extracted essential oil is usually colorless or yellowish. In color analysis, visual observations were made, by observing the color of citronella essential oil at a distance of about 30 cm using a white background to make identification easier [17]. If essential oils are left in contact with air and exposed to sunlight or any light directly for a long time, the citronella essential oil can darken, the smell changes, thickens over time, and eventually forms resin [7]. Based on this, the observation of the color of the citronella essential oil obtained can be used as a parameter that the essential oil is in good condition and can be used properly. The results of the color analysis of citronella leaf essential oil are presented in **Table 1**.

Table 1. Color Analysis of Essential Citronella Oil

NaCl Solution (% b/v)	Extraction Time (Minutes)	Color
2 %	30	Pale Yellow
	45	Pale Yellow
	60	Pale Yellow
	75	Pale Yellow
	90	Pale Yellow
4 %	30	Pale Yellow
	45	Pale Yellow
	60	Pale Yellow
	75	Pale Yellow
	90	Pale Yellow
6 %	30	Pale Yellow
	45	Yellow
	60	Yellow
	75	Thick Yellow
	90	Thick Yellow
8 %	30	Yellow
	45	Yellow
	60	Thick Yellow
	75	Thick Yellow
	90	Thick Yellow
10 %	30	Thick Yellow
	45	Thick Yellow
	60	Thick Yellow
	75	Thick Yellow
	90	Thick Yellow

In **Table 1.**information is obtained that variations in the concentration of the salt solution and also the extraction time have an effect on the color of the citronella essential oil. If analysed as a whole, the extraction time and concentration of the salt solution will give a significant difference in color when the variations in the long extraction time and the concentration of the salt solution are large. The longer the extraction time, the more citronella essential oil will be obtained [7]. This will certainly make it easier to observe the color. The longer the extraction time, the thicker the color of the citronella essential oil will be obtained. Based on that condition so that yield of citronella essential oil also effects on analysing color result. Meanwhile, the effect of variations in the concentration of the salt solution was also quite significant on the visible color of the citronella essential oil obtained. This is comparable to the research of [4] which states that variations in the concentration of salt solutions have an effect on the resulting color but do not affect the resulting odor. The greater the concentration of the salt solution, the thicker the citronella essential oil will be, so that the color difference will be clearly visible. Usage of NaCl solution affects yield quantity citronella essential oil on extraction process using microwaves and stabilizing surface tension at separation process. The bigger concentration of salt solution will generate much more citronella essential oil, just until concentration of 6% (b/v). Concentration of salt solution more than 6% (b/v) will decrease citronella essential oil due to solution being more saturated. Usage of NaCl salt will not affect the smell of citronella essential oil produced. It is proven that with variations in the concentration of any salt solution, will get citronella essential oil which has a smell like citronella leaves before the extraction process is carried out. The observation results obtained were then compared with ISO 3848 : 2016 (E) concerning specifications for the quality

of citronella essential oil internationally. From the observations, the color of the citronella essential oil was pale yellow to thick yellow. This shows that the oil obtained is in accordance with ISO 3848 : 2016 (E) which has been determined, which is in the range of pale yellow to pale yellowish brown.

CONCLUSION

The addition of salt and extraction time have a significant effect on the yield. The higher the addition of salt and extraction time, the higher the yield. The best results of extraction of citronella essential oil using the Ultrasound Microwave Assisted Extraction method were obtained at the condition of adding 6% salt and 60 minutes of extraction time with a yield of 1.975% and yellow color of essential citronella oil (*Cymbopogon nardus* L.).

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