The effect of method, type of solvent and extraction time towards the yield of oil on essential oil extraction from lime peel (*Citrus aurantifolia*)

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Abstract. Essential oil is a volatile substance and has a distinctive scent that can be found on plants. Lime peel oil is one part of the plant which can be processed to make an essential oil so that it can be used as a natural fragrance in soap making. The purpose of this study is to produce essential oil from lime peel, which can be used as a substitute for synthetic fragrance ingredients in soap making. The other purpose is to obtain the optimum method, type of solvent, and the optimum extraction time to produce essential oil. The raw material used in this study is lime peel powder sized 50 mesh. The water content of raw material on lime peel powder is 76,5 %. The extraction method used in the research is maceration with stirring equal to 150 rpm and Soxhlet with extraction time for 6, 9, and 12 hours. In this study, the ratio of raw material to the solvent is 1:10, and the type of solvent used is hexane, ethanol, and distilled water. The result in this study shows that Soxhlet is the optimum method, which obtains yield equal to 6,15% for 12 using hexane as solvent.

1. Introduction

Most soaps, shampoos, and other bath products contain synthetic fragrances. Most of the fragrance ingredients flow as waste. In most wastewater treatment methods cannot decompose fragrance compounds. These compounds tend to accumulate and are difficult to decompose [1]. Nowadays, consumers prefer natural and environmentally friendly perfumes made from pure, safe ingredients [2]. Therefore, we need materials that can give the aroma or fragrance that many consumers like, which come from natural ingredients. One of the additional components that can be used as a scent is essential oils.

Essential oils can be produced from fruit peels, one of which is lime peel [3]. It is necessary to study the utilization of lime peel waste as a raw material for essential oil to increase the economic value of lime peel waste and to increase the income of lime peel farmers. Various extraction methods are used in the manufacture of essential oils, this method used usually depends on what kind of plant is used [4].

One of them is maceration. Maceration is an inexpensive and widely considered method of obtaining an active ingredient from plants. Maceration is a solid-liquid extraction method in which the bioactive material in plants is extracted by immersing the plant material in a certain solvent for a particular period [5]. Another method that can be used is Soxhlet. Soxhlet has several advantages such as reducing the use of organic solvents, because the sample will be repeatedly contacted with fresh solvent [6]. Another advantage of this method is that it uses more time efficiently. The extraction process will run continuously without having to increase the volume of the solvent so that a more concentrated extract will be obtained [7].

The purpose of this research is to produce essential oil from lime peel so it can be used as a substitute for synthetic fragrance in soap and to find the best method, type of solvent, and extraction time in producing it.
2. Research description
The research begins with the preparation of raw materials. Lime peels were cut into small pieces, then cleaned to remove dirt and microbes. After that, lime peels were dried in the oven until the water content of the raw material is 10%. The dry lime peel was blended and then sieved with a 50 mesh sieve.

For maceration extraction, lime peel powder and solvent were mixed in a ratio of 1:10 (w/v). The solution was stirred using a stirrer with speed of 150 rpm for 6, 9, and 12 hours. The mixture of lime peels powder and solvent were filtered using filter paper. Then the solution, which is a mixture of lime peel oil and the solvent, were separated by a vacuum rotary evaporator.

For Soxhlet extraction, lime peel powder was weighed as much as 20 grams and wrapped in filter paper, and put into a thimble. The solvent was put into a 200 ml flask, then it was heated according to the boiling point of the solvent. The extraction was carried out for 6, 9, and 12 hours. After the extraction is complete, the extract obtained is separated between lime peel oil and solvent using a vacuum rotary evaporator.

In this study, several standard analyzes were carried out such as the yield of oil obtained, the color of oil, oil density using pycnometer, and oil refractive index with a refractometer based on ISO 3519: 2005 (E). The samples used were analyzed to determine the presence of limonene in lime peel powder using FTIR (Fourier Transform Infrared Spectroscopy).

3. Results and Discussions

3.1. Identification of Limonene in Lime Peel Using FT-IR
To prove that the raw material used in this research contains limonene, which is a compound that affects the distinctive aroma of lime peel, a functional group analysis of lime peel was carried out using FTIR (Fourier Transform Infrared Spectroscopy).

According to Auta et al. (2018) and Boughendjoua and Samah (2017), the functional groups that indicate the presence of limonene are C-H (alkane), C-H (aromatic), C-N (aromatic), C-N (aliphatic), and C=C. The functional groups have different wavelengths.

From the FTIR spectrum above, there is an absorption band that shows the groups present in lime peel. The following table shows the functional group data from limonene, the wave number from the functional group as a result of the analysis, and the wave range refers to Pavia et al. (2009).

| Functional Group | Wave Number (cm⁻¹) | Wave Number Range (cm⁻¹) [11] |
|------------------|--------------------|--------------------------------|
| C-H (alkanes)    | 2919,60            | 3000-2850                     |
| C-H (aromatic)   | 3091,83            | 3150-3050                     |
| C-N (aromatic)   | 1318,43            | 1350-1250                     |
| C-N (aliphatic)  | 1232,98            | 1250-1000                     |
| C=C              | 1606,39            | 1680-1600                     |

Table 1. List of Functional Groups within the Limonene
According to the wave numbers obtained from the analysis, it can be concluded that the sample, namely lime peel, contains limonene. The results of this functional group test have the same results as the previous studies conducted by Auta et al. (2018) and Boughendjioua and Samah (2017).

3.2. The Effect of Extraction Time on % Yield of Essential Oil From Lime Peel

In this research, the essential oil extraction from lime peel was carried out using two methods, which was maceration and Soxhlet, using three different solvents, namely hexane, ethanol, and water. The relation that shows the effect of time on yield with the maceration method can be seen in Figure 2. (a), and the Soxhlet method can be seen in Figure 2.(b).

![Figure 2. (a) The Effect of Extraction Time on % Yield of Essential Oil from Lime Peel Using Maceration](image)

Figure 2. (a) shows the extraction using maceration method. The yield of lime peel oil increased with increasing extraction time. At 6, 9, and 12 hours extraction times for hexane, the oil yield was 4.93%, 5.13%, and 5.24%. At the extraction time of 6, 9, and 12 hours for water solvent, the oil yield was 1.51%, 1.59%, and 1.67%. However, in the extraction using ethanol as a solvent, the oil yield fluctuates. In the 6 hours extraction time, the oil yield was 3.69%, then increased in 9 hours to 4.20%, and finally decreased at 12 hours to 4.07%.

![Figure 3. (b) The Effect of Extraction Time on % Yield Using Soxhlet](image)

Figure 3. (b) shows that in the extraction using the Soxhlet method, the oil yield increased along
with the increase in extraction time. At 6, 9, and 12 hours extraction times for hexane, the oil yield increased by 5.79%, 5.97%, and 6.15%. While the extraction using ethanol solvent with extraction times of 6, 9, and 12 hours, the resulting oil yield was 4.41%, 4.64%, and 4.89%. Extraction, using water as solvent, cannot be carried out because there is no circulation in the chiffon section of the Soxhlet tool, causing the thimble to be filled with water so that the water comes out from the F pipe section, which should function as a steam route in the extraction process in Soxhlet extraction.

Extraction time is a factor that influences the extraction process. A longer extraction time generally results in a higher yield. The increase in time will cause an increase in the contact time between the solvent and the lime peel powder solids, thereby facilitating the transfer of the mass of essential oils contained in lime peels to the solvent used [13].

The results obtained by maceration and Soxhlet methods in this study have the same results as previous research by Chairunnisa et al. (2019) on the extraction of essential oils from jujube leaves and research by Giwa et al. (2018) on the extraction of essential oils from the orange peel, which shows that the longer the extraction time, the greater the yield obtained.

### 3.3. The Effect of Extraction Method on % Yield of Essential Oil From Lime Peel

One of the main factors determining oil quality in essential oil extraction is the extraction method. Improper procedures can cause damage to the oil so that natural and bioactive compounds will be lost. The examples of oil that has been damaged can be seen from discoloration of oil, the odor of oil, and other characteristics, so it should be avoided [16].

In this study, the essential oil of lime peel was obtained by two methods. The first method is maceration, using stirring at 150 rpm. The maceration process with stirring can shorten the extraction time to 6 to 24 hours [17]. The second method was Soxhlet, this process requires a heating process with temperature that equal to the boiling point of the type of solvent used. The best lime peel oil yield for each method can be seen in Figure 4.

![Figure 4. The Yield of Lime Peel Oil Using Maceration and Soxhlet Method](image)

Figure 4 shows the highest oil yield using the maceration method and the Soxhlet method were 5.24% and 6.15%, respectively. The highest yield in maceration and Soxhlet methods was obtained with an extraction time of 12 hours using hexane as solvent. From the results obtained, it can be concluded that the Soxhlet method is more effective than the maceration method.

This is similar to the research of Lee et al. (2017) who extracted essential oils from agarwood leaves using two methods, which was maceration and Soxhlet. The highest oil yield obtained using the Soxhlet method. Research from Gahlot et al. (2018) that extracted lychee leaves, curry leaves, jamun leaves, and catechu (a by-product of acacia trees), using maceration and Soxhlet methods, obtained the best yield of oil using the Soxhlet method.
The Soxhlet extraction method is an extraction method that runs continuously with the aid of heating in which the lime peel sample is in direct contact with the solvent at a predetermined extraction time and undergoes circulation. Compared to the maceration method, Soxhlet extraction gives a higher yield [21].

3.4. The Effect of Type of Solvent on % Yield of Essential Oil from Lime Peel

Selection of the right solvent is one of the essential factors in extraction. Selectivity, solubility, cost, and safety must be considered. Solvents with a polarity value close to the polarity of the solute tend to have better performance and vice versa [7]. In this study, essential oil from lime peel was obtained using three solvents, namely hexane, ethanol, and water.

The purpose of using three different types of solvents is to compare the yield of the extracted oil, so that a more effective solvent can be found. Data of the best lime peel oil yield obtained for each type of solvent can be seen in Figure 5.

![Figure 5. The Yield of Lime Peel Oil Hexane, Ethanol, and Water](image)

Figure 5 shows that the highest oil yield was obtained using the Soxhlet method for 12 hours using hexane solvent at 6.15%, using ethanol at 4.89%, and using water solvent at 1.67%. When compared to these three solvents, the hexane solvent is more effective than ethanol and water.

The results of this study are similar to research by Lee et al. (2017), who extracted essential oils from agarwood leaves using four different types of solvents, namely water, ethanol, isopropanol, and hexane, where extraction using hexane as solvents had the highest yield. The solute will dissolve more easily in the solvent if they have a polarity that is close enough.

Lime peel oil is a non-polar compound that tends to dissolve in non-polar solvents as well, where hexane, ethanol, and water have a polarity index of 0.1; 3.9; and 10.2. The greater the polarity index, the more polar the compound. Therefore, hexane will more easily dissolve lime peel oil because it is non-polar so that the yield obtained will be greater than ethanol and water.

However, the extraction using water with Soxhlet method can’t be done. In Soxhlet extraction, there are several requirements of the solvent, namely the solvent is more volatile, the solvent's boiling point is low, and the properties of the compound to be extracted are similar with the solvent. Between hexane, ethanol, and water, water is a solvent that has a higher boiling point and is more difficult to evaporate than the other two solvents. The properties of lime peel oil and water are also different, where water is a polar compound and hexane is a non-polar compound. So that the extraction process with Soxhlet method using water as the solvents cannot be done. Water can be used as a solvent if it is mixed with other nonpolar solvents with a certain ratio, as was done by Febryanto (2017).

3.5. Analysis of the Quality of Essential Oil of Lime Peel According ISO 3519:2005 (E)

The characteristics of the extraction of essential oil from lime peel were analyzed and compared with the quality standard of lime peel oil based on ISO 3519: 2005 (E), as shown in Table 2.
Table 2. Result of Qualitative Analysis of Lime Peel Essential oil

| Parameters                  | Essential Oil of Lime Peel Extracted | ISO 3519:2005(E) Based Lime Peel Essential Oil |
|-----------------------------|--------------------------------------|-----------------------------------------------|
| Color of Oil                | Hexane: greenish-yellow, Ethanol: yellowish-green, Water: yellowish-green | Colorless to greenish-yellow                  |
| Density, 20°C (g/cm³)       | 0.8580                               | 0.858 – 0.866                                 |
| Refractive Index, 20°C      | 1.476                                | 1.474 – 1.477                                 |

Table 2. shows that in general, the essential oil from lime peel produced is still within the range of the essential oil quality standard based on ISO 3519: 2005 (E).

4. Conclusions
1. The lime peel powder sample contains limonene because there are functional groups in the form of C-H (alkane), C-H (aromatic), C-N (aliphatic), and C = C. The best type of solvent is obtained with a hexane solvent. The best oil yield was 6.15%, which was obtained at 12 hours using the hexane solvent method.
2. The best extraction time was obtained at 12 hours. The best extraction method is obtained by the Soxhlet method. The best type of solvent is obtained with a hexane solvent.
3. The resulting lime peel essential oil has a greenish-yellow color for hexane and water solvents and yellowish-green for ethanol solvents. The resulting essential oil has a refractive index of 1.476 and a density of 0.8580 g/cm³.

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