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Quality Characteristics of Clove Leaf Essential Oil Based on Leaf Pretreatment Using the Steam Distillation Method

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ABSTRACT

Cloves are a plant that is often used as an ingredient in cooking or as an addition to cakes. The quite diverse chemical content in clove leaves provides quite open opportunities for use, one of which is being used as a raw material for producing essential oils. The aim of this research is to determine the suitability of the quality of the essential oil obtained in the form of yield, specific gravity, refractive index and solubility in 90% alcohol in accordance with the Indonesian National Standard (SNI) produced from clove leaves using the steam distillation method. This study used an experimental method to evaluate the quality of essential oils obtained from clove leaves through a steam distillation process. The study was conducted in a laboratory with several treatments on clove leaf samples consisting of four variations of leaf shape, namely, whole clove leaves (S1), chopped clove leaves with a size of 50% (S2) and 25% (S3) of the total length of clove leaves, and crushed clove leaves (S4).The parameters of this research are yield, specific gravity, refractive index, and solubility in 90% alcohol. The results of the research showed that the best treatment was obtained from clove leaf samples chopped at 25% of the total leaf length which had the highest average yield and specific gravity, namely 2.88% and 1.061 g/ml. The refractive index value and the average solubility of clove leaf essential oil in each sample treatment met the Indonesian national standard (SNI 06-2387-2006).
Keyword: Clove leaves, Essential oils, Distillation,Quality

ABSTRAK

Cengkeh merupakan salah satu tumbuhan yang sering digunakan sebagai bahan masakan atau tambahan dalam kue. Kandungan bahan kimia yang cukup beragam dalam daun cengkeh memberikan peluang pemanfaatan yang cukup terbuka, salah astunya yaitu digunakan sebagai bahan baku penghasil minyak asiri. Tujuan penelitian ini adalah untuk mengetahui kesesuaian mutu minyak asiri yang diperoleh yang berupa rendemen, berat jenis, indeks bias, dan kelarutan dalam alkohol 90% sesuai dengan Standar Nasional Indonesia (SNI) yang dihasilkan dari daun cengkeh dengan metode destilasi uap. Penelitian ini menggunakan metode eksperimental untuk mengevaluasi kualitas minyak asiri yang diperoleh dari daun cengkeh melalui proses destilasi uap. Penelitian dilakukan di laboratorium dengan beberapa perlakuan pada sampel daun cengkeh yang terdiri dari empat variasi bentuk daun yaitu, daun cengkeh utuh (S1), daun cengkeh dirajang dengan ukuran 50% (S2) dan 25% (S3) dari total panjang daun cengkeh, dan daun cengkeh yang dihancurkan (S4). Parameter penelitian ini yaitu rendemen, berat jenis, indeks bias, dan kelarutan dalam alkohol 90%. Hasil penelitian menunjukkan bahwa perlakuan terbaik diperoleh pada sampel daun cengkeh yang dirajang sebesar 25% dari total panjang daun yang memiliki rata-rata rendemen dan berat jenis tertinggi yaitu 2,88% dan 1,061 g/ml. Nilai indeks bias dan hasil rata-rata kelarutan minyak asiri daun cengkeh di setiap perlakuan sampel memenuhi standar nasional Indonesia (SNI 06-2387-2006).
Keyword: Daun cengkeh, Distilasi, Minyak asiri, Kualitas

Various types of plants grow well and abundantly in Indonesia, especially spices and biopharma plants. One of them is the clove plant (Syzygium aromaticum). Clove plants as a type of spice plant have high economic value, so clove plants always experience an increase in production (Sasongko et al., 2022). Apart from being able to be used to produce essential oil, most of the clove leaves are used in the cigarette industry, ranging from 80-90% (Nurdjannah, 2004).

Based on data from the Central Statistics Agency in 2021, clove production in Indonesia reached 135.7 thousand tons in 2021. This number decreased by 2.44% compared to 139.1 thousand tons in 2020. Based on Indonesian Data in 2022, Maluku is the largest clove producer in Indonesia, reaching 20,454 tons. Maluku also has one of the best types of clove plants with a selling price of up to IDR 125,000/kg. Meanwhile, South Sulawesi is in second place with clove production of 20,144 tonnes, while based on NTB one data, West Nusa Tenggara produces 231.37 tonnes of cloves, with the largest production being in West Lombok Regency at 93.53 tonnes. The part of the clove plant that is used optimally is the flower as raw material for cigarettes. Apart from that, other parts of the clove plant, namely the stem and leaves, contain essential oils that can be utilized, thereby increasing the use value of the clove plant. The most dominant component contained in clove leaf waste and which is the active ingredient is the phenol eugenol (Nurdjannah, 2004). The phenol compound eugenol, has a potential for utilization that has not been fully optimized. One way to increase the utility value of clove leaves is to produce essential oils, which have extensive benefits in various industries.

Clove leaves contain quite a variety of chemicals so there are quite a lot of opportunities for their use, including being used as a raw material for producing essential oils. Essential oil is a type of vegetable oil that has many benefits. The physical characteristics of essential oils are that they are a thick liquid that can be stored at room temperature. To produce essential oils, the raw materials used are obtained from various parts of plants such as leaves, flowers, fruit, seeds, seed coats, stems, and roots or rhizomes. One of the main characteristics of essential oils is that they are easy to evaporate and have a distinctive aroma. Therefore, this oil is widely used as a basic ingredient for making perfumes and cosmetics (Rusli, 2010).

Isolation of essential oils is an attempt to separate essential oils from plants or plant parts of origin. The essential oils in plants are found in the inside of the hair glands and gland cells. Essential oils can only be separated from plant cells if water vapor or other solvents reach the oil, which will then cause the oil droplets to evaporate together. To obtain essential oils, the parts of the plant used must be reduced first. The smaller the size of the material used, the faster the contact of the essential oil with water vapor (solvent) (Koensoemardiyah, 2010). Reducing the size of the material can facilitate the release of essential oils after the material is penetrated by steam (Sastrohamidjojo, 2004).

It is hoped that the essential oil produced will be of good quality. The quality of essential oils can be seen from their natural characteristics and whether or not foreign objects or materials are mixed in them. Apart from that, other supporting factors that can determine the quality of essential oils include: physico-chemical properties of the essential oil, type of raw material used, harvest age, pre-treatment of the material before the refining process, type of equipment used and processing conditions, treatment of the oil obtained after refining, type of packaging, and storage (Nugraheni, 2012).

There are three methods in the distillation process, namely the water distillation method, the water and steam distillation method and the steam distillation method. The water distillation method has the advantage that the process is easier because it uses a very simple method, namely boiling and the time required is short. The advantage of the water-steam distillation method is that it requires only a small amount of water so the time required is shorter and the equipment used is simple so it is more efficient. The advantages of distillation using the direct steam distillation method are that it requires a relatively short process time, so it has a higher distillation efficiency, the resulting yield is high and the quality of the resulting yield is also high because it does not mix with water, but requires more expensive and complex equipment (Porawati & Kurniawan, 2019).

Based on this description, research was carried out on refining clove leaf essential oil (Syzygium aromaticum) using the water and steam distillation method to produce essential oil in accordance with the Indonesian National Standard (SNI).

1.1. Research Purposes
To determine the effect of treatment on the suitability of essential oil quality in the form of yield, specific gravity, refractive index and solubility in 90% alcohol in accordance with the Indonesian National Standard (SNI) produced from clove leaves using steam distillation methods.

2. Research Methods

2.1. Tools and materials

The tools used are a set of distillation tools, stopwatch, digital thermometer (type MB558 and type NTC 10K/3435), separating funnel (extraction tool), funnel, beaker, measuring cup, dropper pipette, digital refractometer (type DR301-95), vials, analytical scales, gas stoves, LPG cylinders, scissors, buckets and pump machines. Meanwhile, the ingredients used are dried clove leaves, water and ice cubes.

2.2. Method

The research method used in this research is an experimental method by conducting experiments in the laboratory. Dried clove leaves were given four treatments, namely, whole leaves (S1), chopped leaves at 50% (S2) and 25% (S3) of the total length of clove leaves, and crushed leaves (S4). The ingredients have been treated, then the ingredients are weighed before being put into the kettle in the amount of 500 grams. Distillation of the material was carried out for 2 hours with 2 repetitions of each treatment using the steam distillation method. Next, the distillation kettle is heated so that the oil will be released from the oil glands in the leaf tissue. The resulting distillate is then collected in a container (measuring cup) and separated between water and oil using an extraction tool. After the oil has been separated from the water, an analysis is carried out on the quality of the essential oil produced in the form of yield, specific gravity, refractive index and solubility in 90% alcohol.

2.3. Research Parameters

The parameters used in this research are as follows:
Rendement
The yield of the separated essential oils is transferred into vials, the yield of each essential oil obtained is calculated. (Holili, 2021) said, that the measurement of yield aims to determine the comparison of the amount of product produced with the materials used in percentage form. The process of determining the yield of essential oils is carried out by comparing the products produced in the form of essential oils with the samples used. The yield is calculated using equation 1 (Zaituni et al., 2016):

$$\text{Rendement (\%)} = \frac{\overline{bm}}{\overline{bb}} \times 100\% \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

information:
bm: Weight of refined oil (g)
bb: Weight of material used (g)

Specific gravity
This method is based on a comparison between the weight of oil at a specified temperature and the weight of water at a volume of water equal to the volume of oil at a specified temperature. The specific gravity testing procedure is, the vial is washed, cleaned and dried, then weighed (m), then the vial is filled with distilled water first. After that the vial is weighed with its contents (m_1). The vial is emptied, washed and dried. Next, the vial is filled with the oil sample and weighed (m_2). Specific gravity is calculated using equation 2 (Zaituni et al., 2016):

$$BJ = (m_2\text{-}m)/(m_1\text{-}m) \ldots\ldots\ldots\ldots\ldots\ldots (2)$$

information:
BJ : specific gravity
m : mass of empty vial (g)
m_1 : mass of vial containing distilled water (g)
m_2 : mass of the vial containing the sample (g)

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Refractive Index

Refractive index testing can be used to determine the purity of essential oils. Measuring the refractive index using a refractometer is the same as measuring the brix. The measurement starts from calibrating the instrument with distilled water, then the sample is dropped on the prism of the refractometer, then the results will be immediately read on the refractive index scale. The refractive index is measured using a digital refractometer (type DR301-95) producing more accurate readings.

Solubility in Alcohol 90%

The solubility of clove leaf oil in alcohol is the solubility of the oil in alcohol at a certain concentration which is expressed in a ratio to the clear state. Solubility in alcohol can be tested by mixing clove leaf oil with 90% alcohol at a concentration of 1:1 (1 ml : 1 ml), then shaking until a solution is as clear as possible.

3. Results and Discussion

3.1. Rendement

The yield measurement aims to determine the percentage of oil in the material that can be isolated under certain conditions that are used as treatment. The average yield from clove leaf distillation can be seen in Table 1.

Table 1. Average Results of Clove Leaf Essential Oil Yield (%)

Treanment	Test		Total	Average
	1	2		
S1	1,52	1,56	3,08	1,54
S2	2,33	3,23	5,56	2,78
S3	2,97	2,78	5,75	2,88
S4	2,24	2,29	4,53	2,27

The graph of the results of observations of the average yield from distillation of clove leaves can be seen in Figure 1 below:

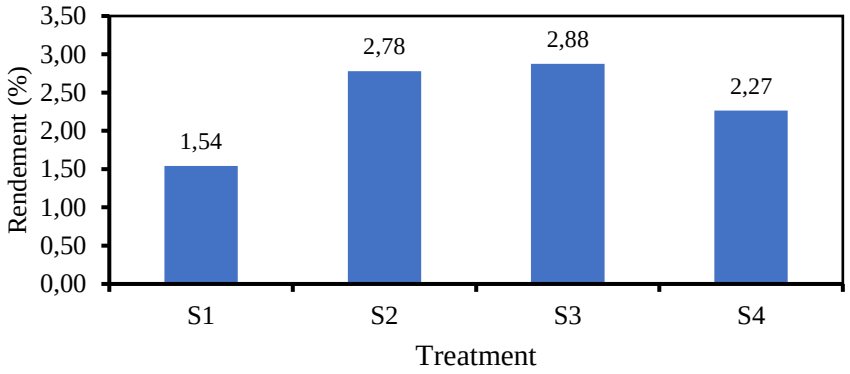

Figure 1. Average yield of clove leaf essential oil

S1: Whole Leaf
S2: Leaves Chopped 50%
S3: Leaves Chopped 25%
S4: Crushed Leaves

Based on Figure 1, the results of the analysis show that in sample 3 with the leaf treatment chopped by 25% of the total leaf length, the highest average yield was 2.88%. Then the second highest average yield was produced in sample 2 with the chopped leaf treatment at 50% of the total leaf length, namely 2.78% and the third highest average yield was produced in sample 4 with the crushed leaf treatment, namely 2.27%, while the average The lowest average yield was produced in sample 1 with whole leaf treatment at 1.54%. This difference occurs because in materials treated with whole leaves, the oil cells are still closed so that the process

of releasing oil from the leaves is still difficult, which causes little oil to come out.

According to (Feriyanto et al., 2013), chopping is an effort to expand the evaporation area and contact with water so that essential oils are more easily extracted. The high yield in the chopped leaf treatment is 25% of the total length of the leaf, because the smaller the size of the leaf being chopped, it can cause the oil pockets to break quickly, causing the oil to come out easily and evaporate when it comes into contact with water vapor. (Hernawati et al., 2012) said, that the smaller the size of the leaves being chopped, the greater the resulting yield. Apart from that, the chopping process causes the hydrodiffusion process to run faster. This is in line with research by (Perdana et al., 2015), which shows that a distillation or refining process using smaller material sizes tends to produce a higher yield of essential oils. This was different in sample 4 with the crushed leaf treatment which produced the third largest average yield. This is in line with research by (Kurniawan et al., 2016) which states that if the raw materials used are too fine during the distillation process, steam channels can form between the materials in the kettle, which can reduce distillation efficiency, which is caused by contact between steam. with the ingredients in the kettle not being perfect.

According to (Utomo & Mujiburohman, 2018), the first factor that affects the yield of essential oils is the sample size, small samples have a larger surface area so that there is more contact with the solvent that will extract the essential oil. The second factor is the length of distillation time, the longer the time used for distillation, the greater the yield of essential oils will be, and the third factor is the condition of the sample. Leaf samples with wet conditions or high water content will reduce the yield of essential oils produced because they contain too much water so that the extraction of essential oils is less than optimal.

3.2. Specific gravity

Specific gravity is the ratio between the weight of oil at a certain volume and the weight of distilled water (distilled water) at the same volume and temperature. The specific gravity of an oil is influenced by the ratio of the components that make up the oil. If components that have a high molecular weight are present in larger quantities, the specific gravity value of the oil will be higher. Specific gravity is also an important criterion in determining the quality of essential oils (Nugraheni, 2012).

The graph of the results of observations of the average specific gravity of clove leaf distillation can be seen in Figure 2 below:

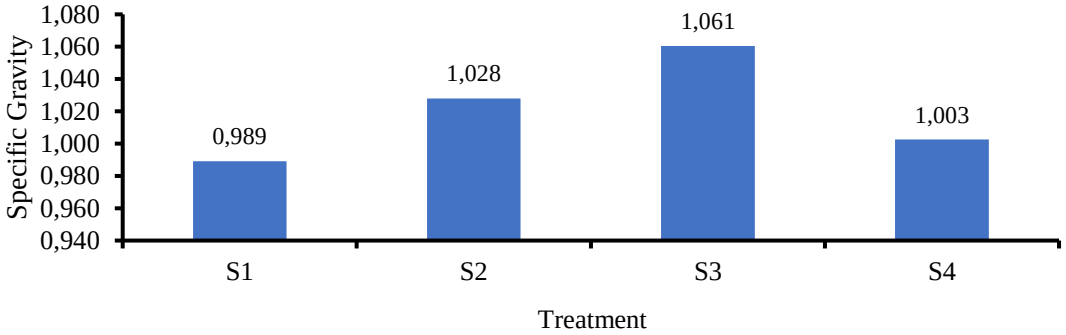

Figure 2. Results of the average specific gravity of clove leaf essential oil

S1: Whole Leaf
S2: Leaves Chopped 50%
S3: Leaves Chopped 25%
S4: Crushed Leaves

Based on Figure 2, the results of the analysis of the specific gravity of clove leaf oil show that in sample 3 with the leaf treatment chopped 25% of the total leaf length produced the highest average specific gravity, namely 1.061 g/ml. Then the second highest average specific gravity was produced in sample 2 with the leaf treatment chopped at 50% of the total leaf length of 1.028 g/ml. Meanwhile, sample 1 with whole leaf treatment produced the lowest specific gravity value, namely 0.989 g/ml. Then the second lowest specific gravity value was produced in sample 4 with crushed leaf treatment of 1.003 g/ml. Based on the Indonesian National Standard (SNI) for clove leaf essential oil, the specific gravity values produced in samples 2 and 3 with chopped leaf

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treatment were 50% and 25% of the total leaf length in accordance with SNI 06-2387-2006 specific gravity of clove leaf oil, namely 1.025-1.049 g/ml. Meanwhile, samples 1 and 4 with whole and crushed leaf treatment had specific gravity values lower than the values set by SNI.

The specific gravity of essential oils is often related to the component content in them. According to (Khasanah et al., 2015), the higher the concentration of essential oil components, the higher the specific gravity. So, the high specific gravity value of oil is determined by the oil components contained in it. Apart from that, the low specific gravity can be influenced by the heavy fraction that is soluble in water. This heavy fraction will remain in the distilled water, because the separation method used (using a separating funnel) to separate essential oils and water is difficult to separate water-soluble compounds (Yuliarto, 2012).

3.3. Refractive Index

The refractive index of a substance is the ratio of the speed of light in the substance to the speed of light in air. The refractive index can also be expressed as the ratio of the angle of incidence of the ray to the sine of the angle of refraction. The refractive index of an oil will determine its level of purity. Oil that is mixed with other ingredients or other components that are soluble in oil will change the refractive index value of the oil in question.

A graph of the results of observations of the average refractive index from clove leaf distillation can be seen in Figure 3 below:

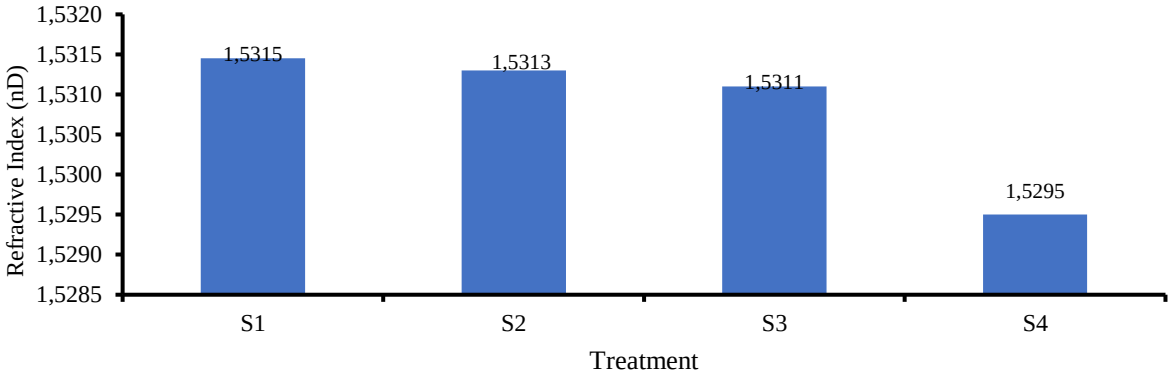

Figure 3. Average Results of Refractive Index of Clove Leaf Extract Oil

S1: Whole Leaf
S2: Leaves Chopped 50%
S3: Leaves Chopped 25%
S4: Crushed Leaves

Based on Figure 3, the highest refractive index value was obtained in sample 1 with whole leaf treatment of 1.5315 nD. Then, the second highest refractive index value was produced in sample 2 with the leaf treatment chopped at 50% of the total leaf length of 1.5313 nD. Meanwhile, the lowest refractive index value was produced in sample 4 with crushed leaf treatment of 1.5295 nD. Then, the second lowest refractive index value was produced in sample 3 with the leaf treatment chopped 25% of the total leaf length of 1.5311 nD. Based on SNI 06-2387-2006, the refractive index of clove leaf oil is 1.528-1.535, so the refractive index produced in this research is in accordance with SNI 06-2387-2006.

The refractive index of clove leaf oil is closely related to the components contained in the clove leaf oil produced. According to (Meyla Aryawati & Nyuwito, 2017), the refractive index is influenced by the length of the carbon chain and the number of double bonds. The higher the refractive index value, the longer the carbon chain and the number of double bonds. One of the causes of low refractive index values is due to the high water content in oil because light is easily refracted in water compared to oil due to the difference in liquid density. Therefore, essential oils with a large refractive index have better purity and quality than essential

oils with a small refractive index because there is less water in the essential oil. The treatment of materials before refining and the method of extracting clove oil are also factors that can influence the refractive index value of the oil (Zainurin et al., 2018). So clove leaf oil with a higher refractive index value will be better than clove leaf oil with a low refractive index value.

3.4. Solubility in Alcohol 90%

The solubility of essential oils in alcohol is determined by the type of chemical components the oil contains. The solubility of oil in alcohol indicates the purity of the oil, if oil is mixed with other ingredients the solubility of oil in alcohol will be high. The solubility of oil in alcohol indicates the polarity of the oil. The average solubility in 90% alcohol in clove leaf oil can be seen in Table 2 below:

Table 2. Results of Average Solubility of Clove Leaf Essential Oil in Alcohol

Treatment	Solubility in Alcohol 90%	color
Whole leaves	1:1	clear yellowish
leaves chopped (50%)	1:1	clear yellowish
leaves chopped (25%)	1:1	clear yellowish
crushed leaves	1:1	clear yellowish

Based on Table 2, the results of the average solubility of clove leaf essential oil show that the solubility level in 90% alcohol is the same. Testing with a ratio of 1 ml of clove leaf essential oil to 1 ml of 90% alcohol, produced a clear yellowish colored solution in each sample treatment which showed that the oil was soluble in a ratio of 1:1. Meanwhile, according to the Indonesian National Standard (SNI), the solubility ratio of clove leaf essential oil is 1:2. From these results it can be seen that the quality of the clove leaf essential oil analyzed is still in accordance with the Indonesian National Standard (SNI 06-2387-2006), so that the solubility of the clove leaf essential oil does not exceed the specified SNI ratio. This shows that the polarity of the oil is quite low and the solubility of the oil in alcohol is quite small. According to (Syarifah, 2017), the smaller the solubility of the essential oil in alcohol (usually 90% alcohol), the better the quality of the essential oil.
The decrease in solubility of oil in alcohol depends on the type of chemical components contained in the oil. Essential oils that contain lots of oxygenated hydrocarbon compounds are more soluble in alcohol and non-oxygenated hydrocarbons are not easily soluble in alcohol. This solubility is based on the level of polarity of a compound that makes up the essential oil (Sebayang, 2011).

3.5. Conclusions and Recommendations

3.5.1. Conclusions

Based on the results of the research that has been carried out, it can be concluded that the best treatment was obtained in sample 3 with the leaves chopped at 25% of the total leaf length which had the highest average yield and specific gravity, namely 2.88% and 1.061 g/ml. The refractive index value and the average solubility of clove leaf essential oil in each sample treatment met the Indonesian national standard (SNI 06-2387-2006).

3.5.2. Suggestion

Based on the results of the research that has been carried out, it is recommended for future researchers to carry out pretreatment on clove leaves to produ ce a more optimal yield and quality of essential oil.

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