EFFECTS OF ELECTRIC VOLTAGE AND WATER VOLUME ON OHMIC HEATING HYDRODISTILLATION (OHHD) OF NUTMEG LEAVES
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PENGARUH TEGANGAN LISTRIK DAN VOLUME AIR PADA HIDRODISTILASI OHMIK HEATING DAUN PALA

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ABSTRACT
The objectives of the study were: to determine the ratio of electrical voltage and volume to the hydrodistillation rate, conductivity, energy, and yield. The method used is hydrodistillation of nutmeg leaves according to the treatment. The treatment of voltage and water volume were 3 levels each. Observations included voltage, current, temperature, and yield. The results showed that the hydrodistillation rate, electrical conductivity, energy, and oil nutmeg leaves yield were influenced by electrical voltage and solvent volume. The maximum oil yield was 1.07% at a voltage of 50 Vac and 750 ml of water.

ABSTRAK
Tujuan penelitian adalah: mengetahui rasio tegangan listrik dan volume terhadap laju hidrodistilasi, konduktivitas, energi, dan rendemen. Metode yang digunakan adalah melakukan hidrodistilasi daun pala sesuai perlakuan. Perlakuan tegangan dan volume air masing-masing 3 level. Pengamatan meliputi tegangan, arus, suhu, dan rendemen. Hasil penelitian menunjukkan bahwa laju hidrodistilasi, konduktivitas listrik, energi, dan rendemen minyak daun pala dipengaruhi oleh tegangan listrik dan volume pelarut. Rendemen minyak maksimum sebesar 1,07% pada tegangan 50 Vac dan air 750 ml.

INTRODUCTION
Distillation is one of the methods used to extract distillates in the form of oil or other materials contained in certain materials. Distillation is the process of heating a liquid or solid until it turns into steam, which is channeled into a separate vessel, then condensed with a cooler. In distillation, a mixture of substances is boiled so that it evaporates, and this steam is cooled back into liquid form. Substances that have a lower boiling point will evaporate first.

The distillation method includes physical operations that involve heat transfer and mass transfer. There are 2 types of distillation methods, i.e., the dry distillation method and the wet distillation method. The dry distillation method is carried out without using water, while wet distillation uses water. There are 3 types of water distillation methods, i.e., the material is boiled in water in one vessel, the material is steamed using water vapor in one vessel, and the material is steamed with water vapor in a separate vessel. Each of the three methods has advantages and disadvantages for the results and quality of the distillate produced. These three methods are commonly used to extract materials from plants containing essential oils.

Plants that produce essential oils include cloves, nutmeg, patchouli, and so on. Essential oils have a selling value and are among Indonesia's leading export commodities. According to the latest report from the Ministry of Industry, Indonesia's essential oil production in 2020 reached around 8,500 tons. From 2017 to 2020, an increase in the value of essential oil exports was recorded, with a total value of essential oil exports from January to September 2021 reaching USD 185 million (*Mukhtarom*, *2024*). Nutmeg is a plant that produces essential oils. Indonesia is the largest producer of nutmeg in the world, i.e., between 70% and 75% *(Balai Komoditi Industry, Deptan, 2009*). Essential oil from nutmeg plants can be obtained from fruit, mace, and nutmeg leaves. The highest oil content is obtained from fruit and mace, while nutmeg leaves have a low content. The yield of essential oil from 1 kg of fresh nutmeg leaves is 0.4% (*Damayanti, 2017*). Although the content of nutmeg leaf oil is small, if the amount is large, it will still provide economic value.

The extraction of nutmeg leaf essential oil by craftsmen/farmers is done by distilling a mixture of steam and water. Steaming the leaves in a closed vessel produces a mixture of water vapor, which is then routed into a cooling pipe for condensation. The condensation results in a liquid consisting of water and nutmeg leaf oil. The energy used for steaming nutmeg leaves comes from firewood. The use of firewood for the distillation process is not environmentally friendly because it can pollute the environment due to the smoke from burning firewood.

Currently, many researchers are developing environmentally friendly distillation and extraction methods. One distillation method is using ohmic heating. Ohmic heating is a heating process that occurs due to alternating electric current in a liquid that has conductivity. Ohmic heating (OH) is a volumetric heating technique that emerged as an alternative to conventional heating by utilizing alternating electric current to heat the product (*Sain et al., 2024; Talha et al., 2024*). Heat will be generated in the liquid due to the collision of particles in the liquid. Heating in this way is environmentally friendly because it does not produce smoke and other hazardous materials. Research utilizing ohmic heating for extraction includes the extraction of phytochemical component products from potatoes (*Pereira et al., 2016*), the extraction of essential oils of aromatic plants (*Gavahian et al., 2020*), the extraction of olive leaves (*Safarzadeh et al., 2022*), the effectiveness of ohmic heating technology for the extraction of phenolic compounds from red beet roots (*Cabas and Icier, 2021*), grape pomace (*Ferreira-Santos et al., 2024*), the extraction of phenolic content and antioxidant activity of cocoa bean skin (*Sánchez et al., 2023*), and the phenolic extraction of avocado leaves (*Gumustepe et al., 2023*).

The conditions for heating in ohmic heating are the presence of electrical voltage and liquid materials that have conductivity values. The mixture of water and nutmeg leaves has a conductivity value, so if given an electric voltage, it will be able to flow an electric current. The electric current flowing in the mixture of water and nutmeg leaves will generate heat. The heat will increase the temperature of the mixture of water and nutmeg leaves until it reaches boiling point (100°C). The water vapor formed contains a mixture of water and oil; if condensed, it will produce separate water and oil. The use of ohmic heating for nutmeg leaf extraction can be done using the water distillation or hydrodistillation method. Hydrodistillation is done by boiling essential plant materials and water in one vessel. The success of ohmic heating is influenced by many factors such as electrical conductivity, field strength, particle size, concentration, ionic concentration, and electrodes (*Kaur & Singh, 2016*).

The electrode in ohmic heating is a connection between a solid-state conductor (i.e., the current feeder) and a liquid-state conductor (i.e., the heating medium) (*Samaranayake & Sastry, 2005*). There are several types of electrodes used for heating using ohmic heating, i.e., aluminum (*Uemura et al., 1994*), carbon (*Getchell, 1935*), glassy carbon, titanium (*Amatore et al., 1998*), platinum (*Tzedakis et al., 1999*), rhodium (*Palaniappan & Sastry, 1991*), stainless steel (*Assiry et al., 2003*), and platinized titanium (*Stirling, 1987*). Based on the description above, the author is interested in knowing the amount of electrical energy needed in the process of hydrodistillation of nutmeg leaves using ohmic heating. The use of different electrical voltages and electrode areas will affect the energy and also the time for the distillation process of nutmeg leaves. The objectives of this study are (a) to determine the effect of voltage and water volume on the hydrodistillation rate, (b) to determine the effect of voltage and water volume on electrical conductivity, (c) to determine the effect of voltage and water volume on hydrodistillation energy consumption, and (d) to determine the effect of voltage and water volume on the yield of nutmeg leaf oil.

MATERIALS AND METHODS

Materials

The ohmic heating hydrodistillation equipment used consists of an outer pan made of aluminum and an inner pan made of stainless steel. The outer pan functions as a protector. The inner pan is a treatment room as a heating place. There are 2 inner pans, i.e., the one located on the edge with an open position with a height of 106 mm and a diameter of 182 mm. This pan functions as a place for materials and water for the boiling process as well as a neutral electrode. The second pan with an open position with a diameter of 88 mm and a height of 82 mm is located in the middle and functions as a phase electrode (fig. 1). Other equipment needed is a 1000 W step-up/down transformer, ampere meter, volt meter, measuring cup, analytical balance, camera, and stopwatch. The material used is dried nutmeg leaves. The treatments carried out were 3 voltage levels, i.e., 50 Vac, 75 Vac, and 100 Vac, and 3 levels of water volume treatment, i.e., 750 ml, 1000 ml, and 1250 ml.

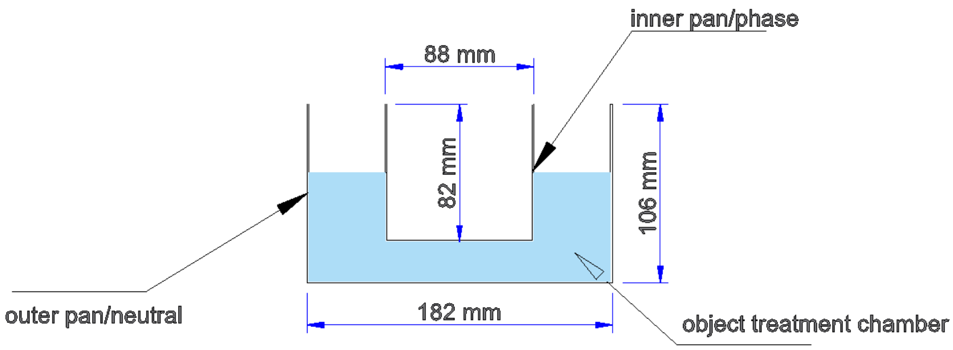

Fig. 1 - Treatment chamber scheme

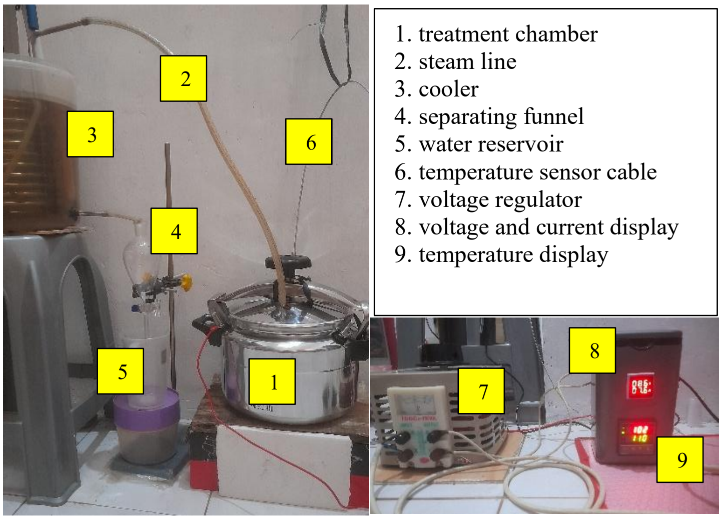

Fig. 2 - Ohmic heating device

Research stages

1. Designing and assembling the 1.3 liter ohmic heating hydrodistillation device (fig. 2)

2. Testing the ohmic heating hydrodistillation device by (a) Weighing 100 g of dried nutmeg leaves and cutting them into several parts. (b) Inserting the cut dried leaves into the treatment chamber and adding distilled water according to the treatment. (c) Closing the treatment chamber and flowing alternating current with the voltage according to the treatment. (d) Turning on the switch so that the hydrodistillation process takes place. (e) Recording the temperature, voltage, and electric current during ohmic heating hydrodistillation. (f) Collecting oil and water from the hydrodistillation process. (g) Separating the water and nutmeg leaf oil using a separating funnel. (h) Weighing the nutmeg leaf oil. (i) Conducting ohmic heating hydrodistillation of nutmeg leaves with different voltages and ratios of water and nutmeg leaves. (j) Repeating the steps above according to the treatment, and each is repeated 3 times.

3. Conduct observations on changes in voltage, changes in electric current, changes in temperature, oil yield, and oil characteristics according to SNI.

4. Process data and present it in the form of tables and graphs.

5. Electrical conductivity (σ) is calculated using the following equation (*Assiry et al., 2003; Zell et al., 2009; Salengke et al., 2007*).

$$\sigma = \frac{L}{A} x \frac{I}{V}$$

where:

σ = Electrical conductivity (S/m);
L = distance between electrodes (m);
A = area of the material to be heated (m^2);
I = electric current (Amperes);
V = Electric voltage (Volts).

RESULTS

Voltage

The requirement for heating in ohmic heating hydrodistillation is the presence of electric voltage flowing in the liquid to be heated. The liquid must have a conductivity value; if the conductivity approaches zero, then the electric current does not flow and will not generate heat. The mixture of water and nutmeg leaves has a conductivity value, so if given an electric voltage, it will be able to flow an electric current. The electric current flowing in the mixture of water and nutmeg leaves will generate heat. One of the factors that affects the success of the hydrodistillation process is electric voltage. In this study, 3 voltage levels were used, i.e., 50 Vac, 75 Vac, and 100 Vac. The volume of water used was 3 levels, i.e., 750 ml, 1000 ml, and 1250 ml. The results of the study on the treatment of electric voltage and water volume are presented in Fig. 3 and Fig 4.

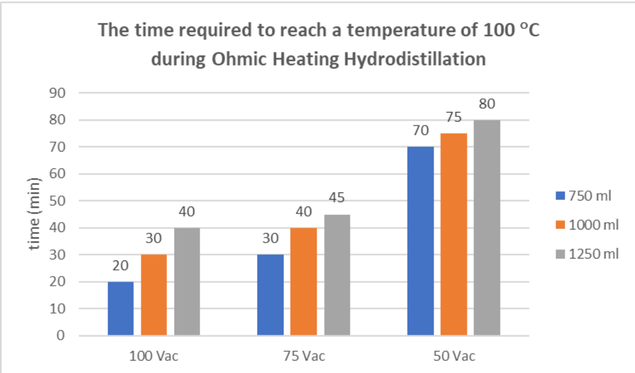

Fig. 3 - Time required to reach a temperature of 100°C

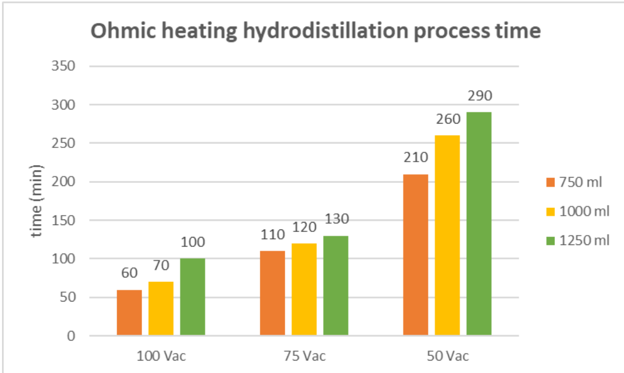

Fig. 4 - Time required for ohmic heating Hydro distillation process

Based on Fig. 3, it can be seen that the magnitude of the voltage affects the heating of the mixture of nutmeg leaves and water. At a voltage of 100 Vac, the heating time to reach a temperature of 100°C requires a shorter time compared to a voltage of 75 Vac and a voltage of 50 Vac. At the same water volume of 750 ml, the time required to reach a temperature of 100°C at a voltage of 100 Vac is 20 minutes; at a voltage of 75 Vac, it takes 30 minutes; and at a voltage of 50 Vac, it takes 70 minutes. At a water volume of 1000 ml, with a voltage of 100 Vac, it takes 30 minutes to reach a temperature of 100°C, while at a voltage of 75 Vac, it takes 40 minutes, and at a voltage of 50 Vac, it takes 75 minutes. At a larger water volume of 1250 ml, the time to reach a temperature of 100°C at a voltage of 100 Vac is 40 minutes, while at a voltage of 75 Vac it is 45 minutes, and at a voltage of 50 Vac it is 80 minutes. The higher the voltage, the faster the time to reach the boiling point of the liquid.

From Fig. 3, it can be seen that at the same voltage, the time required for ohmic heating hydrodistillation process is influenced by the volume of water used. The greater the volume of water used, the longer the time to reach the boiling point. The fastest time to boil a mixture of water and nutmeg leaves is at a volume of 750 ml, followed by 1000 ml, and the longest is 1250 ml. The higher the volume of water, the heating rate will decrease. The heating process using ohmic heating is influenced by many factors such as electrical conductivity, field strength, particle size, concentration, ionic concentration, and electrodes (*Kaur & Singh, 2016*).

Based on Figure 4, it can be seen that at the same voltage, the hydrodistillation process is influenced by the voltage and water used. The higher the voltage and the less volume of water used, the shorter the hydrodistillation process will be. This is like using a voltage of 100 Vac with 750 Watts; the hydrodistillation process takes 60 minutes. At a low voltage of 50 Vac with a water volume of 1250 ml, the longest time is required during the hydrodistillation process, which is 290 minutes. At a higher voltage of 100 Vac, the heat generation process is faster, while at a lower voltage of 50 Vac, the heating process is slow.

Electrical conductivity

Electrical conductivity (σ) is a measure of a material in accommodating the movement of an electric charge, i.e., the ratio of current density to electric field strength. The unit of electrical conductivity is Siemens per meter (S/m) (*Assiry et al., 2003; Salengke & Sastry, 2007; Zell et al., 2009*). Electrical conductivity is the ability of a material to conduct heat and electricity. Electrical conductivity works based on the physical movement of electrons and the transfer of energy between electrons. The existence of electrical conductivity is very necessary in ohmic heating. The requirement for the heating process in ohmic heating, in addition to the presence of electric voltage, is that the liquid must have a conductivity value. The electrical conductivity value is influenced by the content of conductive materials in the liquid, such as acid, base, salt, or materials containing electrolytes such as silver, copper, gold, aluminum, zinc, nickel, and brass.

The measurement of the initial electrical conductivity value of a mixture of distilled water and nutmeg leaves before the hydrodistillation process using ohmic heating averaged 34 µS/cm. Pure water has a conductivity close to zero because it contains little electrolyte (*Asgarpanah & Kazemivash, 2012*). The addition of nutmeg leaves to pure water can increase the conductivity value even though the value is relatively small. The electrical conductivity value of the mixture of water and nutmeg leaves during the hydrodistillation process varies depending on the volume of water, voltage, and temperature. The results of the calculation of the electrical conductivity value during the hydrodistillation process of nutmeg leaves using ohmic heating on the same water volume are presented in Fig. 5–Fig. 7.

In general, at the start of heating, the electrical conductivity of the same volume of water is low, but it increases with process time. Each voltage's electrical conductivity has a maximum value, which decreases over time. The higher the voltage, the faster it reaches its maximum conductivity value. The magnitude of conductivity during the ohmic heating hydrodistillation process at the same volume of water is affected by the magnitude of voltage.

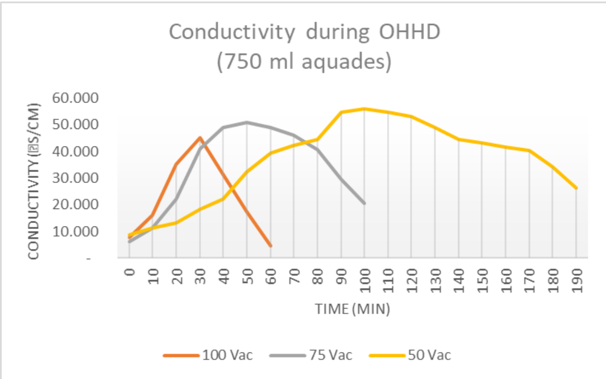

Fig. 5 - Conductivity during OHHD (750 ml of distilled water)

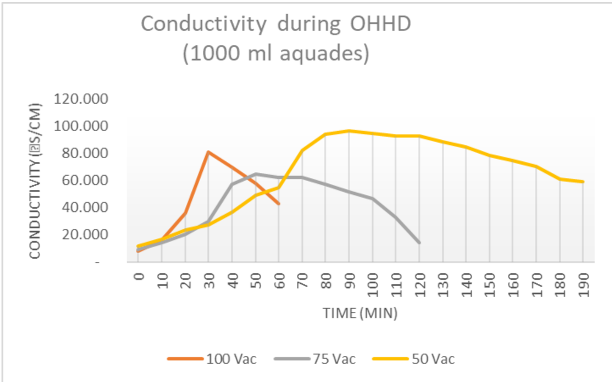

Fig. 6 - Conductivity during OHHD (1000 ml of distilled water)

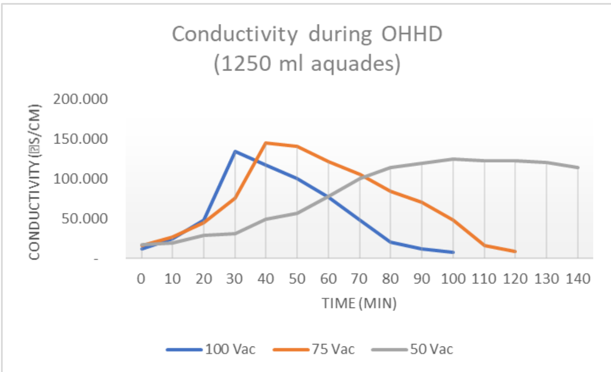

Fig. 7 - Conductivity during OHHD (1250 ml of distilled water)

Electrode area

Electrode area in ohmic heating are important components for heat to occur. Without electrodes, there will be no heating. The electrodes used in ohmic heating are selected from materials that do not change properties (corrosive) due to electricity flowing and do not dissolve in the heated solution. In this study, stainless steel was used as the electrode because it has corrosion-resistant properties (*Assiry et al., 2003*).

The electrodes used are a pair of parallel plates in the form of a cylinder/tube. The inner and outer electrodes have different areas. The cavity between the two electrodes functions as a place for agricultural materials that are given heating treatment or a hydrodistillation chamber. The outer electrode (neutral) has a tube cover area of 592.2 cm² with a tube base area of 253.2 cm², while the inner electrode (phase) has a tube cover area of 227.1 cm² and a base area of 61.1 cm². The area of the outer electrode (blanket and base) is 845.4 cm², and the area of the inner electrode (blanket and base) is 288.2 cm². When given different treatments of adding water and nutmeg leaves, the electrode plates immersed in water are also different. The difference in the water that is immersed causes a difference in the contact area of the electrode or a difference in the electrode area. Table 1 presents the relationship between water volume and electrode surface area.

Table 1

Electrode area

water volume (ml)	water level (cm)	outer electrode area (cm²)	inner electrode area (cm²)	average electrode area (cm²)
750	*3,9*	*473,1*	*105,4*	*289,3*
1000	*5,1*	*540,8*	*138,6*	*339,7*
1250	*6,7*	*631,0*	*182,9*	*407,0*
full	*10,5*	*845,4*	*288,2*	*566,8*

The electrode area, or the area of contact between the object and the electrode, also affects the success of ohmic heating hydrodistillation. The electrode area will affect the value of electrical conductivity. The results of the calculation of the electrical conductivity value during the nutmeg leaf hydrodistillation process using ohmic heating at the same electrical voltage are presented in figures 8, 9, and 10. Based on the figure, in general, at the same voltage, the conductivity value is influenced by the volume of water used. The greater the volume of water used, the greater the conductivity value.

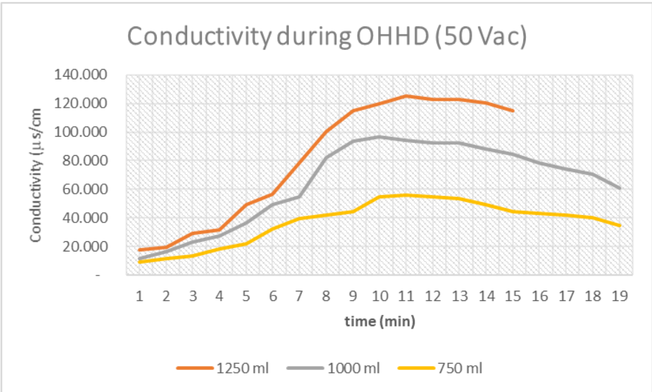

Fig. 8 - Conductivity during OHHD (Voltage 50 Vac)

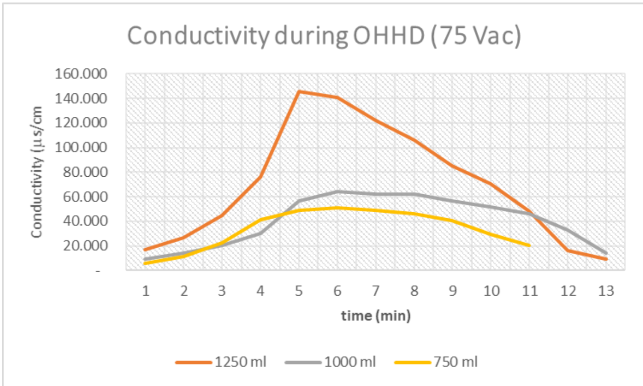

Fig. 9 - Conductivity during OHHD (Voltage 75 Vac)

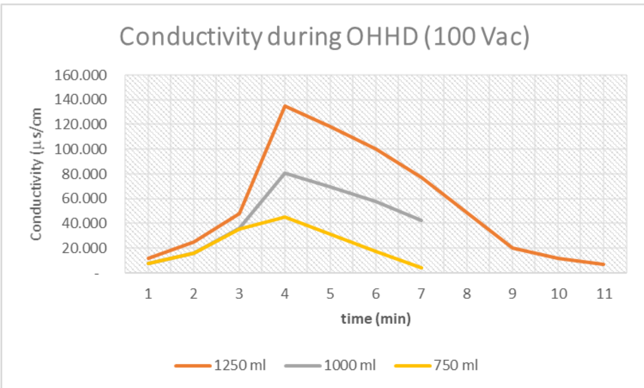

Fig. 10 - Conductivity during OHHD (Voltage 100 Vac)

The greater the volume of water used, the higher the water level in the treatment chamber. The higher the water level in the treatment chamber, the greater the surface area of the electrode. So, at the same voltage, the wider the electrode surface, the greater the electrical conductivity value. Electrical conductivity in the ohmic heating hydrodistillation process is influenced by the distance between electrodes, surface area, electric current, and electric voltage (*Zell et al., 2009*).

During the ohmic heating hydrodistillation process, energy is required from electricity. The electric current is converted into heat energy to boil the mixture of water and nutmeg leaves. In ohmic heating hydrodistillation, heating occurs due to the electric current in the mixture of water and nutmeg leaves. The heating rate is influenced by the amount of voltage and the volume of water used. In this study, 3 different voltage levels were used, i.e., 50 Vac, 75 Vac, and 100 Vac, and 3 levels of water volume that affect the duration of the heating process. Both factors will affect the amount of energy required during the ohmic heating hydrodistillation process. The amount of energy during ohmic heating hydrodistillation is as shown in Fig. 11 and Fig. 12.

The amount of hydrodistillation energy varies from each water volume treatment and voltage treatment. Figure 11 shows that the energy consumption pattern for a water volume of 1250 ml and a water volume of 1000 ml gives the same results. At the same water volume, the higher the voltage, the greater the energy required during the process.

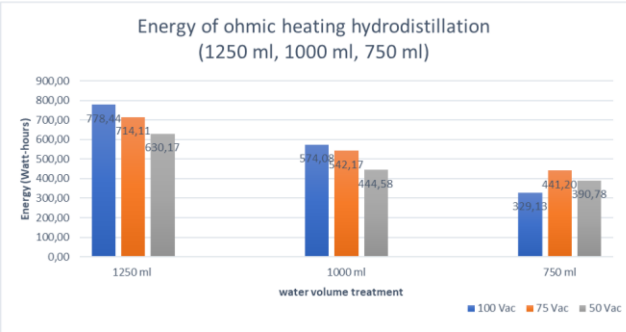

Fig. 11 – Energy of OHHD at 1250 ml, 1000 ml, and 750 ml

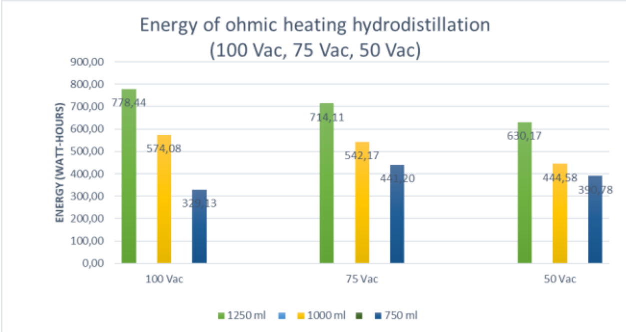

Fig. 12 – Energy of OHHD at 100 Vac, 75 Vac, and 50 Vac

A different pattern occurs in a water volume of 750 ml, i.e., at a voltage of 100 Vac, the energy produced during the process is smaller than the energy at a voltage of 75 Vac and 50 Vac. This is influenced by the duration of the hydrodistillation process. At a voltage of 100 Vac, the time required for the ohmic heating hydrodistillation process is only 50 minutes, while for voltages of 75 Vac and 50 Vac, it is greater, i.e., 100 minutes and 190 minutes. In the combination of 100 Vac voltage and 750 Vac water, the water evaporates faster so that the electric current stops because there is no water.

Ohmic heating hydrodistillation energy at the same voltage is shown in Fig. 12. Based on the figure, it can be seen that the energy required from each voltage has the same pattern. At a voltage of 100 Vac, the energy required for a water volume of 1250 ml is greater than for a water volume of 100 ml and a water volume of 750 ml. At voltages of 75 Vac and 50 Vac, the pattern is the same as at a voltage of 100 Vac. At the same voltage, energy consumption is influenced by the amount of water. The greater the volume of water, the greater the energy required to boil the mixture of water and nutmeg leaves.

Oil yield

The results of laboratory analysis indicated that the resulting oil had a refractive index of 1.4776 and an optical rotation of -4.834. The results of GC (gas chromatography) analysis indicated that nutmeg leaf oil had 25 compounds (Fig. 13). The content of nutmeg leaf oil includes main compounds such as Sabinene compounds (19.07%), α-pinene (18.04%), 4-terpineol (11.83%), limonene (8.32%), and β-pinene (7.92%) (*Asgarpanah & Kazemivash, 2012*). According to the measurement of mass spectrometry (MS), the myristicin concentration was 4.80% (SNI min 10%). SNI 06-2388-2006, pertaining to nutmeg oil derived from nutmeg seed distillation, serves as the standard reference for nutmeg oil. Since there is no SNI for nutmeg leaf oil used as a reference, some test results, like optical rotation and myristicin content, do not meet the SNI.

Oil yield is an extract of nutmeg leaves oil produced from the hydrodistillation process using ohmic heating. The resulting nutmeg leaves oil has a pale yellow color (Fig.14). The highest oil yield that could be extracted in this research was 1.07% when 50 Vac voltage and 750 ml of water or a nutmeg leaf to water ratio of 1:0.75 was combined, while the lowest yield of 0.38% was obtained when 75 Vac voltage and 1250 ml of water were combined (Fig. 15). The production of nutmeg leaf oil in ohmic heating hydrodistillation is influenced by the electric voltage and water volume. Ohmic heating not only produces a heating effect but also causes the cell walls of the leaves to permeate. The rate of diffusion through the cell wall, the extraction yield, and the reaction process can all be accelerated by increasing cell wall permeabilization (*Sofi'i & Arifin, 2022*).

Results of laboratory tests on the content of nutmeg leaf oil using the Soxhlet method chemical solvent gave a yield of 1.49%, while according to *BSN (2006*), the oil content of nutmeg leaves is no more than 1.7%. If the hydrodistillation method using ohmic heating with a water solvent is compared to the Soxhlet method with an n-hexane solvent, it shows that the ohmic heating hydrodistillation method has an oil extraction efficiency of 1.07% divided by 1.49% or 71.8%.

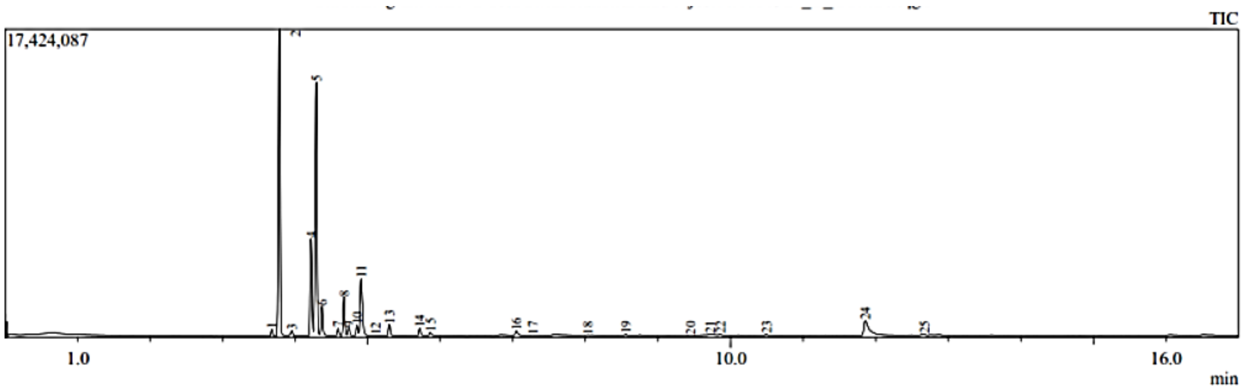

Fig. 13 – Chromatogram of nutmeg leaves oil

Fig. 14 – Nutmeg leaves oil

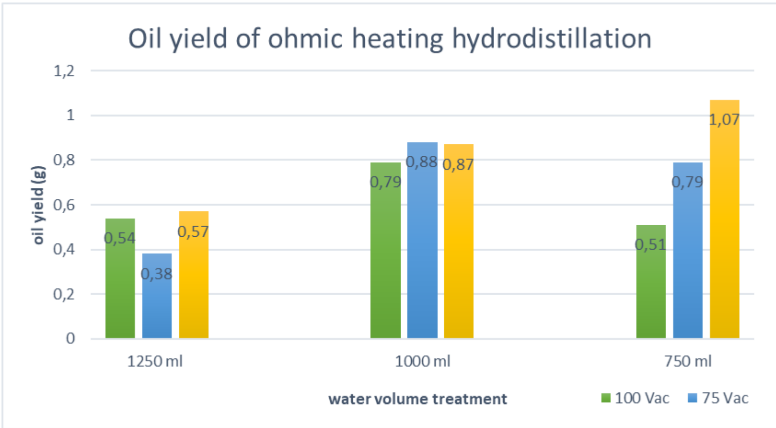

Fig. 15 - Nutmeg leaves oil yield

CONCLUSIONS

The higher the voltage, the faster the time to reach the boiling point of the liquid. The higher the volume of water, the lower the heating rate. The duration of the hydrodistillation process is influenced by the voltage and water used. The electrical conductivity value of the mixture of water and nutmeg leaves during the hydrodistillation process varies depending on the volume of water, voltage, electrode surface area, and temperature. At the same volume of water, the higher the voltage, the greater the energy required during the process. The greater the volume of water, the greater the energy required to boiling the mixture of water and nutmeg leaves. In ohmic heating hydrodistillation, the magnitude of the electric voltage and the volume of water used affect the yield of nutmeg leaf oil. The maximum oil yield that can be extracted is 1.07% at a combination of 50 Vac voltage and 750 ml of water, while the lowest yield is 0.38% at a combination of 75 Vac voltage and 1250 ml of water.

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