Essential oil processing of pepper process with aluminium condenser

Y Setiawan, R P Prayitnoadi and E Saputra

Department of Mechanical Engineering, Universitas Bangka Belitung, Bangka 33172, Indonesia.

E-mail: priyokopravitnoadi@gmail.com

Abstract. There are many ways to produce essential oils from pepper, one of which is the distillation system. This paper aims to determine the results of essential oils of pepper through a process of distillation of water and steam produced from 500 grams of pepper seeds using a time variation of 4 hours, 6 hours and 8 hours using water and steam distillation, but heating using an electric stove and condenser coil made of aluminium. The results of the most volatile oil volume at 6 hours were obtained 11.8 ml with a specific gravity of 0.87, the refractive index of 1.48 and solubility in 95% ethanol with an essential oil content of 2.36%. This innovation provides an interdisciplinary approach to study the environmental systems and find solutions to resolve the problems associated with it for sustainability.

1. Introduction

The program to increase the production of sustainable plantation crops through the application of right cultivation technology has promoted as a form of support for optimizing the use of land resources to produce agricultural products into useful products, one of which is an essential oil. Indonesia is one of the largest volatile oil-producing countries in the world. Indonesia's natural and geographical conditions support a variety of plants producing essential oils to grow. After going through the production process such as distillation, essential oils are exported to many countries. According to data from the Ministry of Industry, Indonesia can supply 90% of the world's needs for patchouli essential oils that are widely used in the perfume industry.

Other in Indonesia, at least about 40 types of essential oil plants grow and are produced from a total of around 150 types of essential oils traded in the international market [1]. Essential oils have many uses such as ingredients for the pharmaceutical, food, beauty care, and especially perfume industries. In the food industry, essential oils are used for flavoring and fragrance. In the cosmetics industry and perfume are used as fragrance ingredients for the manufacture of soaps, toothpaste, shampoos, lotions, and perfumes. While the pharmaceutical industry uses it for painkillers, infections, and bacterial killers, essential oils in the health sector can be used as an antiseptic, anti-inflammatory, analgesic, and sedative [2].

One ingredient for producing essential oils is pepper plants. Pepper or also called pepper (Piper nigrum L.) comes from the family Piperaceae [3]. Pepper seeds are long-known ingredients, many of which are planted in Indian, Arabic and Ceylon countries. With the development of history, pepper plants enter Indonesia, which is still centered in southern Sumatra (Jambi, Lampung and Bangka Island). Pepper production still mainly uses traditional methods with different hygiene conditions [4]. Pepper contains several minerals such as potassium, calcium, zinc, manganese, iron, magnesium, and vitamins.
Piperin, as the main component of the alkaloid contained in pepper, besides acting as an antioxidant, also has anti-hypertensive activity [5]. Essential oil from Piper sarmentosum leaves have anti-crawl activity [6], and it has an insecticide activity [7]. According to Souza, [8] the essential oil content of pepper leaves has toxic properties in termites. Pepper essential oil contains monoterpenes and sesquiterpenes, which can kill insects. Moreover, Kolhe et al., [9] posited the view that piperin in pepper has anti-inflammatory, antimalarial, weight loss, reduced fever, neutralizing snake venom, antiepileptic properties, and helps increase absorption of specific vitamins.

The physicochemical properties and analysis of the chemical components of essential oils in two Bangladeshi and Indian black pepper varieties using the GC-MS method. The physicochemical properties of Bangladeshi black pepper essential oil are colorless, specific gravity 0.84, refractive index 1.48, optical rotation -10.67° and solubility in 95% alcohol with a ratio of 1:5. Indian black essential oil is slightly greenish, specific gravity 0.84, refractive index 1.48, optical rotation -9.33, and solubility in 95% alcohol with a ratio of 1:5. The results of analysis by GC-MS contained a total of 18 chemical components of essential oils from Bangladeshi black pepper and 14 total chemical elements from Indian black pepper. The difference in the number of chemical compounds from the two varieties is due to differences in geographical location and ecological conditions [10].

Essential oils known as volatile oils or flying oils are compounds that are generally liquid, obtained from parts of the plant roots, bark, leaves, fruit, seeds, or from flowers by distillation. Essential oils can be obtained by extraction using organic solvents or by pressing or pressing and enzymatically. The results of different essential oils are influenced by two factors, namely, the age of the plant and the amount of rainfall [11]. The active ingredients derived from essential oils are expected to be more selective and less persistent when compared to the active ingredients of synthetic pesticides so that their use is relatively safer for the life of organisms and the surrounding environment [12].

| Table 1. Pepper essential oil quality standards. Source: Edition IV Food Chemical Codex (FCC) |
|---------------------------------------------------------------|
| Essential Oil | Specific Gravity (gr/ml) | Refractive Index 25°C | Solubility |
|----------------|-------------------------|-----------------------|------------|
| Pepper Oil     | 0.86 - 0.88             | 1.47 – 1.48           | In ethanol 95% 1:3 Dissolves |

Distillation methods carried out in the Goods Quality Testing and Certification Centre (BPSMB) in Medan using a Stahl based on 4 hours, 6 hours, and 8 hours of variation. The test performed to determine the essential oil content and grade Black pepper water shows that the essential oils obtained have different essential oil content, with a variation of 4 hours having essential oil content of 2.28%, 6 hours having essential oil content of 2.57% and 8 hours having essential oil content of 2.85%, while for black pepper water content has a level of 13.2%. So that to get an adequate time is to use 6 hours by the procedures of SNI 01-0005-1995 [13]. Extraction of essential oils by distillation method using the Stahl tool, by testing 500 grams of dried pepper, the volume of essential oil obtained was 6.3 ml from 500 grams of crushed pepper using a blender and yielding a specific gravity of 0.937 [14].

The distillation process of essential oils is usually often also called the distillation process or distillation (essential oil distillation) is the process of separating components from a mixture in the form of a liquid solution where the characteristics of the mix are interfering and volatile. Also, these components have vapor pressure differences and the results of their separation into components or component groups. Wesolowska et al., [15] mention that a pure steam distillation method and the operating cost are not expensive. Meanwhile, Hui, [16] said that uses the soxhlet extraction method with n-hexane solvent, which requires more expensive operating costs and requires complex equipment.
2. Research Methodology

2.1. Research Samples
The sample used in this study is the pepper (Piper nigrum L.) part of the seed. Samples were taken from the community gardens of Bangka Island. Samples identified at The University of Bangka Belitung’s Laboratory.

2.2. Tools
The equipment used is a set of steam distillation equipment, a set of Gas Chromatography tools - Mass Spectrometry (GC-MS), analytic balance, micropipette 20-200 μL, test container, wrapping, aluminum foil, oven, autoclave, Petri dish, vial bottle, pumpkin measure 10 mL, jar, Erlenmeyer

2.3. Research Procedure

2.3.1. Essential Oil Steam Distillation. Prepare 300 grams pepper seeds, fill the water on the distilled kettle with a water level of about 100 mm from the bottom of the pot, and then close it. Turn on the electric stove and then turn on the stopwatch to see the distillation process as specified. Control the water coming out of the condenser coil towards the separating funnel when evaporation has occurred on the distilled kettle so that no flooding occurs in the separating pipe by periodically removing water through the tap. Take the separating tube from the distillate then do the disposal of water through the valve under the separating funnel as limited as the essential oil obtained. From the results obtained measure the volume of essential oils obtained using a measuring cup. The measured oil volume is stored in a bottle of essential oil storage and stored at room temperature and protected from direct sunlight because it will affect the quality of essential oils. After all the first stage of the testing process is complete, then the weakness of the distillation tool is carried out when testing the first stage.

2.3.2. GC-MS Analysis Pepper Seeds Essential Oil. Pepper seeds essential oil was analysed by its constituent components using Gas Chromatographic Mass Spectrometry (GC-MS) is a method of separating organic compounds using two methods of compound analysis, namely gas chromatography (GC) to quantitatively analyse the number of compounds and mass spectrometry (MS) to analyse the molecular structure of analytic compounds.

2.3.3. Specific gravity. The pycnometer is washed and cleaned, then rinsed successively with ethanol and diethyl ether. The inside of the pycnometer is dried with dry air currents and closed. Pycnometer is left in the weighing cabinet for 30 minutes and weighed \( m \). The pycnometer is filled with distilled water which has been boiled and left at room temperature while avoiding air bubbles. Then the pycnometer is dipped in a water bath at room temperature for 30 minutes, and the pycnometer is dried. The pycnometer is left in the weighing cabinet for 30 minutes, weighed by its contents \( m_1 \). The pycnometer is emptied, washed with ethanol and diethyl ether, dried with a dry air stream. The pycnometer is filled with oil samples and is avoided by air bubbles. Pycnometer is dipped back into the water bath at room temperature for 30 minutes. Insert the lid and dry the pycnometer. Pycnometer is weighed for 30 minutes until a constant weight \( m_2 \) is obtained.

\[
\text{Density} = \frac{(m_2-m)}{(m_1-m)} \tag{1}
\]

2.3.4. Refractive Index. Water is flowed through the refractometer to the temperature according to the reading. The temperature must not differ more than ± 2 ° C from the reference temperature and must be maintained with a tolerance of ± 0.2 ° C. Before the oil is placed inside the tool, the oil must be at the same temperature as the temperature at which the measurement will be carried out, which is 20°C. Then the reading is done when the heat is stable.
2.3.5. **Solubility in Ethanol.** The essential oil of 1 mL was put into a test tube and added 3 mL of 95% ethanol drop by drop and then shaken. After that, it was seen the solubility of essential oils in ethanol. Then the essential oil sample from black pepper was continued by analysis using GC-MS.

3. Results and Discussion

3.1. **The Volume of Essential Oils Production Results of Steam and Water Distillation**

The time used increases the volume of volatile oil in a variation of 4 hours over a variation of 6 hours, but the variation in time of 6 hours to 8 hours decreases due to several factors. Namely, essential oils contained in pepper seeds have run out, and the longer time it makes Essential oils are reduced because the basic properties of volatile oils are volatile, so the essential oils contained in the separating funnel experience evaporation.

![Figure 1. Volume of essential oil](image)

3.2. **Quality Standards for Distillation Results**

3.2.1. **Specific gravity.**

In heavy testing, the type uses a pycnometer. The results of quality testing that have been carried out based on the specific gravity of pepper essential oil can be seen in Table 2:

| Type of test | Time   | Specific gravity (gr/ml) | FCC Edition |
|-------------|--------|--------------------------|-------------|
|             | 4 hours| 0.88                     |             |
|             | 6 hours| 0.87                     |             |
|             | 8 hours| 0.88                     | IV          |

Specific gravity tested was different for each variation of time and the highest specific gravity produced was in the distillation time variation carried out for 4 hours, namely 0.8821, but the overall time variation fulfilled the quality of essential oils according to the standards issued by FCC edition IV. The size of the material strongly influences the amount of specific gravity in various essential oils, and the length of distillation carried out. The greater the weight fraction contained in the oil, the greater the value of its species weight. The higher the value of type weight, the more components contained in these substances with high molecular weight and long carbon chains [17].

3.2.2. **Solubility in Ethanol.**

The results of the quality testing of essential oils that have been carried out based on solubility in ethanol can be seen in Table 3.
Table 3. Solubility in ethanol

| Type of Test       | Time  | FCC Edition |
|--------------------|-------|-------------|
| Solubility in ethanol 95% | 4 hours | Soluble |
| 1:3 dissolves      | 6 hours | Soluble |
|                   | 8 hours   | Soluble |

In Table 3, it can be seen that the overall quality testing of essential oils based on time variations ranging from 4 hours, 6 hours and 8 hours, has fulfilled the requirements in testing solubility in ethanol. Solubility in ethanol is expressed in the amount of ethanol needed to dissolve 1 ml of oil. The greater the solubility, the better the quality. Solubility in alcohol illustrates whether the oil is easily dissolved or not. The more easily dissolved oil in alcohol, the more content of polar compounds in the oil. Alcohol solubility is an important factor in testing essential oils because it can determine the quality of essential oils [18].

3.3. Content of Essential Oil from Water and Steam Distillation

Essential oil content based on variations, namely essential oil content obtained by an average time variation of 4 hours 1.92%, for 6 hours 2.36% and for 8 hours 2.33%. The essential oil content of a material is influenced by the variety, geographical environment of growth, age, and quality of the raw materials used and the way of distillation.

4. Conclusion

The volume of essential oil based on time variations for 4 hours obtained 9.6 ml, for 6 hours got 11.8 ml and 8 hours obtained 11.6 ml. Based on the time variation, the volume of essential oil is best in a variation of 6 hours, because with a variation of 6 hours the essential oil has been fully lifted from the pepper seeds and the essential oil is not reduced due to evaporation over a 6 hour time variation. Essential oil quality based on the specific gravity obtained at 4 hours variations of 0.88, 6 hours variations of 0.87 and 8 hours by 0.88, based on the refractive index obtained at 4 hours variations of 1.47, 6 hours variations of 1.48 and 8 hours by 1.47, based on solubility in 95% ethanol all variations of time dissolve and all quality testing meets the standards of essential oils. Essential oil content obtained by an average time variation of 4-hour is 1.92%, for 6-hour is 2.36% and 8-hour is 2.33%.

Acknowledgment

We gratefully acknowledge the support from USAID through the SHERA program – Centre for Development of Sustainable Region (CDSR). In the year 2017-2021 CDSR is led by Centre for Energy Studies – UGM.

References

[1] Citra P I 2013 Identifikasi Komponen Minyak Atsiri Pada Beberapa Tanaman Dari Indonesia Yang Memiliki Bau Tidak Sedap (Bandung: Universitas Pendidikan Indonesia)
[2] Yuliani S and Satuhu 2012 Panduan Lengkap Minyak Atsiri (Jakarta : Penebar Swadaya)
[3] Vasavirama K and Upender M 2014 Piperine: A Valuable Alkaloid from Piper Species, Int. J. of Pharmacy and Pharmaceutical Sciences 6 34-38.
[4] Duarte and Medeiras 1999 Salmonella in Brazillian black pepper : Causes and Method of Control. Paper presented at 21st Peppertech Meeting. International Pepper Community. Kuching, Malaysia, 22 July 1999.
[5] Risfaheri 1996 Masalah dan Standar Mutu Lada, Monograf Tanaman Lada (Bogor: Balai Tanaman Rempah dan Obat. Bogor)
[6] Chieng T C, Assim Z B and Fasihuddin B A 2008 Toxicity and Antitermite Activities of the Essential Oil from Piper Sarmentosum, The Malaysian J. of Analytical Science 12 234-239.
[7] Francois T, Michel J D P, Lambert S M, Ndfor F, Vyry W N A, Henri A Z P and Chantal M 2009
Comparative Essential Oils Composition and Insecticidal Effect of Different Tissues of Piper capense L., Piper guineense Schum. et Thonn., Piper nigrum L. and Piper umbellatum L. grown in Cameroon, *African J. of Biotechnology* **8** 424-43.

[8] Souza A D, Lopes E M C, Cordeiro I, Young M C M, Sobral M E G and Moreno P R H 2010 Chemical Composition and Acetylcholinesterase Inhibitory Activity of Essential Oils of Myrceugenia myrcioides (Cambess.) O. Berg and Eugenia riedelian A. Berg, Myrtaceae, *Brazilian J. of Pharmacognosy* **20** 175-179.

[9] Kolhe S R, Borole P, and Patel U 2011 Extraction and Evaluation of Piperine from Piper nigrum, *Int. J. of Applied Biology and Pharmaceutical Technology*, 144-149.

[10] Aziz S, Naher S, Abukawsar M D and Roy S K 2012 Comparative Studies on Physicochemical Properties and GC-MS Analysis of Essential Oil of the Two Varieties of the Black Pepper (Piper nigrum Linn.) *Int. J. of Pharmaceutical and Phytopharmacological Research* **2** 2249–6084.

[11] Guenther E 1987 *Minyak Atsiri I, Ketaren, S. (alih bahasa). Jilid I* (Jakarta: UI Press)

[12] Regnault-Roger C 2005 *New insecticides of plant origin for the third millennium in B.J.R. Regnault-Roger et al. eds. Bio-pesticides of plant Origin*. Lavoisier Publishing Inc. p. 313

[13] Ayu P W 2014 *Pengaruh Waktu Destilasi Terhadap Kadar Minyak Atsiri dan Penentuan Kadar Air Pada Lada* (Medan: Universitas Sumatera Utara)

[14] Vetty N 2014 *Uji Ekstrak Minyak Atsiri Lada Putih (Piper Nigrum Linn) Sebagai Anti Bakteri Bacillus Cereus* (Bengkulu: Universitas Bengkulu)

[15] Wesolowska A, Jadcza K and Grzeszczuk M 2010 *Influence of Distillation Time on The Content and Composition of Essential Oil Isolated from Lavender (Lavandula angustifolia Mill.). Kerva Polonica*, **56**(3) 24-35.

[16] Hui Y H 2007 *Handbook of Food Products Manufacturing* (New Jersey: John Wiley & Sons, Inc.)

[17] Guenther E 2006 *Minyak Atsiri Jilid IV, Ketaren, S. (alih bahasa)* (Jakarta: UI Press, Jakarta)

[18] Khasanah U L, Kawaji, Rohula U and Aji Y M 2015 *Pengaruh Perlakuan Pendahuluan Terhadap Karakteristik Mutu Minyak Atsiri Daun Jeruk Purut (Citrus hystrix DC)*, *J. Aplikasi Teknologi Pangan*. **4** 48-55