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Eugenol isolation of Clove (Syzygium aromaticum) flower

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Abstracts

Corresponding author: heru.nurcahyo@poltektegal.ac .id Received: 10 May 2020, Revised: 28 May 2020, Accepted: 23 June 2020. The use of alternative and complementary therapies as a treatment is growing rapidly, one of them is by alternative medicine that uses steam from essential oils / essential oils from various kinds of plants that can be inhaled and smeared to cure various conditions. Clove plants (Syzygium aromaticum) are one of the producers of essential oils that can be used as an alternative treatment for herbs. In clove oil consists of several types of components. The main component possessed by clove is eugenol which has the highest number among other components. Eugenol are important compounds in clove oil. It is a clear liquid to pale yellow compound with a refreshing and spicy aroma like dried clove flowers, giving a distinctive aroma to clove oil. The purpose of this study was to determine the eugenol content of clove flower essential oil. The use of clove flowers in this study is used to improve the competency of local products that in the future can make the outputs of competitive and economical value. This study begins with the distillation process of clove flower water to get oil up to isolation of eugenol. The identification of eugenol with TLC and testing of eugenol levels with GC-MS. The results showed that the essential oil yield was 3.16%, eugenol yield 42.92%, hRf value 11.46 and with grade 97%.

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Keywords: clove, essential oil, eugenol

1. Introduction

Currently, the use of alternative and complementary therapies as treatment is growing rapidly [1]. Eugenol compounds are the main components contained in clove oil (*Syzygium aromaticum*) with content that can

reach 70-96% [6]. Eugenol and its derivatives have pharmacological activities as analgesic, anti-inflammatory, antimicrobial, antiviral, antifungal, antiseptic, antispasmodic, antiemetics, stimulants, local anesthetics [2][3][5]. The analgesic activity of eugenol compounds is widely used as raw material for ointments that can be used to reduce pain due to rheumatism, as well as raw materials for toothache medicine, cologne, and aromatherapy products [2].

2. Experiments Procedure

Water Distillation

Oil extraction method in this study using water distillation. An amount of approximately 250 grams of dried clove flowers is put in a round bottom flask and then water is added to it. The distillation process is carried out until the oil is obtained, the percentage yield is calculated. This process was replicated 3 times.

Eugenol Insulation

Isolation of eugenol was carried out using clove oil from the results of distillation carried out using the liquid-liquid extraction method. 25 mL of clove oil was taken (replicated 3 times with the same volume) in each isolation. Then the sample was added with 1N KOH, shaken for 5 minutes, then heated in a water bath at 37-38°C for 10 minutes, then shaken again for 5 minutes.When the base is neutralized with 10% H2SO4. Extract with 15 mL ether 3 times in a separating funnel, collect the ether phase into one, evaporate over a water bath until a thick phase is obtained.

Eugenol Identification Test by TLC

The eugenol obtained was identified by TLC. In this identification, the stationary phase was used in the form of a silica gel plate and the mobile phase was hexane:ethyl acetate (96:4). The resulting spots are then determined by their hRf values.

Eugenol analysis by GC-MS

Determination of eugenol levels was carried out by GC-MS at PT.Sucofindo Semarang.

3. Result and Discussion

The clove flower is processed by distillation of water to obtain the oil. The clove flower essential oil produced is organoleptic with a dark yellow color and a distinctive smell like cloves. Clove oil has a density greater than water, which is approximately 1.0664(Kusumadewi, 2011). This causes the essential oil to be at the bottom while the water is at the top. The following is the result of the distillation of clove flower essential oil yields from several replications.

| Replication | Sample Weight | Amount of Oil | ma yield (%v/w) | |
|-------------|------------------|------------------|------------------------|--|
| | (grams) | (mL) | | |
| 1 | 250.01 | 18.64 | 7.46 | |
| 2 | 250.00 | 17.33 | 6.93 | |
| 3 | 250.04 | 17.55 | 7.02 | |
| 4 | 250.03 | 17.70 | 7.08 | |
| 5 | 250.02 | 18.30 | 7.32 | |
| Average | | 17.90 | 7.16 | |

The average distillation of clove essential oil was 17.90 mL or the yield was 7.16% v/w. Clove flower oil was isolated to obtain its eugenol. In isolation, clove oil is reacted with 1N KOH so that a reaction occurs so that the K-eugenolate salt will be separated from other components contained in clove oil.

The reaction of clove oil with KOH is exothermicbecause when a reaction occurs to replace the H+ group with K+, heat is released from the system to the environment. This is indicated by the warm glass beaker. The color of the solution is dark brownish yellow. The eugenol solution was added with 1N KOH then the next process was stirred until homogeneous. KOH is used because the K+ ion is more strongly bound to the eugenolate. Besides that The purpose of the addition of KOH is also that the eugenol component of clove oil can be isolated. Eugenol and KOH will form potassium eugenolate which is soluble in water. Thus, the non-eugenol part was extracted with the addition of ether which is an inorganic acid to produce a free potassium salt of eugenol.

This eugenol was then purified by evaporation over a water bath and then the yield was calculated.

| Table 2. Eugenol Ren yield | | | | | | |
|----------------------------|----------------------------|-----------------------------|------------------|--|--|--|
| Replication | Number Sample s (mL) | Total Eugenol (grams) | %Yield (w//v) | | | |
| 1 | 25 | 9.90 | 39.60 | | | |
| 2 | 25 | 10,40 | 41.60 | | | |
| 3 | 25 | 10.70 | 42.80 | | | |
| Total average | | 10.33 | 41.33 | | | |

The average amount of eugenol was 10.33 mL or the yield obtained was 41.33% w/v. The eugenol obtained was identified by TLC using a stationary phase in the form of silica gel and a mobile phase in the form of hexane:ethyl acetate (96:4). Based on the polarity, it can be ascertained that the method used is normal phase TLC where the stationary phase is more polar than the mobile phase. A combination of semi-polar and non-polar mobile phases is used because the isolated sample is a non-polar compound so that the sample will be eluted far along with the eluent while the stationary phase will hold impurities and other polar compounds. In the identification process with TLC. theoretical standards are used (Stahl, 1985). The results of the elution were read under UV light at a wavelength of 366. The spots obtained were then calculated for their hRf values.



Figure 1. TLC test results

The results of the hRf values are listed in table 3. The result of the average hRf value for eugenol is 11.46, this value is in the standard value range of 10-15 so that the eugenol sample

isolated from clove flower essential oil is known to contain eugenol.

To ensure the purity of the isolated eugenol, GC-MS analysis was performed. GC-MS is the best analytical technique to identify volatile materials or compounds, so that the potential of the eugenol can be known [7]. The results of the GC-MS analysis showed that the eugenol content obtained was 97%.

| Table | | | | |
|-----------------|-----------------------------|-----------------------------|-------|----------------------|
| Replicati on | Solvent distance (cm) | Sample distanc e (cm) | hRf | hrf. standar d |
| 1 | 8 | 0.90 | 11.25 | 10-15 (Stahl, |
| 2 | 8 | 0.93 | 11.63 | |
| 3 | 8 | 0.92 | 11.50 | |
| Total average | | 0.92 | 11.46 | 1705) |

4. Conclusion

The yield of essential oils in each distillation obtained an average of 17.90 mL or 7.16% v/w. The results of eugenol isolation in each replication obtained an average of 10.33 grams or 41.33%. The results of the TLC identification test showed an average hRf value of 11.46 with the eugenol content determined by GC-MS was 97%.

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