Deodorization of Calophyllum Inophyllum oil using Steam distillation method

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Abstract. Deodorization is a crucial refining stage with an important effect on the quality of oil refined. We used steam distillation method to remove the smell of calophyllum inophyllum oil. We investigated the effect of various factors such as vaporization temperature, oil volume to the deodorization process. In laboratory conditions, at vaporization temperature of 140°C and oil volume of 200mL, the optimal time of deodorization process is 21 hours with 95% oil’s smell removed. In the other hands, deodorized oil still remains the benefit compents. The aim of this study was to eliminate unpleasant pungent smell of calophyllum inophyllum oil. In the other hands, deodorized oil still retain the beneficial component and properties eminence. Thereby, creating a substrate with great potential in pharmaceuticals and cosmetics. Also, deodorized calophyllum inophyllum oil will help increase the economic value of traditional calophyllum inophyllum oil.

1. Introduction
Natural products represent a source of biologically active molecules that have an important role in drug discovery and food products [1-5]. The Calophyllum Inophyllum is widely distributed in the southern coastal belt of India, East Africa, Australia and Southeast Asia. In Vietnam, the calophyllum inophyllum tree was found most in the south of Vietnam. Calophyllum Inophyllum oil is non-edible fat oil and contains 50-73% of lipids [6-8]. It was extracted from calophyllum inophyllum seeds. The total lipids included 92.0% of neutral lipids, 6.4% glycolipids and 1.6% phospholipids. Calophyllum Inophyllum oil contains fatty acids such as neutral lipid, glycolipids and phospholipids which have a ability to penetrate into skin and regenerate cell of the skin.

The previous studies show that Oil also has the capacity to cure physical and chemical burns, radio-dermatitis and anal fissures. Recently some scientists have discovered anti-HIV compounds from calophyllum inophyllum seed report by Patil et al (1993) and Taylor et al (1994), Spino et al (1998) [9,10]. In addition, it also plays an important medicine to treat skin diseases and different purposes ranging from treatment of burns, cuts, scars to sciatica, sores to neuralgia, eczema and so on. Moreover, many researches have discovered application in the cosmetics field in order to prevent skin aging and regenerate damaged skin and [11,12]. Besides, the lastest studies described Calophyllum Inophyllum oil biological activity such as anti-septic, anti-inflammatory, antioxidant and so on [13].

Of various steps in the refining process, deodorization is critical for its capabilities to remove free fatty acids (FFAs), oxidation products and volatiles, which in turn improves the quality of the refined oil [14]. The aim of this process is the elimination of undesired volatile odoriferous elements in oils including free fatty acids, aldehydes, ketones, and carbohydrates [15]. Since traditional methods of deodorization such as steam distillation (SD) and molecular distillation (MD) requires high
temperatures (190 - 275°C) and low pressure (0.2-1 kPa), it is suggested that heating may induce a serious of chemical changes such as cyclization, cis-trans isomerization, and double bond migration. At present, three types of deodorizers including batch, semi-continuous and continuous are applied in the practice. Deodorized calophyllum inophyllum oil will be a potential substrate in the cosmetic and pharmaceutical. In the other hands, deodorization will increase the economic value calophyllum inophyllum oil. However, there are very little researchs on calophyllum inophyllum oils deodorization as well as deodorized calophyllum inophyllum oils effective have not been reported and compared systematic. Thus, the optimization of the parameters in deodorization process is a large challenge for removing maximally undesirable components but still remaining maximally desirable valuable minor components. The objective of this study is optimizing deodorization process by steam distillation method. This process was conducted based on physicochemical parameters such as vaporization temperature, oil volume.

2. Material and methods

2.1. Sample preparation

Calophyllum Inophyllum oil was provided by AoTa Joint Stock Company. Chemicals used for analysing and evaluating of calophyllum inophyllum oil indicators: Sodium thiosulfate pentahydrate (Na2S2O3.5H2O) 99.5% (Xilong Scientific Co., Ltd), Potassium hydroxide (KOH) 85% (Xilong Scientific Co., Ltd), Hydrochloric acid (HCl) 36-38% (Xilong Scientific Co., Ltd), Phenolphthalein (C20H14O4), Starch soluble (C6H10O5), Xylong Scientific Co., Ltd., 99% Potassium Iodide (KI), anhydrous Na2SO4 99% (Xilong Scientific Co., Ltd), Acetic acid glacial (CH3COOH) 99.5% (Quangdong Guanghua Sci-Tech Co., Ltd) made in China. Chloroform LR (CHCl3) 99% purchased from Alpha Chemika made in India. Diethyl ether (C4H10O) 99.5% purchased from VN - Chemsol Co.

2.2. Deodorization process of Calophyllum inophyllum oil by steam distillation

The steam is generated by heating the flask 1 (distilled water flask) then for steam to escape through the flask 2 (oil flask) which was heated at a temperature of 50°C. Variable parameters such as evaporation temperature 130°C – 150°C, volume of deodorant 100mL – 300mL. This process is carried out for every 2 hours will take out the sample once to check out until 90% - 95% oil’s odor was removed, recording time of deodorization process. The deodorized mixture will be filtered water. We continue anhydrous by Na2SO4. Finally, we obtain the deodorized calophyllum inophyllum oil.

2.3. Chemical analysis of calophyllum inophyllum oils by GC-MS analysis

Chemical composition of the oil sample was analyzed with following procedure. First, 25 µL essential oil was introduced into 1.0 mL n-hexane, followed by dehydration with Na2SO4. The GC machine was Agilent 6890 N with MS 5973 inert, HP5-MS column, and head column pressure of 9.3 psi. Operating conditions are as follows: injection volume 1.0 µL, split 1:100, carrier gas He, flow rate 1.0 mL/min, injection temperature 250 °C. The temperature progressed from 50 °C for to 80 °C at 2 °C/min, from 80 °C to 150 °C at 5 °C/min, from 150 °C to 200 °C at 10 °C/min, from 200 °C to 300 °C at 20 °C/min and was kept at 300 °C for 5 min.

3. Result and discussion

3.1. Effect of vaporization temperature and Effect of ratio of vaporization/oil volume

The influence of temperature to the deodorization process is show in figure 1. The figure 1 shows that higher temperatures gave higher effective deodorization, due to increased speed of motion of a molecule and volatile components. For calophyllum inophyllum oil decrease in the deodorization time from 25 hours to 22 hours was observed when increasing the temperature from 130°C to 140°C. But a further temperature increase to 145°C resulted in an increase in the deodorization time to 23 hours. This may be because the vaporization of water is too fast should not enticing the smell component of the calophyllum inophyllum oil. Therefore, the proper temperatures for deodorization process by steam distillation is 140°C.

The influence of ratio of vaporization/oil volume to the deodorization process is show in figure 2. The figure 2 shows when increase oil volume then lower effective deodorization. When increase oil volume then time deodorization process also increase. When increasing the volume of calophyllum inophyllum oil then the time deodorization also increasing turns sequence 1.7/100; 1.7/150; 1.7/200; 1.7/250; 1.7/300 (mLH2O/minute)/mL oil and 20; 21; 21.5; 22 hours, respectively. That shows...
200mL calophyllum inophyllum oil with 21 hours time deodorization is optimal. Thus the optimal volume of calophyllum inophyllum oil is 200mL.

![Figure 1. Effect of temperature](image1)

![Figure 2. Effect of ratio of vaporation/oil volume](image2)

3.2. Effect of distillation method

The influence of distillation method to the deodorization process is show in figure 3. The figure 3 show deodorization by steam distillation method (21 hours) more effective hydro distillation method (26 hours) at the same con. Because the hydro distillation method the process of evaporation of the water hampered by calophyllum inophyllum oil and this method needs more heat. In the other hand, high temperature makes the calophyllum inophyllum oil denaturation lead to affects the quality of the calophyllum inophyllum oil.

![Figure 3. Effect of distillation method](image3)

3.3. Physicochemical properties of calophyllum inophyllum oil before and after deodorized

| Table 1. Physicochemical properties of Calophyllum Inophyllum Oil |
|-----------------|-----------------|-----------------|
| STT             | Property            | Original Oil | Deodorized Oil |
| 1               | Density at 25°C (kg/m³) | 1.013        | 0.946        |
| 2               | Kinematic Viscosity (cst) | 92.2         | 94.8         |
| 3               | Free Fatty Acid (mgKOH/g) | 21.385       | 24.346       |
| 4               | Saponification Value (mgKOH/g) | 63.580       | 61.710       |
| 5               | Peroxide Value (m Eq/kg) | 0.385        | 0.201        |
| 6               | Este Value          | 59.784       | 57.456       |
| 7               | AV Value (mgKOH/g)   | 3.796        | 4.254        |
The index such as AV, PV, EV, and SV of crude and deodorized calophyllum inophyllum oils were presented in table 1. Physicochemical index of calophyllum inophyllum oil before and after deodorized with no significant changes. Crexi et al (2010)[16] suggested that AV is usually associated with free fatty acids (FFA) and undesirable flavor present in fats and oils. However, The AV index of crude calophyllum inophyllum oil was 3.796 mgKOH/g after deodorization it was increase to 4.254 mgKOH/g. That shows some of the AV index as well as FFAs of calophyllum inophyllum oil do not affect the smell of oil. The PV of crude calophyllum inophyllum oil was 0.385 mEq/kg and it was reduced to 0.201 mEq/kg after deodorization with steam distillation method. In addition, the PV index reduction also show of the freshness of the deodorized calphyllum inophyllum oil higher original oil. The SV index was reduced from 63.580 mgKOH/g rest was 61.710 mgKOH/g. This reduction in SV was explained for effectiveness removing phosphatides and soap during the deodorization process, describe by Menegazzo et al (2014)[16]

3.4. Calophyllum Inophyllum oil chemical analysis by GC-MS method
After determining the optimal parameters (temperature, volume of calophyllum inophyllum oil). Acid composition of product was determined by GC-MS. Results of GC-MS analysis is show in figure 4. The figure 4 show content acid of calophyllum inophyllum oil change is not significant after deodorization process. This show deodorized calophyllum inophyllum oil retains the acid composition and properties eminence.

![Figure 4](image)

**Figure 4.** Acid component of calophyllum inophyllum oil before and after deodorized

4. Conclusion
The results of this study were achieved: optimizing parameters (vaporization temperature, oil volume) of the deodorization process by steam distillation at laboratory conditions. The physicochemical index and acid content of calophyllum inophyllum oil were determined by two titration methods according to the standard TCVN and GC-MS. Deodorized calophyllum inophyllum oil will be a potential substrate in the cosmetic and pharmaceutical. In the other hands, deodorized oil still retain the beneficial component and properties eminence. This study, we not yet able to of the parameters optimization of the deorization process of calophyllum inophyllum oil by steam distillation method and could not detect odor-causing components of oil. If I have time I will continue to investigate and further research to find out the optimal parameters as also odor-causing components of oil.

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