Essential oil from *Amomum Longiligulare* T.L. Wu cultivated in Ninh Thuan province, Vietnam

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Abstract. *Amomum Longiligulare* T.L. Wu fruit (*A. Longiligulare*) is a traditional Chinese medicine used to treat indigestion, cough and abdominal pain. In addition, it can create a nice refreshing effect in the mouth and tends to reduce body weight. In this research, *A. Longiligulare* fruit essential oil was successfully extracted by the hydrodistillation method. *A. Longiligulare* from Ninh Thuan province in Vietnam gave high extraction yield, which was about 4.6 ml/100g on weight dried basis. The optimum condition for hydrodistillation without immersing *A. Longiligulare* powder at particle size smaller than 1 mm, at 1/8 material-water ratio, at water flow rate 2.7 g/min after 3 h distillation. Significantly, the high concentration of D-Camphor (46.714 %) and Bornyl acetate (31.809 %) were detected in essential oil from Ninh Thuan’s *A. Longiligulare*.

1. Introduction

*Amomum Longiligulare* T.L. Wu (*A. Longiligulare*) belongs to the genus *amomum*, the family Zingiberaceae (ginger). This is a perennial herb, 1.5 - 2.5 m high, hygrophilous and shade-loving plant. *A. longiligulare* has a distribution area from China Hainan Island to Central Vietnam. In Vietnam, *A. Longiligulare* is also known as Mac Neng (Tay) or Sa Ngan (Dao) [1]. Traditionally, *A. Longiligulare* dried fruit is widely used as a food additive to develop the food’s distinct flavor because of its spicy taste, warmth and nice aroma [2]. Recently, *A. Longiligulare* dried fruits have attracted intensive attention as a functional food because of their various biological activities, such as antioxidant, antifungal, antitumor, and neuroprotection properties [3]. In addition, dried fruit also has been valued in traditional medicine for treating indigestion, coughing, abdominal pain and tends to lower body weight [2,4].

*A. Longiligulare* contains five main classes of chemical components including saponins, flavonoid glycosides, organic acids, inorganic components and especially volatile oil - approximately contain 1.7–3% [5]. *A. Longiligulare* essential oil contains monoterpane hydrocarbons, oxygenated monoterpenes, sesquiterpene hydrocarbons, oxygenated sesquiterpenes. The main constituents of *A. Longiligulare* essential oil from the seed are pinene, camphor, borneol acetate, and D-limonene [6]. Several methods could be applied to extract Cardamom essential oils such as hydrodistillation, steam distillation, volatile solvent extraction, supercritical fluid extraction (CO₂) or microwave-assisted hydrodistillation. Commonly, hydrodistillation is applied for extracting essential oil from herbal plants since it’s cheap and also could get high extraction yield and good quality essential oils [7].
Until now, a few research were done to determine the chemical compositions of essential oil from different parts of *A. Longiligulare* grew in Vietnam [8, 9]. In this study, the best conditions for extraction of the essential oil by using hydrodistillation were investigated and chemical composition of essential oil of *A. Longiligulare* fruit in Ninh Thuan, Vietnam was determined by Gas Chromatography- Mass Spectrometry (GC-MS).

2. Materials and methods

2.1. Materials

*Amomum Longiligulare* T.L. Wu (*A. Longiligulate*) fruits were harvested from Ninh Thuan province, Vietnam and dried under sunlight before transported to Nguyen Tat Thanh University, Ho Chi Minh city, Vietnam.

2.2. Hydrodistillation process

Dried *A. Longiligulare* fruits were cleaned and the shells were removed to obtain cleaned kernels. The nut was ground and sieved to obtain a consistent powder size. The measured moisture content of *A. Longiligulare* powders were in the range of 11.50-12.57%. Hydrodistillation was applied for extracting the essential oil from *A. Longiligulare* powder. In each experiments, 40 g of *A. longiligulare* powder and water mixture was put into a 1 liter glass flask and then heated by magnetic stirring heating mantle from Genlab under normal pressure for boiling. Before distillation, the *A. Longiligulare* powders were pretreated with a range of Sodium chloride (NaCl) solution with concentration from 0 – 18 mg/l. Several parameters that may influence the distilling process were varied and conducted as shown in the Table 1.

| No | Parameters                   | Condition          |
|----|------------------------------|--------------------|
| 1  | Particle size (mm)           | < 1 mm; > 1 mm     |
| 2  | Material / solvent ratio (g/ml) | 2.5, 2.7, 3.4, 3.7 |
| 3  | Water flow rate (ml/min)     | 2.5, 2.7, 3.4      |
| 4  | Distillation time (min)      | 30 – 210           |
| 5  | Concentration of NaCl (mg/l) | 0 – 18             |

2.3. Extraction yield (Y)

The extraction yield was calculated based on absolute dry weight as shown in Equation 1:

\[ Y (\text{ml/100g}) = \frac{V_o (\text{ml})}{m(1-\alpha)} \times 100 \]  

(1)

where \( V_o \) (ml) is volume of essential oil; \( m \) (g) is the mass of water and \( \alpha \) (%) is moisture content of the material.

2.4. Composition determination (GC-MS)

The essential oil were determined by GC-MS, GC-456 SQ with SCION performance RESTEK Rxi-5ms (30 m x 0.25 mm (for example), 0.25 µm df). The gas Helium constant flow rate was set at 1 mL/min. The injector temperature was set at 250 °C and the rate of Division is 1:30.

3. Results and discussions

3.1. Factors that affect extraction yield (Y)

3.1.1. Particle size. The effect of particle size on extraction yield is shown in Figure 1. As could be seen, the powder with a particle size less than 1 mm gave higher extraction yield than that of a particle size
larger than 1 mm (1.71% and 3.96%). It can be explained, smaller size powder lead to larger mass transfer area between material and water and also more cells had been broken during grinding process. Due to that, the essential oil is easier to diffuse out from the powder, and thus increase the extraction yield [10, 11].

![Figure 1](image1.png)

**Figure 1.** Effect of particle size on extraction yield

3.1.2. Raw material to solvent ratio. In hydrodistillation, ratios of materials to solvents also an important factor of extraction process because it not only affects the extraction yield but also on the capacity of the extraction process and operating cost [12]. Materials to solvents ratio from 1/7 to 1/11 were investigated. As shown in Figure 2, the highest extraction yield was achieved in sample with material to water ratio of 1/8. Less water may not enough to penetrate through the cell membrane and broke the shell to release the essential oil. But too much water could force more energy to warm up and boiling and also increase device size.

![Figure 2](image2.png)

**Figure 2.** Effect of material to solvent ratio on extraction yield.

3.1.3. The flow rate. The effect of water vapor flow rate ranging from 2.5 – 3.4 g/min on the extraction yield also has been investigated. It could be seen from Figure 3 that the extraction efficiency was increased from 4.0% to 4.6% when the flow rate raised from 2.4 g/min to 2.7 g/min. However, further enhancement of the water flow rate caused a reduction in the extraction yield. The results could be
explained by high mass transfer could be got at high flow rate of water, but if it too high that may result in a large amount of steam and got lost during the condensation phase [13].

![Figure 3](image3.png)

Figure 3. Effect of water flow rate on extraction yield.

3.1.4. Time of extraction. Longer time distillation not just increase operating cost but also degrade the quality of the oil [14]. In this research, the extraction time was investigated from 30 min to 210 min. From the data in Figure 4, longer distillation time caused higher extraction yield of essential oil. As could be seen that the steady increase of extraction yield during the first 180 min but no more essential oil that could be extracted when extraction time increase to 210 min.

![Figure 4](image4.png)

Figure 4. Effect of extraction time on extraction yield.

3.1.5. Concentration of NaCl. Some previous researches have shown that by immersing the sample with NaCl solution before extraction may increase extraction yield [15]. For that reason, in this research A. Longiligulare powder had been soaked into NaCl solution with concentration range of 0 to 18 mg/l for 2 h. The results are shown in Figure 5. A gradual increase in the oil yield were achieved as the concentration of NaCl increased up to 18 mg/l. But, there was no difference when comparing with the experiment without immersion of material into NaCl solution. It could be come from the formation of a starch film around the particle since the high content of flour in A. Longgiligulare seeds.
Figure 5. Effect concentration of NaCl in pretreatment solvent on extraction yield.

3.2. Chemical composition of A. longiligulare essential oils from Ninh Thuan

A. longiligulare essential oils from Ninh Thuan obtained by hydrodistillation method was analyzed using GC-MS for determining the its chemical composition. About 14 components have been identified and shown in Table 2. The major components were D-Limonene (3.794%), D-Camphor (19.15%), Camphor (3.957%), (-)-Bornyl acetate (65.14%), and (E)-Nerolidol (4.64%). With higher bornyl acetate content it could make this essential oil and A. longiligulare powder devalued since bornyl acetate is widely used in food additive, flavouring agent, and odour agent.

Table 2. Chemical compound of A. Longiligulare essential oil by hydrodistillation

| S/N | Rt   | Compounds          | (%)  | Mass | HMS  |
|-----|------|--------------------|------|------|------|
| 1   | 5.343| Camphene           | 1.570| 136  | 945  |
| 2   | 5.970| β– Myrcene         | 1.747| 136  | 950  |
| 3   | 6.772| D-Limonene         | 3.794| 136  | 927  |
| 4   | 9.437| D-Camphor          | 46.714| 152 | 937  |
| 5   | 9.903| Camphol            | 4.86 | 154  | 944  |
| 6   | 12.882| (-)-Bornyl acetate| 31.809| 196 | 931  |
| 7   | 16.249| α-Santalene       | 0.740| 204  | 927  |
| 8   | 16.313| Caryophyllene     | 2.922| 204  | 936  |
| 9   | 17.097| Sesquisabinene     | 0.576| 204  | 905  |
| 10  | 17.798| D-Germacrene      | 1.112| 204  | 918  |
| 11  | 18.353| β – Bisabolene    | 2.027| 204  | 926  |
| 12  | 19.593| (E)-Nerolidol     | 0.491| 222  | 889  |
| 13  | 22.068| β – Bisabolol     | 1.585| 222  | 860  |
| 14  | 22.335| cis-α-Santalol    | 0.627| 220  | 815  |

4. Conclusion

In this study, the optimal condition for hydrodistillation of essential oils from A. Longiligulare seeds growing in Ninh Thuan were investigated. Moreover, GC-MS analysis method revealed the chemical composition of the distilled oil. The optimal condition for hydrodistillation A. Longiligulare powder was at the powder size ≤ 1mm, the ratio of material to water is 1/8 solvent (g/ml), water flow rate at 2.7 g/min, extraction time is 3 h time and using powder without soaking into NaCl solution. With those distillation conditions, the highest extraction yield that obtained was 4.6 ml/100 g dried seed. In addition, 14 main components in A. Longiligulare seeds’ essential oil were detected. The presence of D-Camphor (46.714 %) and Bornyl acetate (31.809 %) as two major ingredients in the essential oil demonstrates the
potential usage of *A. Longiligulare* oil from Ninh Thuan to reduce inflammation, relieve pain and also as a food additive, flavouring agent, and odour agent.

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