

STUDY ON THE ESSENTIAL OIL AND *TRANS*-ANETHOL OBTAINED FROM SUMMER VIETNAMESE ANISE STAR

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ABSTRACT

Summer anise star collected from Lang Son in 2008 was studied. The material was characterized by the essential oil content of 8,75%, moisture content of 13,2%, acid index of 4.5 mg KOH/g, ester index of 254.17 mg KOH/g and the relative density of 0,981. The procedures of essential oil distillation and trans-anethol isolation have been studied based on the properties of anise material. The yield of proposed procedure reached 76,4 % and the purity of anethol isolated was 96,83%.

1. INTRODUCTION

Star anise (*Illicium verum* Hook. f.) belongs to the *Magnoliaceae* family. It is believed that the place of origin of anise is Southwestern of China and Northern of Vietnam [1]. Today it is largely cultivated in China, Vietnam, India, Laos, Philippines, Japan and Korea [2]. However the commercial production of star anise has been limited to China and Vietnam. In Vietnam, Lang Son province is the most important area where provide major amount of anise, but other provinces such as BacKan, ThaiNguyen, CaoBang and Quang Ninh also contribute [1].

The fruit of anise has function as antibacterial, carminative, diuretic, stimulant and stomachic. It is found in receipt for treatment of abdominal pain, digestive disturbances and complains such as lumbago [1, 4]. Recently, shikimic acid extracted from star aniseed is one of the main materials in the antiviral H1N1 Tamiflu drug for influenza. The essential oil of anise is used to flavor soft drinks, bakery products and liqueurs. It is also used as a flavoring agent in confectionery, candy and chewing gum. The oil finds application in a small way in perfumery and in the pharmaceutical industry [5].

The objective of this study was to investigate the essential oil from anise star material of Lang Son harvested in summer, and to develop the method to isolate *trans*- anethol from this essential oil.

2. MATERIALS AND METHOD

2.1. Distillation of essential oils

Star anise was collected in Lang Son on June in 2008. The material was then powdered with crusher. The essential oil was obtained by steam distilling the powders. The oil layer was dried with anhydrous sodium sulfate. The speed of distillation is defined by the ratio of volume distillate in an hour to volume of total aqueous material, expressed in percentage in a hour (%/h).

2.2. Moisture content, Acid index, Ester index and Relative density

The moisture content, acid index, ester index and relative density of anise essential oil have been identified by *TCVN 6119-6128:1996*.

2.3. Anethol analysis

The content of anethol in the essential oil was determined by GC analysis. GC conditions were following: Equipment: SHIMADZU 2010; Column: Equity (30 m × 0.25 mm i.d., 0.25 μm film thickness); Column temperature: 70°C (2 min) - 230°C (10 min) at 7°C/min; Carrier gas: N₂; Flow rate: 1.2 mL/min; Detector: FID; Detector temperature: 230°C.

3. RESULT AND DISCUSSION

3.1. Moisture content, Acid index, Ester index and relative density

Analyses have been carried out in triplicate and the result was the average of the three values obtained.

Moisture content of anise star material was 13,2%.

Acid index of the essential oil of anise star was 4.5 mg KOH/g.

Ester index of the essential oil of anise star was 254.17 mg KOH/g.

The relative density of the essential oil of anise star was 0,981.

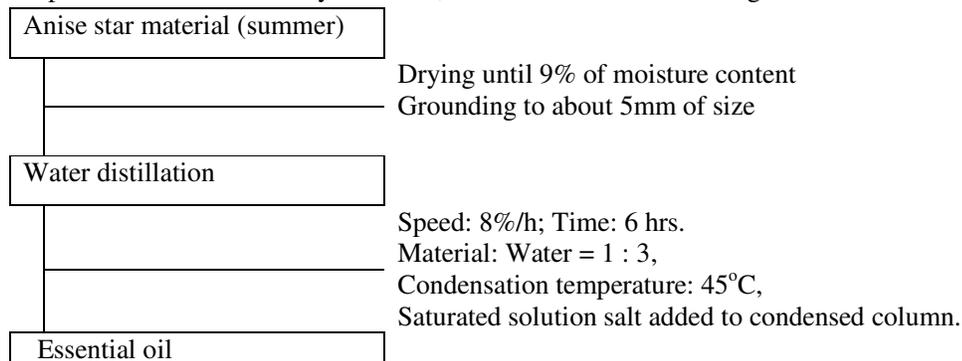
These values of acid and ester index have indicated that the essential oil has just analyzed after being distilled and the types of ester are various. The density obviously showed that the difference between water and anise star oil is too small so that they can be separated on the liquid phase, this makes difficulty on the separation during the process of distillation.

3.2. Essential oil distillation

The content of essential oil yielded from Lang Son summer anise star was 8.75% (calculated by drying mass), which is quite low in comparison with season material (about 13%) [8].

Concerning to the density of anise star essential oil (0,981) and water as well as anise star moisture content which is rather high (13,2%) the ordinary distillation has been studied for better separation.

The procedure final with the yield of 76,4% is described as following:



Scheme 1. Anise star essential oil distillation procedure

3.3. Anethol analysis

Anethol content of the summer anise star essential oil was 88.82 % at relative percentage as shown in Figure 1, which is in the range reported earlier for season anise essential oil (more than 80%)

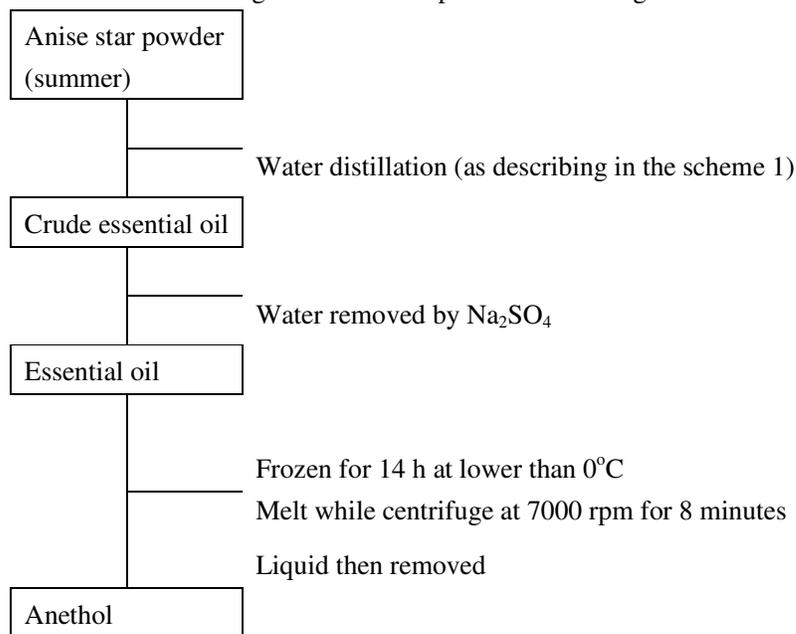
3.4. Extraction of anethol from anise star

Anethol is one of the aromatic compounds. It occurs widely in nature essential oils. It is used as flavoring agent. In addition, it has sweet taste, 13 times sweeter than sugar. Anethol is used in alcoholic drinks, seasoning and confectionery productions [2].

The essential oils obtained from Vietnamese summer anise star have been used to isolate anethol. The essential oil was frozen for 14 hours at less than 0°C, then melted while centrifuging at 7000 rpm for 8 min at room temperature. Anethol was obtained after removing liquid.

The anethol obtained was analyzed by GC. The GC analysis was performed with the same conditions as reported earlier for essential oil analysis.

The procedure has been investigated and developed is as followings:



Scheme 2. Method of isolation of anethol from anise star

The obtained anethol content was 96.83 % by GC analysis (Figure 2). The yield from essential oils was 80% (about 80 ml anethol from 100 ml essential oil, which is of 87% of theoretical).

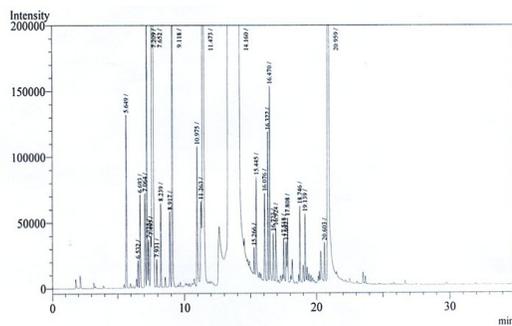


Figure 1. Chromatogram of anise star essential oil

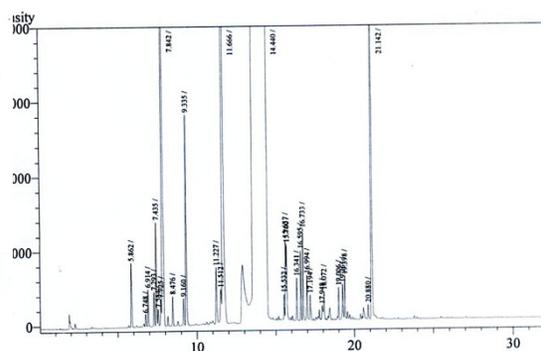


Figure 2. Chromatogram of isolated purified anethol from anise star essential oil

4. CONCLUSION

Summer anise star from LangSon has been used for distillation; the essential oil obtained was at 8.75%, a quite low amount as usual as season anise star material. The essential oil was analyzed for trans-anethol content, an index for the quality of anise essential oil. Acid index of 4.5 mg KOH/g, ester index of 254.17 mg KOH/g, the relative density of 0,981 and the anethol content of 88.82% have been indicated that the anise star material despite from summer harvest with low content of oil but it was as good as being used as source for flavor extraction. Based on these properties as indicator, a procedure for essential oil distillation has been studied on several factors including material moisture, distillation speed, ratio of the material and water. The yield of proposed procedure reached 76,4%. Another procedure for anethol isolation has been developed and the anethol content of 96.83% has been obtained.

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REFERENCES

1. K. V. Peter - Handbook of herbs and spices, Woodhead Publishing, 2004, pp. 290-296.
2. R. Susheela - Handbook of spices, seasoning, and flavorings, CRC Press, 2006, pp.173-174.
3. E. A. Weiss - Spice crops, CABI Publishing, 2002, pp. 228-231.
4. http://en.wikipedia.org/wiki/Star_anise
5. B. H. Henry - Source book of flavors, Springer, 1981, pp.221-222.
6. <http://www.chem-station.com/molecule/archives/2007/10/-tamiflu-oseltamivir-1/>
7. K. A. Anderson, W. T. Cobb and B. R. Loper - Analytical method for determination of shikimic acid, Plant anal. **32** (17&18) (2001) 2831-2840.
8. Nguyen Van Duong – Project: Situation of equipment for anise essential oil production in Vietnam – Tendency for design of distillation system at small scale, Forest Science Institute of Vietnam, 2009

TÓM TẮT

NGHIÊN CỨU QUY TRÌNH TÁCH TINH DẦU VÀ *TRANS* ANETHOL TỪ ĐẠI HỒI VỤ CHIÊM CỦA VIỆT NAM

Tinh dầu hồi trong nguyên liệu đại hồi Lạng Sơn thu hoạch vào vụ chiêm năm 2008 chỉ chiếm 8,75% và có chứa hàm lượng trans-anethol là 88,82%. So với đại hồi nguyên liệu thu hoạch chính vụ, hàm lượng tinh dầu thấp hơn đáng kể (13%). Và tỉ trọng của tinh dầu này (0,981) gần với tỉ trọng của nước do vậy việc phân tách tinh dầu và nước trong quá trình chưng cất tương đối khó khăn. Quy trình chưng cất đạt hiệu suất 76,4% dựa trên các yếu tố vận tốc chưng cất (8%) và nhiệt độ ngưng tụ (45°C) đã được nghiên cứu. Tuy nhiên hàm lượng trans-anethol có chứa trong tinh dầu thu được khá cao (88,82%) do vậy quy trình tách anethol, một trong những hợp chất thơm tự nhiên có giá trị cao và được sử dụng rộng rãi trong các lĩnh vực của sản xuất và đời sống, như dùng làm hương liệu trong thực phẩm và dược liệu cũng đã được đề xuất thông qua quá trình kết tinh lạnh, kết quả thu được anethol có hàm lượng 96,83%.

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