Title: METHOD TO EXTRACT BORNEOL FROM THE EXUDATES OF DRYOBALANOPS AROMATICA

Abstract: The invention relates to a method to extract Borneol from the exudates of Dryobalanops aromatica. Extraction from exudates will not have major injuries to the plant, thus leading to the sustainability for the source of Borneol. More importantly, Dryobalanops aromatica is found to have high distribution in Peninsular Malaysia. Borneol is an expensive compound that has been used in various traditional medicine applications. Due to the shortage of natural source of Borneol and the high cost involved, the majority of the Borneol in the market is the synthetic Borneol which has toxic side effect. It presents naturally in two different forms, namely as d-Borneol and l-Borneol. d-Borneol is the main component found in Dryobalanops aromatic. d-Borneol can be extracted from the exudates by using fractional distillation method of this invention.
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Method to Extract Borneol From the Exudates of Dryobalanops Aromatica

Field of Invention

The present invention relates to a method to extract Borneol from the exudates of Dryobalanops aromatica. Extraction from exudates will not have major injuries to the plant, thus leading to the sustainability for the source of Borneol.

Background of the invention

Borneol (C10H18O) is a bi-cyclic mono-terpenoid alcohol, one of the valuable medical material, senior aromatic species, chemical materials, has been used in food and also folk medicine in China and India. According to the Pharmacopoeia of People's Republic of China, borneol is an important ingredient among about 63 herbal products. There are two different kinds of borneol; (a) synthetic borneol which is a mixture of DL-borneol and isoborneol, in which the DL-borneol content should be no less than 55%, and; (b) natural borneol whose main component is D-borneol, which should be more than 95% of natural borneol. Previous studies showed that synthetic borneol degraded slowly during storage and noxious camphor level might be as high as 45 - 97% with
concomitant lower levels of synthetic borneol. These results indicated that camphor was a degradation product of synthetic borneol. It is widely known that camphor is toxic, whereas natural borneol is non-toxic. Therefore, natural borneol is preferred instead of synthetic borneol in different borneol products.

Natural borneol is mainly extracted from the essential oils of numerous medicinal plants of the families Dipterocarpaceae (for example, Dipterocarpus turbinatus tree), Lamiaceae (for example, Rosmarinus officinalis or Salvia officinalis), Valerianaceae (for example, Valeriana officinalis) or Asteraceae (for example, Matricaria chamomilla). Since there was a shortage of natural source, the price of natural borneol was gradually increased. New sources of natural borneol would be urgently needed.

Borneol is an expensive compound that has been used in various traditional medicine formulas. It is a monoterpane that present in crystalline form; and is easily sublimated although has a high melting point (−202–208 °C) and boiling point (212 °C). It presents naturally in two different forms, namely as d-Borneol and 2-Borneol. d-Borneol is the main component found in Dryobalanops aromatica while I-Borneol can be found in Blumea balsamifera. Dryobalanops aromatica
is distributed mainly in Peninsular Malaysia, Borneo and Sumatra while *Blumea balsamifera* can be found in South China.

Due to the shortage of natural source of Borneol and the high cost involved, the majority of the Borneol in the market is the synthetic Borneol, *Borneolum syntheticum* that produced from turpentine oil or camphor in order to fulfill the high market demand. Nevertheless, there have been studies showing that synthetic Borneol possesses toxicity and side effects due to the presence of iso-Borneol and trace amount of Camphor. On the other hand, d-Borneol has been suggested to have greater biological effect with less toxicity compared to the 2-Borneol and *Borneolum syntheticum*.

Therefore, extraction of the natural d-Borneol with high purity is crucial for better medicinal value. In recent years, China managed to extract high purity of Borneol from the leaves of *Cinnamomum camphora* (78.6%) by using Clevenger typed hydrodistillation followed by sublimation process.

In view of the high distribution of *Dryobalanops aromatica* in Peninsular Malaysia and its greater source for d-Borneol, a study is being conducted to extract Borneol from the
exudates samples without any major injuries to the plants, thus leading to the sustainability for the source of Borneol.

These and other objects and advantages of the present invention will become apparent to those skilled in the art from a consideration of the following specification and claims.

**Summary of the Invention**

Borneol is an expensive compound that has been used in various traditional medicine applications. Due to the shortage of natural source of Borneol and the high cost involved, the majority of the Borneol in the market is the synthetic Borneol which has toxic side effect.

It presents naturally in two different forms, namely as d-Borneol and l-Borneol. d-Borneol is the main component found in Dryobalanops aromatica which is available easily in Peninsular Malaysia. d-Borneol can be extracted from the exudates by using fractional distillation method of this invention.
Brief Description of the Drawings

Other objects, features, and advantages of the invention will be apparent from the following description when read with reference to the accompanying drawings. In the drawings, wherein like reference numerals denote corresponding parts throughout the several views:

Figure 1 shows the fractional distillation apparatus used for extracting Borneol from the exudates of Dryobalanops aromatica.

Detailed Description of the Preferred Embodiments

In the following detailed description, numerous specific details are set forth in order to provide a thorough understanding of the invention. However, it will be understood by those of ordinary skill in the art that the invention may be practiced without these specific details.

In other instances, well-known methods, procedures and/or components have not been described in detail so as not to obscure the invention. Reference will now be made in detail to the preferred embodiments of the present invention, examples of which are illustrated in the accompanying drawings.
With reference to Figure 1, Borneol was extracted from the exudates using fractional distillation apparatus, consisting of heating mantle (10), round bottom flask (12), fractionating column (13), plain stillhead (14), cone screwthread adapter connected to thermometer (15), Liebig condenser (16), plain bend still (17) and collecting bottle (18).

Briefly, exudates were fractional distilled in double distilled water, where the heating level at heating mantle (10) was adjusted by using heat control level (11) from 10.0 to 5.0 for 20 to 30 minutes. Once the sample mixture started to boil slowly and evenly, the heating level was controlled by using heat control level (11) at 1.0 to 3.0. Resulting vapor pressure produced will bring up the more volatile vapor mixture through the fractionating column (13), plain stillhead (14), cone screwthread connected to thermometer (15), and condensed at Liebig condenser (16), finally collected as distillate product at the collecting bottle (18).

The vapor temperature was detected by the connected thermometer at cone screwthread adapter (15). The temperature was maintained at 85 to 90°C for two hours with constant boiling of sample mixture.
Following the process, the apparatus was cooled down at room temperature for about two hours. The final distillate containing volatile essential oil and distilled water was separated using separating column (width of 1cm).

On the other hand, some less volatile compounds condensed at the fractionating column (13), plain stillhead (14), cone screwthread adapter (15) and Liebig condenser (16) were collected as well by rinsing with absolute ethanol. All the samples collected were stored at -20°C.

Following gas chromatography mass spectrometry (GCMS) analysis, d-Borneol was found to be present in fractionating column (13).

Besides fractional distillation, Borneol can be extracted and purified from the exudates samples using column chromatography, followed by solid phase extraction method.

Briefly, exudates was layered on top of the column bed packed with Silica gel 60 at the height of 20 to 25cm with diameter of 3cm. Samples eluted with a stepwise gradient solvent system of n-hexane with increasing proportions of ethyl acetate. The collected fractions was analyzed by HPTLC
and was pooled based on the Rf value on TLC plate. A C18 reverse phase SPE cartridge was used to further purify Borneol from the Borneol containing fractions, where the fraction was eluted with an isocratic solvent system of 60:40 acetonitrile in water. Single band of Borneol was detected on TLC.

As will be readily apparent to those skilled in the art, the present invention may easily be produced in other specific forms without departing from its essential characteristics. The present embodiments is, therefore, to be considered as merely illustrative and not restrictive, the scope of the invention being indicated by the claims rather than the foregoing description, and all changes which come within therefore intended to be embraced therein.
Claims

1. A fractional distillation apparatus to extract Borneol from the exudates of Dryobalanops aromatica comprising:

a heating mantle (10) to heat up the exudates together with the distilled water which are contained in a round bottom flask (12);

a fractionating column (13) with its bottom opening to be connected to the round bottom flask (12) to perform fractionation of the exudates of Dryobalanops aromatic vapor pressure;

a plain stillhead (14) with its bottom opening to be connected to the upper opening of fractionating column (13), the upper opening to be connected to a cone screwthread adapter connected to thermometer (15) and side opening to be connected to the upper inlet of a Liebig condenser (16); and

a collecting bottle (18) to be connected to the lower outlet of the Liebig condenser (16) through a plain bend still (17).

2. The fractional distillation apparatus to extract Borneol from the exudates of Dryobalanops aromatica as claimed in claim 1, wherein the fractionating column (13) height will need to be 300 to 600mm.
3. The fractional distillation apparatus to extract Borneol from the exudates of Dryobalanops aromatica as claimed in claim 1, wherein the Liebig condenser length will need to be 250mm.

4. The fractional distillation apparatus to extract Borneol from the exudates of Dryobalanops aromatica as claimed in claim 1, wherein the exudates of Dryobalanops aromatica which is mixed with double distilled water to be heated by the heating mantle (10) by adjusting the heat control level (11) from 10.0 to 5.0 for 20 to 30 minutes.

5. The fractional distillation apparatus to extract Borneol from the exudates of Dryobalanops aromatica as claimed in claim 1, wherein the heat control level (11) will be controlled at 1.0 to 3.0 once the mixture starts to boil slowly and evenly.

6. The fractional distillation apparatus to extract Borneol from the exudates of Dryobalanops aromatica as claimed in claim 1, wherein the temperature will be maintained at 85 to 90°C for two hours with constant boiling of the mixture by using the thermometer which is connected at the cone screwthread adapter (15).
7. The fractional distillation apparatus to extract Borneol from the exudates of Dryobalanops aromatica as claimed in claim 1, wherein the final distillate will contain volatile essential oil and distilled water which can be separated by using separating column with the width of lcm.

8. The fractional distillation apparatus to extract Borneol from the exudates of Dryobalanops aromatica as claimed in claim 1, wherein the extracted Borneol will need to be stored at -20°C.

9. The fractional distillation apparatus to extract Borneol from the exudates of Dryobalanops aromatica as claimed in claim 1, wherein the d-Borneol was found to be present in the fractionating column (13) by using gas chromatography mass spectrometry (GCMS) analysis.

10. A method of extracting Borneol from the exudates of Dryobalanops aromatica by using fractional distillation, said method comprising the steps of:
heating up the exudates together with the distilled water which are contained in a round bottom flask (12) by using a heating mantle (10);
fractionating the exudates of Dryobalanops aromatic vapor pressure by using a fractionating column (13) with its bottom opening to be connected to the round bottom flask (12); condensing the exudates of Dryobalanops aromatic vapor pressure from the fractionating column (13) by using a Liebig condenser (16); and collecting the distillate by using a collecting bottle (18) which is connected to the lower outlet of the Liebig condenser (16) through a plain bend still (17).
## A. CLASSIFICATION OF SUBJECT MATTER

**BOW 3/14 (2006.01)**

According to International Patent Classification (IPC) or to both national classification and IPC.

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched.

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

Google. Keywords: dryobalanops aromatica

STN, CAPlus: DRYOBALANOPS OR KAPUR OR DIPTEROCARP? OR PTERIGIUM OR SHOREA OR (BORNEO OR TREE OR MALAY OR SUMATRAN) (A) CAMPHOR (L) DISTILL? OR FRACTIONAT?

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

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Date of the actual completion of the international search  
24 October 2013

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