

METHOD OF PREPARING PATCHOULI EXTRACT

BACKGROUND

Technical Field

5 [0001] The present disclosure relates to a method for preparing a patchouli extract, and in particular to a method for extracting a patchouli extract having patchouli alcohol.

Description of Related Art

[0002] Patchouli oil is the main medicinal ingredient of patchouli. With the increase of research in pharmacological activity and chemical composition of patchouli oil, the value of patchouli oil has received increasing attention. Patchouli oil has good effects in terms of
10 anti-inflammatory, antibacterial activity, anti-oxidation and analgesic effects.

[0003] Patchouli alcohol is a natural monomer compound with a special aromatic odor extracted from patchouli. Patchouli alcohol can be used in the fragrance or perfume industry. In addition, patchouli alcohol can be used to treat fungal skin diseases, and can also be used as a
15 base material synthesized through other compounds, and therefore the commercial value of patchouli alcohol is increased.

[0004] Therefore, how to find a method for extracting patchouli oil with high-purity patchouli alcohol from patchouli is an issue that practitioners are currently trying to solve.

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SUMMARY

[0005] The present disclosure provides a method for preparing a patchouli extract, which can effectively obtain a patchouli extract having high-purity patchouli alcohol.

[0006] Embodiments of the present disclosure provide a method of preparing a patchouli extract. The method includes the following steps. First, a patchouli material is grounded into

patchouli powder and pelleted to obtain patchouli granules. Then, liquid carbon dioxide is introduced to perform a supercritical carbon dioxide extraction process on the patchouli granules, wherein the extraction pressure is about 110 bar to 180 bar and the extraction temperature is about 40°C to 60°C. During the supercritical carbon dioxide extraction process, the extracted product obtained, based on the total weight of the introduced liquid carbon dioxide not exceeding 10 times the weight of the patchouli granules, is collected as the first patchouli extract, wherein the first patchouli extract includes the first patchouli oil. During the supercritical carbon dioxide extraction process, the extracted product obtained, based on the total weight of the introduced liquid carbon dioxide exceeding 10 times the weight of the patchouli granules but not exceeding 15 times the weight of the patchouli granules, is collected as the second patchouli extract, wherein the second patchouli extract includes the second patchouli oil.

[0007] In an embodiment of the present disclosure, the method further includes drying the patchouli material, such that the moisture content of the dried patchouli material is about 15% or less.

[0008] In an embodiment of the disclosure, the patchouli material includes a patchouli leaf, a patchouli stem, or a combination thereof.

[0009] In an embodiment of the disclosure, the size of the patchouli powder is about 140 mesh or less.

[0010] In an embodiment of the disclosure, the patchouli granules have a diameter of about 2 mm to 10 mm, and the thickness of the patchouli granules is about 2.0 mm to 4.5. mm.

[0011] In an embodiment of the disclosure, the extraction pressure is about 120 bar to 150 bar.

[0012] In an embodiment of the disclosure, the first patchouli oil has a melting point of about 10°C to 40°C, and the second patchouli oil has a melting point of about 40°C to 70°C.

[0013] In an embodiment of the disclosure, the amount of the patchouli alcohol in the first

patchouli oil is about 10 wt% to 40 wt%, and the amount of the patchouli alcohol in the second patchouli oil is about 10 wt% to 30 wt%.

[0014] In an embodiment of the disclosure, the method further includes centrifuging the first patchouli extract to separate the first patchouli oil from the patchouli wax in the first patchouli
5 extract.

[0015] In an embodiment of the present disclosure, the amount of the patchouli alcohol in the patchouli wax is about 10 wt% to 30 wt%.

[0016] Embodiments of the present disclosure provide a method of preparing a patchouli extract. The method includes the following steps. First, a patchouli material is grounded into
10 patchouli powder and pelleted to obtain patchouli granules. Then, liquid carbon dioxide is introduced to perform a supercritical carbon dioxide extraction process on the patchouli granules, wherein the extraction pressure is about 110 bar to 180 bar and the extraction temperature is about 40°C to 60°C. The flow rate of the liquid carbon dioxide is 15 kg/hour. During the supercritical carbon dioxide extraction process, the extracted product obtained within an
15 extraction time not exceeding 0.5 hour is collected as the first patchouli extract, wherein the first patchouli extract includes the first patchouli oil. During the supercritical carbon dioxide extraction process, the extracted product obtained within an extraction time exceeding 0.5 hour but not exceeding 1 hour is collected as the second patchouli extract, wherein the second patchouli extract includes the second patchouli oil.

20 [0017] In an embodiment of the present disclosure, the method further includes drying the patchouli material, such that the moisture content of the dried patchouli material is about 15% or less.

[0018] In an embodiment of the disclosure, the patchouli material includes a patchouli leaf, a patchouli stem, or a combination thereof.

- [0019] In an embodiment of the disclosure, the size of the patchouli powder is about 140 mesh or less.
- [0020] In an embodiment of the disclosure, the patchouli granules have a diameter of about 2 mm to 10 mm, and the thickness of the patchouli granules is about 2.0 mm to 4.5. mm.
- 5 [0021] In an embodiment of the disclosure, the extraction pressure is about 120 bar to 150 bar.
- [0022] In an embodiment of the disclosure, the first patchouli oil has a melting point of about 10°C to 40°C, and the second patchouli oil has a melting point of about 40°C to 70°C.
- [0023] In an embodiment of the disclosure, the amount of the patchouli alcohol in the first patchouli oil is about 10 wt% to 40 wt%, and the amount of the patchouli alcohol in the second
10 patchouli oil is about 10 wt% to 30 wt%.
- [0024] In an embodiment of the disclosure, the method further includes centrifuging the first patchouli extract to separate the first patchouli oil from the patchouli wax in the first patchouli extract.
- [0025] In an embodiment of the present disclosure, the amount of the patchouli alcohol in the
15 patchouli wax is about 10 wt% to 30 wt%.
- [0026] Based on the above, the method for preparing the patchouli extract of the present disclosure extracts the patchouli extract having patchouli alcohol from the patchouli granules by using supercritical carbon dioxide, which not only can effectively improve the extraction efficiency, but also can obtain the patchouli oil with high-purity patchouli alcohol.
- 20 [0027] In order to make the aforementioned features and advantages of the disclosure more comprehensible, embodiments accompanying figures are described in detail below.

BRIEF DESCRIPTION OF THE DRAWINGS

- [0028] FIG. 1 is a pipeline flow diagram of a supercritical fluid extraction device according to

an embodiment of the present disclosure.

FIG. 2 is a gas chromatography-mass spectrometry diagram of patchouli oil.

FIG. 3 shows the normalized extraction curves of Example 12, Example 13, Comparative Example 1 and Comparative Example 2.

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DESCRIPTION OF THE EMBODIMENTS

[0029] A method for preparing a patchouli extract according to an embodiment of the present disclosure can be used for extracting a patchouli extract having the ingredient of patchouli alcohol. In this manner, a patchouli extract having high-purity patchouli alcohol can be
10 obtained.

[0030] The following examples are given to illustrate the details or conditions of the production method of the present disclosure, but these examples are not intended to limit the scope of the present disclosure. The drawings are schematic diagrams presented for illustrative purpose, and should not be construed as a limitation to the actual method, condition or device.

15 [0031] **[Preparation of Patchouli Extract]**

[0032] In this embodiment, a supercritical fluid extraction (SFE) device as shown in FIG. 1 can be used to prepare the patchouli extract. FIG. 1 is a pipeline flow diagram of a supercritical fluid extraction device according to an embodiment of the present disclosure.

[0033] First, a patchouli material is dried (for example, by using an oven) so that the moisture
20 content of the dried patchouli material is about 15% or less. In an embodiment, the moisture content of the dried patchouli material is about 12% or less. The patchouli material is, for example, a patchouli leaf, a patchouli stem, or a combination thereof.

[0034] Next, the patchouli material is grounded and/or cut through a mechanical or physical process until the patchouli powder having a size of about 200 mesh or less is obtained. In an

embodiment, the size of the patchouli powder is about 40 mesh to 200 mesh. In an embodiment, the size of the patchouli powder is about 40 mesh to 140 mesh.

[0035] In this embodiment, the patchouli powder may be further pelleted to obtain patchouli granules or pellets. The patchouli granules have a diameter of about 2 mm to 10 mm, and the patchouli granules have a thickness of about 2.0 mm to 4.5 mm. In an embodiment, the patchouli granules have a thickness of about 2.5 mm to 3.5 mm. In an embodiment, the patchouli granules have a thickness less than 2.7 mm. If the thickness of the patchouli granules is greater than 5 mm, it will cause difficulty in permeation of carbon dioxide and a problem of lower mass transfer rate. In addition, performing extraction by using the patchouli granules can prevent occurrence of channeling during extraction. Moreover, since the patchouli powder is pelleted to form the patchouli granules, the bulk density of the patchouli granules can be significantly increased from 0.3-0.4 to 0.5-0.6, which increases the production rate of the supercritical fluid extraction device.

[0036] Then, the patchouli granules are placed in an extractor 30 of 5L and subjected to a supercritical carbon dioxide extraction process. Specifically, liquid carbon dioxide is pumped into the extractor 30 from a working tank 40 through a high pressure pump 12. In this embodiment, the extraction pressure is about 110 bar to 180 bar, and the extraction temperature is about 40°C to 60°C. In an embodiment, the extraction pressure is 120 bar ~150 bar. The flow rate of carbon dioxide is about 10 kg/hour to 40 kg/hour. In an embodiment, the flow rate of carbon dioxide is from about 15 kg/hour to about 30 kg/hour. The flow rate of carbon dioxide is measured by a coriolis flow meter 20. The temperature of the extractor 30 is controlled by a circulating water heater 35, and the pressure of the extractor 30 is controlled by a back pressure regulator 37.

[0037] The mixture of the extracted carbon dioxide fluid and the patchouli extract enters a

separator 10 for decompression separation. The pressure in the separator 10 is about 45 bar to 65 bar, and the temperature in the separator 10 is about 40°C to 60°C. The temperature of the separator 10 is controlled by the circulating water heater 15.

[0038] During the decompression separation process, the carbon dioxide fluid is heated and gasified, thereby reducing the solubility of the patchouli extract, leaving the patchouli extract in the separator 10 and achieving gas-liquid separation. The gasified carbon dioxide is cooled by a condenser 50 to be liquefied and sent back to the working tank 40 to achieve recirculation of carbon dioxide.

[0039] During the supercritical carbon dioxide extraction process, the patchouli extract at different time points is collected by opening the valve at the bottom of separator 10. In an embodiment, during the supercritical carbon dioxide extraction process, when the total weight of the introduced carbon dioxide does not exceed 10 times the weight of the patchouli granules, the first patchouli extract is obtained and collected. In an embodiment, during the supercritical carbon dioxide extraction process, when the total weight of the introduced carbon dioxide exceeds 10 times the weight of the patchouli granules but not exceed 15 times the weight of the patchouli granules, the second patchouli extract is obtained and collected. In an embodiment, the flow rate of the carbon dioxide is 15 kg/hour, and during the supercritical carbon dioxide extraction process, when the extraction time does not exceed 0.5 hour, the first patchouli extract is obtained and collected. In an embodiment, the flow rate of the carbon dioxide is 15 kg/hour, and during the supercritical carbon dioxide extraction process, when the extraction time exceeds 0.5 hour but not exceed 1 hour, the second patchouli extract is obtained and collected.

[0040] In this embodiment, the melting point of the first patchouli extract is lower than the melting point of the second patchouli extract. The first patchouli extract has a melting point not higher than room temperature, and therefore the first patchouli extract is an oily extract at room

temperature. The first patchouli extract is orange. The second patchouli extract has a melting point higher than room temperature, and therefore the second patchouli extract is a paste extract at room temperature. The second patchouli extract is dark green.

[0041] In this embodiment, the first patchouli extract includes the first patchouli oil and the second patchouli extract includes the second patchouli oil. The melting point of the first patchouli oil is lower than the melting point of the second patchouli oil. In this embodiment, the second patchouli oil has a melting point higher than room temperature. The second patchouli extract can be melted by heating, and the melted product is the second patchouli oil. Therefore, the second patchouli extract can be heated at different temperatures to obtain second patchouli oil of different melting points. In this embodiment, the first patchouli oil has a melting point of about 10°C to 40°C, and the second patchouli oil has a melting point of about 40°C to 70°C.

[0042] In the present embodiment, the amount of the patchouli alcohol in the first patchouli oil is about 10 wt% to 40 wt%, and the amount of the patchouli alcohol in the second patchouli oil is about 10 wt% to 30 wt%.

[0043] In this embodiment, the first patchouli extract further includes patchouli wax, wherein the patchouli wax has a melting point higher than 80°C. In this embodiment, the first patchouli extract may be further centrifuged to separate the first patchouli oil from the patchouli wax. The amount of the patchouli alcohol in the patchouli wax is about 10 wt% to 30 wt%.

20 [0044] [Gas Chromatography-Mass Spectrometry (GC/MS) of Patchouli Extract]

[0045] In this embodiment, the main ingredient in the patchouli oil was analyzed through GC/MS. The column adopted was a DB-5MS capillary column (30 m × 0.250 mm, 0.25 μm, Agilent, USA) installed on the gas chromatography-mass spectrometry (Agilent, 7890B GC/5977A MSD). The temperature of the injector and detector are set at 250°C.

[0046] In the embodiment, for instance, a sample of patchouli oil is prepared in the following manner: the stem and the leaf of the patchouli are grounded by using a grinder, respectively, and the same weight of the stem powder and the leaf powder are mixed to obtain the patchouli powder. Next, 0.5 kg of patchouli powder was extracted by using a supercritical fluid
 5 extraction device similar to that of FIG. 1 to obtain the patchouli oil. The extraction conditions are: extraction pressure is 5 Mpa~5.5 Mpa, extraction temperature is 40°C, flow rate of carbon dioxide is 15 kg/hour, extraction time is 0.5 hour.

[0047] The GC/MS spectrum of the sample is shown in FIG. 2. FIG. 2 is a gas chromatography-mass spectrometry diagram of patchouli oil. In FIG. 2, pentadecane (No. 6) is
 10 adopted for the internal standard IS. The GC/MS spectrum clearly shows the peak position of each of the main ingredients in the patchouli oil, and the result is used as a standard for analysis. The main ingredients of the patchouli oil include: caryophyllene (No. 1), alpha-Guaiene (No. 2), seychellene (No. 3), α -patchoulene (No. 4), beta-maaliene (No. 5), Aciphyllene (No. 7), α -Bulnesene (No. 8) and patchouli alcohol (No. 9).

15 [0048] Table 1 lists the nine ingredients of FIG. 2 and their area fractions.

[0049] [Table 1]

No.	Ingredient	Area fraction (%)	No.	Ingredient	Area fraction (%)
1	caryophyllene	2.76	6	pentadecane	17.35
2	alpha-Guaiene	10.10	7	Aciphyllene	2.08
3	seychellene	6.18	8	α -Bulnesene	13.08
4	α -patchoulene	3.7	9	patchouli alcohol	33.44
5	beta-maaliene	1.7			

[0050] [Calculation of weight fraction of patchouli alcohol]

[0051] In the present embodiment, the weight fraction of the patchouli alcohol in the obtained patchouli oil can be calculated through the following Equation 1 (calibration curve).

[Equation 1]

$$\frac{A_{POH}}{A_{IS}} = m \frac{V_{POH} \times C_{POH}}{V_{IS} \times C_{IS}}$$

In Equation 1, A is the peak area in the GC/MS spectrum, V is the volume, C is the concentration, the subscripted POH and the subscripted IS represent the patchouli alcohol and the internal standard, respectively, and m is the corresponding factor of the calibration curve, m=0.16. The concentration (represented by mg/L) of the patchouli alcohol in the patchouli oil can be obtained through the calibration curve. The total weight of the patchouli oil in 1L solution can be calculated when preparing samples. Therefore, by dividing the concentration of the patchouli alcohol by the total weight of the patchouli oil, the weight fraction of the patchouli alcohol in the patchouli oil can be obtained.

[0052] [The establishment of a normalized extraction curve]

[0053] In this embodiment, in order to evaluate and compare each extraction, a normalized extraction curve is established. The typical extraction curve is a curve diagram of E vs. q, where E is defined as the weight ratio of the patchouli oil to the loaded material, and q is the weight ratio of the accumulated weight of CO₂ to the loaded material. In this embodiment, E can be used as the extraction yield of the patchouli oil. In the normalized extraction curve, E is divided by total extractable oil X_o. The normalized extraction curve means (E/X_o). After E is divided by X_o, the extraction yield is normalized, so it is called the normalized extraction curve.

[0054] In the following, examples will be given to describe the details or conditions of the preparation method of the patchouli extract in the present disclosure.

[0055] Experimental Example 1: Effect of Operation Conditions on Extraction Yield

[0056] [Example 1]

[0057] By using a grinder, the stems and leaves of the patchouli are grounded, respectively, and the same weight of the stem powder and the leaf powder are mixed to obtain the patchouli powder. Next, 1 kg of the patchouli powder was extracted by using a supercritical fluid
5 extraction device similar to that of FIG. 1 to obtain the patchouli oil. The extraction conditions are: extraction pressure is 140 bar, extraction temperature is 40°C, flow rate of carbon dioxide is 15 kg/hour, extraction time is 0.5 hour.

[0058] [Example 2]

[0059] The patchouli oil was prepared in a manner similar to that of Example 1, except that the
10 extraction temperature of Example 2 was 50°C.

[0060] [Example 3]

[0061] The patchouli oil was prepared in a manner similar to that of Example 1, except that the flow rate of carbon dioxide of Example 3 was 30 kg/hour.

[0062] [Example 4]

15 **[0063]** The patchouli oil was prepared in a manner similar to that of Example 1, except that the extraction pressure of Example 4 was 150 bar.

[0064] [Example 5]

[0065] The patchouli oil was prepared in a manner similar to that of Example 1, except that the extraction temperature of Example 5 was 50°C and the extraction pressure was 150 bar.

20 **[0066] [Example 6]**

[0067] The patchouli oil was prepared in a manner similar to that of Example 1, except that the extraction pressure of Example 6 was 150 bar, and the flow rate of carbon dioxide was 30 kg/hour.

[0068] Then, the extraction yield, the amount of the patchouli alcohol in the patchouli oil, and

moisture, acidic value and ester value of the patchouli oil of the patchouli oil collected in Examples 1 to 6 were measured, the measurement results are shown in Table 2.

[0069] [Table 2]

	Extraction yield (%)	Amount of patchouli alcohol (weight fraction) (%)	Moisture of patchouli oil (%)	Acidic value (mg-KOH/g-sample)	Ester value
Example 1	3.85	27.1	0.7	25.9	45
Example 2	3.24	20.7	0.3	21.6	60.9
Example 3	3.67	21.2	1.77	30.3	44.9
Example 4	4.26	26.9	0.9	27	39.3
Example 5	4.59	26.3	0.7	24.8	47.6
Example 6	3.44	24.3	1.6	28.8	45

[0070] It can be seen from the results of Table 2 that the extraction yield under high flow rate of carbon dioxide (i.e., Examples 3, 6) did not reach 4%. It was inferred that a large amount of carbon dioxide in the separator brings the extracted patchouli oil into the working tank, resulting in a decrease in the production of the patchouli oil. Under the condition that the extraction temperature is 50°C and the extraction pressure is 150 bar (i.e., Example 5), the highest extraction yield can be achieved.

10 [0071] **Experimental Example 2: Effect of pelletization on extraction yield**

[0072] [Example 7]

[0073] By using a grinder, the stems and leaves of the patchouli were separately grounded until the powder at a size of 140 mesh was obtained, and the same weight of the stem powder and the leaf powder were mixed to obtain the patchouli powder. Next, the patchouli powder was

pelleted to obtain thick pressed pellets. The thick pressed pellets have a bulk density of 0.63 (g/mL). Then, 1.28 kg of the thick pressed pellets were extracted by using a supercritical fluid extraction device similar to that of FIG. 1 to obtain the patchouli oil. The extraction conditions are: extraction pressure is 150 bar, extraction temperature is 50°C, flow rate of carbon dioxide is 15 kg/hour, extraction time is 0.5 hour.

[0074] [Example 8]

[0075] By using a grinder, the leaves of the patchouli were grounded until the leaf powder at a size of 140 mesh was obtained. Next, the leaf powder was pelleted to obtain thick pressed pellets. The thick pressed pellets have a bulk density of 0.63 (g/mL). Then, 1.3 kg of the thick pressed pellets were extracted by using a supercritical fluid extraction device similar to that of FIG. 1 to obtain the patchouli oil. The extraction conditions are the same as in Example 7.

[0076] [Example 9]

[0077] By using a grinder, the stems and leaves of the patchouli were separately grounded until the powder at a size of 150 mesh was obtained, and the same weight of the stem powder and the leaf powder were mixed to obtain the patchouli powder. Next, the patchouli powder was pelleted to obtain thin loose pellets. The thin loose pellets have a bulk density of 0.549 (g/mL). Then, 1.739 kg of the thin loose pellets were extracted by using a supercritical fluid extraction device similar to that of FIG. 1 to obtain the patchouli oil. The extraction conditions are the same as in Example 7.

[0078] [Example 10]

[0079] By using a grinder, the stems and leaves of the patchouli were separately grounded until the powder at a size of 150 mesh was obtained, and the same weight of the stem powder and the leaf powder were mixed to obtain the patchouli powder. Next, the patchouli powder was pelleted to obtain thin pressed pellets. The thin pressed pellets have a bulk density of 0.562

(g/mL). Then, 2.155 kg of the thin pressed pellets were extracted by using a supercritical fluid extraction device similar to that of FIG. 1 to obtain the patchouli oil. The extraction conditions are the same as in Example 7.

[0080] [Example 11]

5 **[0081]** By using a grinder, the leaves of the patchouli were grounded until the leaf powder at a size of 150 mesh was obtained. Next, the leaf powder was pelleted to obtain thin pressed pellets. The thin pressed pellets have a bulk density of 0.578 (g/mL). Then, 1.516 kg of the thin pressed pellets were extracted by using a supercritical fluid extraction device similar to that of FIG. 1 to obtain the patchouli oil. The extraction conditions are the same as in Example 7.

10 **[0082]** Then, the amount of the patchouli alcohol, the moisture, the acidic value and the ester value of the patchouli oil in the patchouli oil collected in Examples 7 to 11 were measured, and the measurement results are shown in Table 3.

[0083] [Table 3]

	Extraction yield (%)	Amount of patchouli alcohol (weight fraction) (%)	Moisture of patchouli oil (%)	Acidic value (mg-KOH/ g-sample)	Ester value
Example 7	3.72	29.2	1.15	35.3	59.3
Example 8	7.73	24.6	1.79	18.3	40.1
Example 9	5.18	31.1	1.69	30.8	51.2
Example 10	4.87	30.7	1.73	28.1	55
Example 11	7.15	31.8	1.55	16.5	30.2

[0084] As can be seen from the results of Table 3, as compared with the pellets prepared by
15 using the stem powder and leave powder in Examples 7, 9 and 10, the pellets in Example 8 and

Example 11 are prepared by using the leaf powder alone, and the extraction yield of the pellets in Example 8 and Example 11 was significantly higher than Examples 7, 9, and 10.

[0085] [Example 12]

[0086] By using a grinder, the stems and leaves of the patchouli were separately grounded, and the same weight of the stem powder and the leaf powder were mixed to obtain the patchouli powder. Next, the patchouli powder was pelleted to obtain pellets having a diameter of 5.3 mm and a thickness of 2.7 mm. Then, the patchouli pellets were extracted by using a supercritical fluid extraction device similar to that of FIG. 1 to obtain the patchouli oil. The extraction conditions are: extraction pressure is 150 bar, extraction temperature is 50°C, flow rate of carbon dioxide is 15 kg/hour, and the patchouli extract is collected every 20 minutes.

[0087] [Comparative Example 1]

[0088] By using a grinder, the stems and leaves of the patchouli were separately grounded, and the same weight of the stem powder and the leaf powder were mixed to obtain the patchouli powder. Next, the patchouli powder was pelleted to obtain pellets having a diameter of 5.3 mm and a thickness of 4.7 mm. Then, the patchouli pellets were extracted by using a supercritical fluid extraction device similar to that of FIG. 1 to obtain the patchouli oil. The extraction conditions are: extraction pressure is 150 bar, extraction temperature is 50°C, flow rate of carbon dioxide is 15 kg/hour, and the patchouli extract is collected every 20 minutes.

[0089] [Example 13]

[0090] By using a grinder, the leaves of the patchouli were grounded to obtain the patchouli powder. Next, the patchouli powder was pelleted to obtain pellets having a diameter of 5.3 mm and a thickness of 2.7 mm. Then, the patchouli pellets were extracted by using a supercritical fluid extraction device similar to that of FIG. 1 to obtain the patchouli oil. The patchouli extract is collected every 20 minutes.

[0091] [Comparative Example 2]

[0092] By using a grinder, the stems and leaves of the patchouli were separately grounded until the powder having a size of 140 mesh was obtained, and the same weight of the stem powder and the leaf powder were mixed to obtain the patchouli powder. Then, the patchouli powder is
5 extracted by using a supercritical fluid extraction device similar to that of FIG. 1 to obtain the patchouli oil. The extraction conditions are: extraction pressure is 150 bar, extraction temperature is 50°C, flow rate of carbon dioxide is 15 kg/hour, and the patchouli extract is collected every 20 minutes.

[0093] FIG. 3 shows the normalized extraction curves of Example 12, Example 13,
10 Comparative Example 1 and Comparative Example 2. As can be seen from FIG. 3, the initial slope of the normalized extraction curve of Example 12 (i.e., thin pellets) is greater than the initial slope of the normalized extraction curve of Comparative Example 1 (i.e., thick pellets), which means that the oil extraction of the thinner pellets is much faster than the oil extraction of the thick pellets, so the thin pellets have lower solvent to feed ratio as compared with the thick
15 pellets under the same extraction efficiency. This is because the thinner pellets reduce the diffusion distance and thus the mass transfer rate in the flow phase is increased, thus reducing the solvent to feed ratio. In production facility, since the circulation rate of carbon dioxide is fixed under normal circumstances, the solvent to feed ratio is related to the extraction time of the material. Therefore, the lower the solvent to feed ratio, the shorter the extraction time and the
20 less electricity consumed for the circulation of carbon dioxide.

[0094] Further, as can also be seen from FIG. 3, the initial slope of the normalized extraction curve of Comparative Example 2 (i.e., patchouli powder) is smaller than the initial slope of the normalized extraction curve of Example 12 and Example 13 (i.e., pelleted pellets). This is because mass transfer resistance in supercritical fluids for packed beds with pellets can be

significantly reduced due to increased flow rates. In addition, for packed beds with abrasive powder, short paths called channels might occur.

[0095] Furthermore, it can be seen from FIG. 3 that the initial slope of the normalized extraction curve of Example 13 (pellets made of leaf powder only) is slightly lower than that of Example 12 (pellets made of leaf powder and stem powder). This is because the pellets made of stem contain more fibers that help the carbon dioxide permeate into the solid matrix.

[0096] Experimental Example 3: Determination of Extracts of Different Fractions

[0097] [Example 14]

[0098] By using a grinder, the stems and leaves of the patchouli were separately grounded, and the same weight of the stem powder and the leaf powder were mixed to obtain the patchouli powder. Next, the patchouli powder was pelleted to obtain patchouli pellets having a diameter of 5.3 mm and a thickness of 2.7 mm. Then, a supercritical fluid extraction device similar to that of FIG. 1 was used to extract the patchouli pellets. The extraction conditions are: extraction pressure is 150 bar, extraction temperature is 50°C, flow rate of carbon dioxide is 15 kg/hour. The extract (referred to as the first patchouli extract) with an extraction time of 0.5 hour was collected, the extract (referred to as the second patchouli extract) with an extraction time of 0.5 hour to 1 hour was collected, and the extract (referred to as the third patchouli extract) with an extraction time of 1 hour ~1.5 hours was collected. In this embodiment, the first patchouli extract is an oily extract, and the oily extract maintains oiliness at room temperature.

The second patchouli extract and the third patchouli extract are paste extracts.

[0099] The first patchouli extract was further centrifuged at a speed of not less than 2000 rpm by using a centrifuge to separate the patchouli oil from the patchouli wax in the first patchouli extract. About 68.7 wt% of patchouli oil can be obtained from the oily extract, and the patchouli oil contains about 30.7 wt% of patchouli alcohol. The amount of the patchouli

alcohol in the patchouli wax is about 23.4 wt%, and the melting point of the patchouli wax is higher than 80°C.

[00100] The second patchouli extract was heated and the patchouli oil was collected at different temperatures (50°C, 60°C, 70°C, 80°C, above 80°C), that is, the patchouli oil of different melting points was collected. Table 4 lists the weight fraction of patchouli oil of different melting points in the second patchouli extract and the amount of patchouli alcohol in the patchouli oil.

[00101] [Table 4]

melting point (°C)	Weight (gram) of patchouli paste	Weight fraction (%) of patchouli oil	Amount (weight fraction)(%) of patchouli alcohol
50	11.76	0.753	29.46
60	1.22	0.078	26.00
70	0.95	0.061	28.31
80	0.77	0.049	15.95
Above 80	0.69	0.044	33.20

[00102] As can be seen from Table 4, about 75.3 wt% of patchouli oil can be melted from the second patchouli extract (patchouli paste) at 50°C, and the patchouli oil has 29.5 wt% of patchouli alcohol. On average, the amount of patchouli alcohol in the patchouli paste collected within 0.5 hour to 1 hour was 28.6 wt%.

[00103] The third patchouli extract was heated and the patchouli oil was collected at different temperatures (50°C, 60°C, 70°C, 80°C, above 80°C), that is, the patchouli oil of different melting points was collected. Table 5 lists the weight fraction of patchouli oil of different melting points in the third patchouli extract and the amount of patchouli alcohol in the patchouli

oil.

[00104] [Table 5]

melting point (°C)	Weight (gram) of patchouli paste	Weight fraction (%) of patchouli oil	Amount (weight fraction)(%) of patchouli alcohol
50	9.38	0.937	11.59
60	0.342	0.034	12.18
70	0.0696	0.007	11.27
80	0.0399	0.004	9.81
Above 80	0.141	0.014	10.43

[00105] As can be seen from Table 5, about 93.7 wt% of patchouli oil can be melted from the third patchouli extract (patchouli paste) at 50°C, and the patchouli oil has 11.59 wt% of patchouli alcohol. On average, the amount of patchouli alcohol in the patchouli paste collected within 1 hour to 1.5 hours was 11.6 wt%.

[00106] The amount of patchouli alcohol in the patchouli extract decreases with extraction time. Most of the patchouli alcohol in the pellets will be extracted in the first hour. If the extraction is interrupted after collecting the patchouli extract with an extraction time of 0.5 hour and the patchouli extract with an extraction time of 0.5 hour to 1 hour, three different products can be obtained: (1) patchouli oil containing 30.7 wt% of patchouli alcohol; (2) patchouli wax containing 23.4 wt% of patchouli alcohol; (3) patchouli paste containing 28.6 wt% of patchouli alcohol.

[00107] In summary, the method for preparing the patchouli extract of the present disclosure has simple operation steps, good stability, and uses only non-toxic carbon dioxide during the entire purification process, and has the advantages of realizing automation, low consumption of solvent,

being environment, high production rate and so on as compared with conventional preparation methods. Further, the method for preparing a patchouli extract of the present disclosure extracts a patchouli extract having patchouli alcohol from the patchouli pellets by using supercritical carbon dioxide, which not only can effectively improve the extraction efficiency, but also can
5 obtain patchouli oil with high-purity patchouli alcohol.

[00108] Although the present disclosure has been disclosed in the above embodiments, it is not intended to limit the present disclosure, and those skilled in the art can make some modifications and refinements without departing from the spirit and scope of the disclosure. Therefore, the scope to be protected by the present disclosure is subject to the scope defined by the appended
10 claims.

CLAIMS

WHAT IS CLAIMED IS:

1. A method of preparing a patchouli extract, comprising:
grounding a patchouli material into patchouli powder and pelleting to obtain patchouli
5 granules;
introducing liquid carbon dioxide to perform a supercritical carbon dioxide extraction
process on the patchouli granules, wherein an extraction pressure is 110 bar to 180 bar and an
extraction temperature is 40°C to 60°C;
during the supercritical carbon dioxide extraction process, an extracted product obtained,
10 based on a total weight of the introduced liquid carbon dioxide not exceeding 10 times the
weight of the patchouli granules, is collected as a first patchouli extract, wherein the first
patchouli extract comprises first patchouli oil; and
during the supercritical carbon dioxide extraction process, an extracted product obtained,
based on the total weight of the introduced liquid carbon dioxide exceeding 10 times the weight
15 of the patchouli granules but not exceeding 15 times the weight of the patchouli granules, is
collected as a second patchouli extract, wherein the second patchouli extract comprises second
patchouli oil.
2. The method of claim 1, further comprising drying the patchouli material such that the
dried patchouli material has a moisture content of 15% or less.
- 20 3. The method of claim 1, wherein the patchouli material comprises a patchouli leaf, a
patchouli stem, or a combination thereof.
4. The method of claim 1, wherein the patchouli powder has a size of 140 mesh or less.
5. The method of claim 1, wherein the patchouli granules have a diameter of 2 mm to

10 mm, and the patchouli granules have a thickness of 2.0 mm to 4.5 mm.

6. The method of claim 1, wherein the extraction pressure is 120 bar to 150 bar.

7. The method of claim 1, wherein the first patchouli oil has a melting point of 10°C to 40°C, and the second patchouli oil has a melting point of 40°C to 70°C.

5 8. The method of claim 1, wherein an amount of patchouli alcohol in the first patchouli oil is 10 wt% to 40 wt%, and an amount of patchouli alcohol in the second patchouli oil is 10 wt% to 30 wt%.

9. The method of claim 1, further comprising centrifuging the first patchouli extract to separate the first patchouli oil from patchouli wax in the first patchouli extract.

10 10. The method of claim 9, wherein an amount of patchouli alcohol in the patchouli wax is 10 wt% to 30 wt%.

11. A method of preparing a patchouli extract, comprising:

grounding a patchouli material into patchouli powder and pelleting to obtain patchouli granules;

15 introducing liquid carbon dioxide to perform a supercritical carbon dioxide extraction process on the patchouli granules, wherein an extraction pressure is 110 bar to 180 bar and an extraction temperature is 40°C to 60°C, a flow rate of the liquid carbon dioxide is 15 kg/hour;

during the supercritical carbon dioxide extraction process, an extracted product obtained within an extraction time not exceeding 0.5 hour is collected as a first patchouli extract, wherein

20 the first patchouli extract comprises first patchouli oil; and

during the supercritical carbon dioxide extraction process, an extracted product obtained within an extraction time exceeding 0.5 hour but not exceeding 1 hour is collected as a second patchouli extract, wherein the second patchouli extract comprises second patchouli oil.

12. The method of claim 11, further comprising drying the patchouli material such that the dried patchouli material has a moisture content of 15% or less.

13. The method of claim 11, wherein the patchouli material comprises a patchouli leaf, a patchouli stem, or a combination thereof.

5 14. The method of claim 11, wherein the patchouli powder has a size of 140 mesh or less.

15. The method of claim 11, wherein the patchouli granules have a diameter of 2 mm to 10 mm, and the patchouli granules have a thickness of 2.0 mm to 4.5 mm.

16. The method of claim 11, wherein the extraction pressure is 120 bar to 150 bar.

10 17. The method of claim 11, wherein the first patchouli oil has a melting point of 10°C to 40°C, and the second patchouli oil has a melting point of 40°C to 70°C.

18. The method of claim 11, wherein an amount of patchouli alcohol in the first patchouli oil is 10 wt% to 40 wt%, and an amount of patchouli alcohol in the second patchouli oil is 10 wt% to 30 wt%.

15 19. The method of claim 11, further comprising centrifuging the first patchouli extract to separate the first patchouli oil from patchouli wax in the first patchouli extract.

20. The method of claim 19, wherein an amount of patchouli alcohol in the patchouli wax is 10 wt% to 30 wt%.