

METHOD AND APPARATUS FOR PREPARING A BOTANICAL EXTRACT

Cross-Reference To Related Application

5 [0001] This application claims the benefit of priority of Singapore patent application No. 10202204097S, filed 20 April 2022, the content of it being hereby incorporated by reference in its entirety for all purposes.

Technical Field

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[0002] Various embodiments relate to a method and an apparatus for preparing a botanical extract, more specifically an essential oil.

Background

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[0003] Botanical extracts are concentrated plant material originating from various parts of plants such as petals, flowers, leaves, roots, and stems that contain potent bioactive compounds. To obtain botanical extracts, extraction being a process of removing one or several desired plant compounds from the actual plant for further analysis and/or processing is performed.

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[0004] For example, patchouli (*pogostemon cablin*) is an aromatic species of the Lamiaceae, native to Southeast Asia that contains essential oil that is of great economic value. Patchouli oil is largely used in perfumery and aromatherapy due to its aromatic spicy fragrance. The essential oil also has numerous therapeutic properties and traditionally obtained by steam distillation of dry patchouli leaves. A typical steam distillation process involves high temperature and long extraction hours (upto 10 hours) to obtain 1-3% yield of essential oil. Other methods to extract the essential oils may be hydro-distillation and solvent extraction which have drawbacks such as low yields, requiring use of chemicals and solvents, and being not environmentally friendly. A better extraction method is desired to recover higher amount of essential oil within shorter duration, a lower temperature and without the need of a solvent.

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[0005] Extraction of essential oils with supercritical fluid extraction (SCFE) with carbon dioxide (CO₂) is a relatively new technology and not yet fully developed. SCFE with CO₂ may be performed at its critical temperature of 304 K and critical pressure of 7.39 MPa to produce solvent-free extracts. However, if extractions are performed just at critical point
5 (i.e. at low pressure, temperature and CO₂ flow rates), the yields of essential oil are found to be relatively low with a lower amount of patchouli alcohol. To maximize the recovery of essential oil and patchouli alcohol, the extractions are performed at a higher pressure for longer durations. CO₂ extracts prepared at high pressures may include high free fatty acids (FFA), pigments, wax/gum and other impurities rendering the extracts not
10 comparable to market ready essential oils derived from other conventional methods like steam distillation. In other words, the SCFE with CO₂ may result into an undesirable viscous extract requiring manufacturers to perform a wide range of time consuming, costly and labour-intensive post processing steps (such as centrifugation, winterization, filtration, de-acidification and de-colourization) to convert them into market ready high
15 quality essential oils.

[0006] Whilst a prior publication describes the customization of patchouli alcohol during SCFE process, without mention or guidance about any post processing of extract to remove the impurities such as pigments, wax, gum, water. In the prior publication, no attempts were made to post process the viscous SCFE extract to convert into market
20 ready clear oils.

[0007] Thus, there is a need for a technology capable of addressing at least the problems mentioned above to generate high yield and high-quality essential oils and its related products without compromising the yield with CO₂ extraction.

25 Summary

[0008] According to an embodiment, a method for preparing a botanical extract is provided. The method may include subjecting a botanical material to an extract pressure under influence of liquid carbon dioxide to perform a supercritical carbon dioxide
30 extraction process to obtain an extraction mixture; separating an intermediate extract from the extraction mixture; and substantially immediately following the step of

separating, feeding the intermediate extract to a series of stages to perform molecular distillation to obtain the botanical extract with an improved level of one or more parameters as compared to the intermediate extract. The one or more parameters may include at least one of density, acid value, refractive index, appearance, or miscibility.

5 Each stage may be set to a predetermined distillation temperature, a predetermined vacuum pressure and a predetermined rotation speed.

[0009] According to an embodiment, an apparatus for preparing a botanical extract is provided. The apparatus may include a supercritical carbon dioxide extraction module configured to apply an extract pressure under influence of liquid carbon dioxide to a botanical material to perform a supercritical carbon dioxide extraction process to obtain an extraction mixture; a separation module in fluidic communication with the supercritical carbon dioxide extraction module, the separation module configured to separate an intermediate extract from the extraction mixture; and a molecular distillation module in fluidic communication with the separation module. The molecular distillation

10 module may include a series of stages configured to perform molecular distillation to obtain the botanical extract with an improved level of one or more parameters as compared to the intermediate extract; and one or more pipes, each coupled between one stage of the series of stages and a subsequent stage of the series of stages. The one or more parameters may include at least one of density, acid value, refractive index, appearance, or miscibility. Each stage may be operable with a predetermined distillation

15 temperature, a predetermined vacuum pressure and a predetermined rotation speed.

Brief Description of the Drawings

25 [0010] In the drawings, like reference characters generally refer to like parts throughout the different views. The drawings are not necessarily to scale, emphasis instead generally being placed upon illustrating the principles of the invention. In the following description, various embodiments of the invention are described with reference to the following drawings, in which:

30 [0011] FIG. 1A shows a flow chart illustrating a method for preparing a botanical extract, according to various embodiments.

[0012] FIG. 1B shows a schematic view of an apparatus for preparing a botanical extract, according to various embodiments.

[0013] FIG. 2 shows a flow chart involving a method of post processing SCFE extracts, according to various examples.

5 [0014] FIG. 3A shows a photograph depicting an extract obtained from SCFE using patchouli, according to one example.

[0015] FIG. 3B shows a photograph depicting a Stage 2 sample including low patchouli alcohol (PA) oil, a Stage 3 sample including high PA oil and oil containing mainly pigments, iron, wax/gum/fatty acids, according to one example.

10 [0016] FIG. 4 shows a photograph depicting a Stage 2 sample, and a Stage 3 sample - both being clear oil and another part of the Stage 3 sample being nutmeg butter, according to one example.

[0017] FIG. 5 shows a photograph depicting clove buds extract obtained from SCFE, a Stage 2 sample, and a Stage 3 sample and another part of the Stage 3 sample, according to one example.

15 [0018] FIG. 6A shows a photograph depicting an extract obtained from SCFE using ginger, according to one example.

[0019] FIG. 6B shows a photograph depicting a Stage 2 sample, a Stage 3 sample and another part of the Stage 3 sample based on the extract of FIG. 6A, according to one example.

Detailed Description

25 [0020] The following detailed description refers to the accompanying drawings that show, by way of illustration, specific details and embodiments in which the invention may be practiced. These embodiments are described in sufficient detail to enable those skilled in the art to practice the invention. Other embodiments may be utilized and structural, logical, and electrical changes may be made without departing from the scope of the invention. The various embodiments are not necessarily mutually exclusive, as
30 some embodiments can be combined with one or more other embodiments to form new embodiments.

[0021] Embodiments described in the context of one of the methods or devices are analogously valid for the other methods or devices. Similarly, embodiments described in the context of a method are analogously valid for a device, and vice versa.

[0022] Features that are described in the context of an embodiment may correspondingly be applicable to the same or similar features in the other embodiments. Features that are described in the context of an embodiment may correspondingly be applicable to the other embodiments, even if not explicitly described in these other embodiments. Furthermore, additions and/or combinations and/or alternatives as described for a feature in the context of an embodiment may correspondingly be applicable to the same or similar feature in the other embodiments.

[0023] In the context of various embodiments, the articles “a”, “an” and “the” as used with regard to a feature or element include a reference to one or more of the features or elements.

[0024] In the context of various embodiments, the phrase “substantially” may include “exactly” and a reasonable variance.

[0025] In the context of various embodiments, the term “about” as applied to a numeric value encompasses the exact value and a reasonable variance.

[0026] As used herein, the term “and/or” includes any and all combinations of one or more of the associated listed items.

[0027] As used herein, the phrase of the form of “at least one of A or B” may include A or B or both A and B. Correspondingly, the phrase of the form of “at least one of A or B or C”, or including further listed items, may include any and all combinations of one or more of the associated listed items.

[0028] As used herein, the expression “configured to” may mean “constructed to” or “arranged to”.

[0029] Various embodiments may provide for preparation of essential oils and related products thereof.

[0030] FIG. 1A shows a flow chart illustrating a method 100 for preparing a botanical extract, according to various embodiments. In Step 102, a botanical material may be subject to an extract pressure under influence of liquid carbon dioxide to perform a supercritical carbon dioxide extraction process to obtain an extraction mixture. In Step

104, an intermediate extract may be separated from the extraction mixture. In Step 106, substantially immediately following the step of separating, the intermediate extract may be fed to a series of stages to perform molecular distillation to obtain the botanical extract with an improved level of one or more parameters as compared to the intermediate
5 extract. The one or more parameters may include at least one of density, acid value, refractive index, appearance, or miscibility. Each stage may be set to a predetermined distillation temperature, a predetermined vacuum pressure and a predetermined rotation speed.

[0031] In other words, the technology involves a one-step post processing method of CO₂
10 extracts to generate market ready essential oils and its related products after CO₂ extraction. In this method, essential oils and related products may be produced by supercritical fluid extraction (SCFE) of botanicals followed by post processing with molecular distillation technology. Molecular distillation refers to a distillation method operated under high vacuum. Here, the expressions SCFE, SCFE with CO₂, and CO₂
15 extraction may mean supercritical carbon dioxide extraction.

[0032] The proposed solution may be easily implemented by integrating existing SCFE technology with continual molecular distillation post processing. Here, one-step refers to SCFE coupled with molecular distillation. It may also mean a continuous method where SCFE along with separation, and molecular distillation post processing are not performed
20 separately as different batches, thereby providing the molecular distillation post processing substantially immediately following the SCFE along with separation. It should also be appreciated that in a different embodiment, the molecular distillation may be performed in a batch mode of post processing after SCFE.

[0033] The technology is capable of generating high quality essential oils and their
25 derivatives without compromising the yield in a relatively shorter extraction time. Various embodiments may provide a solution to essential oil manufacturers who are struggling to find a way to convert viscous SCFE extracts into market ready clear oils with customizable properties such as functional compounds, acid value, color, viscosity, density and odour.

[0034] In various embodiments, prior to the step 102 of subjecting the botanical material
30 to the extract pressure under influence of liquid carbon dioxide, the method 100 may

further include grounding at least part of a plant into a powder and pelleting the powder to obtain the botanical material in granular form. For example, the powder may have a size of about 140 mesh or less, and the botanical material in granular form may have an average diameter of about 2 mm to about 10 mm, and an average thickness of about 2 mm to about 4.5 mm. In a different example, the botanical material may be in a form of leaves from a plant that do not undergo powdering or pelletizing.

[0035] Prior to grounding the at least part of the plant, the method 100 may further include drying the at least part of the plant to reduce moisture content to 15% or less.

[0036] In various embodiments, the extract pressure may be about 100 bar to about 500 bar. The supercritical carbon dioxide extraction process may be performed under an extract temperature ranging from 40 °C to 60 °C. The liquid carbon dioxide may be provided at a flow rate ranging from 5 kg/h to 30 kg/h for about 10 minutes to about 3 hours.

[0037] In various embodiments, the step 104 of separating may be performed under a depressurized condition of about 40 bar.

[0038] In various embodiments, the step 106 of feeding the intermediate extract may further include heating the intermediate extract at about a temperature ranging from 40 °C to 60 °C into molten form. More specifically, the intermediate extract may be heated to a suitable temperature to facilitate flow and feeding of the intermediate extract. It should be appreciated that this heating step may be performed prior to exposure to the predetermined distillation temperature, the predetermined vacuum pressure, and the predetermined rotation speed at an initial stage of the series of stages.

[0039] The predetermined distillation temperature may be ranging from 30 °C to 110 °C. the predetermined vacuum pressure may vary from 0.2 mbar to 1 mbar. The predetermined rotational speed may be ranging from 100 rpm to 140 rpm.

[0040] In various embodiments, feeding the intermediate extract at Step 106 may include feeding the intermediate extract at a feed rate of more than 1 l/h. For example, when performing the method 100 under laboratory trial conditions, the feed rate may be in a range from 1 l/h to 5 l/h. In one specific example, feeding the intermediate extract at Step 106 may be at a feed rate of about 3 l/h for 20 minutes. On the other hand, in full scale manufacturing, the feed rate may be further increased even to more than 20 l/h.

[0041] In various embodiments, a first stage of the series of stages may be set to the predetermined distillation temperature of about 60 °C and the predetermined rotational speed of about 100 rpm; a second stage of the series of stages may be set to the predetermined distillation temperature of about 90 °C and the predetermined rotational speed of about 140 rpm; and a third stage of the series of stages may be set to the predetermined distillation temperature of about 110 °C and the predetermined rotational speed of about 140 rpm.

[0042] The first stage may precede the second stage, and the second stage may precede the third stage. In this series of stages, the first stage may be the initial stage, while the third stage may be the final stage, or an intermediary stage where subsequent stage(s) may follow to perform further refining and obtain the final botanical extract.

[0043] The at least part of the plant may include patchouli leaves, clove buds, nutmeg, ginger, cinnamon, cassia, black pepper or a plant material containing essential oils. The botanical extract may have an alcohol content of about 25 % to 50 %. The alcohol content may refer to that of an essential oil or a spice oil. Alternatively or additionally, the botanical extract may have one or more functional compounds, e.g. terpenes, eugenol, zingiberene, gingerol, myristicin, amongst others. For example, not all plant essential oils have alcoholic compounds to provide an alcohol content, and for such plant essential oils, the functional compounds may be referred to instead. In other words, the method may be used to process a wide variety of botanicals other than patchouli (pogostemon cablin) oil and its related products. For example, the botanicals or botanical extract may include essential oils, spice oils such as clove buds, nutmeg, ginger, cinnamon, cassia, black pepper, or any other plant materials containing essential oils.

[0044] While the method described above is illustrated and described as a series of steps or events, it will be appreciated that any ordering of such steps or events are not to be interpreted in a limiting sense. For example, some steps may occur in different orders and/or concurrently with other steps or events apart from those illustrated and/or described herein. In addition, not all illustrated steps may be required to implement one or more aspects or embodiments described herein. Also, one or more of the steps depicted herein may be carried out in one or more separate acts and/or phases.

[0045] FIG. 1B shows a schematic view of an apparatus 120 for preparing a botanical extract, according to various embodiments. The apparatus 120 may include a supercritical carbon dioxide extraction module 122 configured to apply an extract pressure under influence of liquid carbon dioxide to a botanical material to perform a supercritical carbon dioxide extraction process to obtain an extraction mixture; a separation module 124 in fluidic communication with the supercritical carbon dioxide extraction module 122 (as denoted by line 128), the separation module 124 configured to separate an intermediate extract from the extraction mixture; and a molecular distillation module 126 in fluidic communication with the separation module 124 (as denoted by line 130). The molecular distillation module may include a series of stages 131 configured to perform molecular distillation to obtain the botanical extract with an improved level of one or more parameters as compared to the intermediate extract; and one or more pipes 133. Each pipe 133 may be coupled between one stage of the series of stages 131 and a subsequent stage of the series of stages 131. The one or more parameters may include at least one of density, acid value, refractive index, appearance, or miscibility. Each stage 131 may be operable with a predetermined distillation temperature, a predetermined vacuum pressure and a predetermined rotation speed.

[0046] The apparatus 120 may include the same or like elements or components as those of the method 100 of FIG. 1A. For example, the one or more parameters and the series of stages 131 described in context of the apparatus 120 may be similar or the same as those described in the method 100 of FIG. 1A.

[0047] In the context of various embodiments, the term “coupled” may mean attached, or connected. The expression “in fluidic communication” may mean being coupled.

[0048] In various embodiments, the separation module 124 may be further configured to perform separation under a depressurized condition of about 40 bar.

[0049] The apparatus 120 may further include a heating module configured to heat the intermediate extract at about a temperature ranging from 40 °C to 60 °C into molten form. In the molten form, the heated intermediate extract may be easily fed through to the molecular distillation module 126.

[0050] The predetermined distillation temperature may be ranging from 30 °C to 110 °C. The predetermined vacuum pressure may vary from 0.2 mbar to 1 mbar. The predetermined rotational speed is ranging from 100 rpm to 140 rpm.

[0051] In various embodiments, the one or more pipes 133 may be arranged to be heated to about 50 °C. This may be to allow the intermediate extract processed in each stage 131 to be in molten form when moving through the pipe 133 to the subsequent stage 131.

[0052] In various embodiments, the series of stages 131 may include a first stage configured to operate at the predetermined distillation temperature of about 60 °C and the predetermined rotational speed of about 100 rpm; a second stage configured to operate at the predetermined distillation temperature of about 90 °C and the predetermined rotational speed of about 140 rpm; and a third stage configured to operate at the predetermined distillation temperature of about 110 °C and the predetermined rotational speed of about 140 rpm. For example, the first stage may precede the second stage, and the second stage may precede the third stage. In one example, the series of stages 131 may include three stages.

[0053] Various embodiments may provide a botanical extract prepared by performing the method 100 of FIG. 1A. The botanical extract may have an alcohol content of about 25 % to about 50 %, or preferably about 29% to about 31%, or more preferably about 40% to about 50%. The botanical extract may be an essential oil.

[0054] Examples of preparing essential oils and related products thereof based on a method of post processing SCFE extracts to convert viscous SCFE extracts to clear essential oils will be described below.

[0055] FIG. 2 shows a flow chart involving the method 200 of post processing SCFE extracts, according to various examples. The method 200 may include or involve the same or like elements or components as those of the method of FIG. 1A and of the apparatus 120 of FIG. 1B, and as such, the same ending numerals are assigned and the like elements may be as described in the context of the method of FIG. 1A and of the apparatus 120 of FIG. 1B, and therefore the corresponding descriptions may be omitted here.

[0056] As seen in FIG. 2, the method 200 includes the following steps. A plant or botanical material 205 (e.g. patchouli) may be fed into a SCFE extractor 222 (e.g. the

supercritical carbon dioxide extraction module 122 of FIG. 1B), for example in the form of ground powder or pellet or in the form of parts of the plant (e.g. leaves) without powdering or pelletizing, extracted at an optimum pressure, temperature and flow rate of carbon dioxide (e.g. as in Steps 102 and 104 of FIG. 1A) and passed through a molecular distillation system 226 (e.g. the molecular distillation module 126 of FIG. 1B) for further purification. In one example, a SCFE separator 224 (e.g. the separation module 124 of FIG. 1B) may be arranged between the SCFE extractor 222 and the molecular distillation system 226 including various stages 231a, 231b, 231c (e.g. the series of stages 131 of FIG. 1B) coupled to one another via pipes 233 (e.g. the one or more pipes 133 of FIG. 1B). The SCFE extract 207 (e.g. patchouli extract) may be first heated to keep it in molten state and then using a feed pump 235 pumped into the molecular distillation system 226 for the separation of water 209, alcohol, plant oil with low plant alcohol 211, plant oil with high plant alcohol 213 and finally a viscous liquid 215 including all the impurities, waxes and pigments. Taking the specific example of the plant or botanical material 205 to be patchouli, the plant oil 211 with low patchouli alcohol may be of about 30% or less, and for the plant oil 211 with less than 1% patchouli alcohol may be referred to as terpenes. The plant oil 213 with high patchouli alcohol may be of about 40% to about 50%. The viscous liquid 215 may also include plant oil with upto 60% patchouli alcohol, depending on the initial patchouli alcohol percentage in the feedstock.

[0057] In various embodiments, the molecular distillation system 226 may include a molecular evaporator, a degassing system, a feeding system, a heating system, a cooling vacuum system, and a control system. A core part of the molecular distillation device may be the molecular evaporator, which mainly has three types, namely falling film type, scraping film type and centrifugal type.

[0058] Depending on the types of plant materials 205 as well as the parameters used for the various stages 231a, 231b, 231c, the percentage values of plant alcohol may differ from the exemplary values and/or range indicated above. However, it should be understood and appreciated that with the parameters set for one stage (e.g. 231b) being distinct from the parameters set for another stage (e.g. 231c), the plant oil at the stage (e.g. 231b) may have a plant alcohol percentage distinguishable from that of the plant oil

at the other stage (e.g. 231c), thereby allowing a comparison of a low plant alcohol and a high plant alcohol between the stages (e.g. 231b, 231c).

[0059] Preparing essential oils and related products thereof may be carried out based on patchouli using the method 200 of FIG. 2. In this case, the plant alcohol oil may be patchouli alcohol (PA) oil. It should be appreciated that the method 200 may be used for processing a wide variety of botanicals other than for patchouli oil and its related products. Some examples may include spice oils such as clove buds, nutmeg, ginger, (which will be subsequently discussed in more detail for illustrative and comparison purposes) as well as cinnamon, cassia, black pepper and so on.

PATCHOULI

Preparation of Patchouli SCFE Extract

[0060] Patchouli leaves may be obtained from Indonesia and ground to a powder ranging 40 mesh to 200 mesh. The powder may be further pelletized to convert into granules of diameter of about 2 mm to about 10 mm, and a thickness of about 2.0 mm to about 4.5 mm. The pellets were then loaded into the SCFE extractor 222 and extracted with carbon dioxide (CO₂) at 100 bar to 300 bar pressure, 40°C to 60°C temperature and 5kg/h to 30kg/h flow rate of CO₂ for 10 minutes to 3 hours. The supercritical CO₂ may then be depressurized at about 40 bars to separate gaseous CO₂ and the SCFE extract 207 into the SCFE separator 224. The total SCFE extract 207 may be collected at the end of the process (e.g. as in Steps 102 and 104 of FIG. 1A) including essential oils, water, pigments, wax and gum.

[0061] The SCFE extract 207 collected at the end of this process may be sticky, viscous and dark green in colour with a total yield of about 1% to 10%. The SCFE extract 207 may be then heated to a temperature from 40°C to 60°C to melt all the wax gums and homogenized before processing with molecular distillation (e.g. as in Step 106 of FIG. 1A).

Post Processing of SCFE Extract by Molecular Distillation (MD)

[0062] Molecular distillation (MD) of SCFE extract may be performed to remove water, pigments, wax and any other impurities carried over by SCFE and obtain clear oil and

related products. For this, viscous SCFE extract 207 prepared with SCFE extraction via the SCFE extractor 222 and the SCFE separator 224 may go into the feed via the feed pump 235 of the molecular distillation system 226, heated to 40°C to 60°C, passing through a first vessel (e.g. Stage 1 231a) to remove the water 209 and subsequently
5 passing through different stages (e.g. Stage 2 231b and Stage 3 231c) of the molecular distillation system 226 to separate essential oils of different quality (e.g. 211, 213). It should be appreciated that the feed rate, interchangeably referred to as flow rate, may depend on the production scale unit, and may be from 1 l/h or more. In some cases, under large manufacturing scale, the feed rate may even be more than 20 l/hr. In an alternative,
10 lower feed rate from 1 l/h (litre per hour) to 5 l/h may also be possibly used, for example, during laboratory trials.

[0063] The distillation temperature may be controlled from 30°C to 110°C, preferably from 30°C to 70 °C, vacuum may vary from 20 Pa to 100 Pa (meaning that the pressure of the vacuum is not fixed and changes along with experiment, at least in absence of any
15 pressure-changing equipment) with rotation speeds of 100 rpm to 140 rpm. The separation of lighter compounds (patchouli alcohol 0% to 5%) or terpenes may occur first depending on the design of the system (e.g. the apparatus 120 of FIG. 1B) followed by oil with a patchouli oil content of 5% to 40% patchouli alcohol (e.g. 211) and finally the patchouli oil with a high patchouli alcohol content of 40% to 60% (e.g. 213) may be
20 obtained. The last sample after collection of all earlier samples may be the remaining pigments, wax gums, essential oil and impurities (e.g. 215) that come out as a heavy sticky product and may be further purified to obtain pure patchouli alcohol as it may contain significantly high patchouli alcohol content typically in the range of 50% to 70%. The further purification may involve subsequent stage(s) (e.g. beyond Stage 3 231c) of
25 controlled temperatures, vacuum pressures and rotation speeds, not described in here in detail.

[0064] A specific example of preparing essential oils and related products from patchouli, along with experimental data will be presented below.

[0065] Patchouli leaves were ground and filtered with 2 mm sieve mesh and pelletized.
30 The granular pellets (2 kg) were then loaded into SCFE extraction vessel (e.g. the SCFE extractor 222) and extracted at 300 bar pressure, 50 °C temperature and 15 kg/h flow rate

of CO₂ for 2 hours. The total SCFE extract 207 was collected at the end of the process including any water, pigments, wax and gum and impurities. A total of 20 kg material was extracted using SCFE and 1.4 kg extract (total yield 7%) was obtained. The extract 207 was heated at 50 °C and homogenized before processing with molecular distillation.

5 A sample of 30 g was taken to check the patchouli alcohol (PA) percentage and acid value of the extract. Water was removed from the extract by a peristaltic pump and collected in a separate bottle. Extracts 207 were kept in molten state all the time by heating the bottle at 60 °C using a hot plate stirrer during post processing with molecular distillation. 1 kg of homogenized extract was used as feed for post processing with the
10 molecular distillation system 226.

[0066] For molecular distillation, the homogenized SCFE extract (1 kg) was fed into Stage 1 231a at a flow rate of 3 l/h in about 20 minutes using a peristaltic pump. Each of Stage 1 231a and Stage 2 231b rotation speed was 100 rpm and Stage 3 231c was 140 rpm. The temperature of pipes 233 was 50 °C. In the molecular distillation system 226,
15 Stage 1 231a was designed to separate any solvent or water 209, Stage 2 231b was to separate low PA oil 211, while Stage 3 231c was to separate high PA oil 213 and oil with impurities such as pigments, wax, gum and so on 215. The temperature of Stage 1 231a was 60 °C, Stage 2 231b was at 90 °C and Stage 3 231c was at 110 °C. At the end of the process, 4 samples were collected at Stage 1 231a, Stage 2 231b, and Stage 3 231c. Stage
20 1 231a sample included traces of water 209, Stage 2 231b sample included low PA% oil (29-31%) 211, and Stage 3 231c sample included high PA oil (40-50%) 213 and separately, oil containing mainly pigments, iron, wax/gum/fatty acids 215.

[0067] FIG. 3A shows a photograph 301 depicting the extract 307 (e.g. the SCFE 207 of FIG. 2) obtained from SCFE, according to one example. FIG. 3B shows a photograph
25 303 depicting the Stage 2 231b sample including low PA oil 311, the Stage 3 sample including high PA oil 313 and oil containing mainly pigments, iron, wax/gum/fatty acids 315. The contents 311, 313, 315 of the samples may be described in similar context to 211, 213, 215 of FIG. 2.

[0068] The samples obtained from SCFE extraction and from different stages of
30 molecular distillation were characterized for colour, appearance (compared as seen in FIGS. 3A and 3B), PA%, refractive index, acid value, density, miscibility in ethanol

according to Indonesian National Standard (SNI) and results are presented in Table 1 below. Other characteristics of the samples may also be assessed but they are not presented here.

[0069] Table 1 - Comparison of properties of SCFE extract and clear oils post processed by molecular distillation process

Properties	Patchouli SCFE Extract 307	Molecular Distilled Stage 2 Sample 311	Molecular Distilled Stage 3 Sample (in one part) 313	Molecular Distilled Stage 3 Sample (in other part) 315
Acid Value	11.24	2.20	9.94	-
Patchouli Alcohol %	44.11	32.74	52.7	54.37
Copaene %	0.51	0.74	0.29	-
Colour	Dark Brown	Light Yellow	Yellow	Dark Green
Appearance	Viscous	Clear oil	Clear oil	Viscous
Refractive Index	-	1.508	1.508	10.47
Density	-	0.961	0.980	-
Miscibility in 90% Ethanol	Fail	Pass	Pass	Fail

[0070] SCFE extract 307 failed most of the tests performed on the samples, whereas the samples obtained after molecular distillation 311, 313 passed all test and showed superior results in terms of density, acid value, refractive index, appearance, miscibility in ethanol and patchouli alcohol.

[0071] After post processing with molecular distillation process weights of all samples at different stages 231a, 231b, 231c were measured and recoveries were calculated accordingly as given in Table 2 below.

[0072] Table 2 - Results for samples obtained from molecular distillation processing of SCFE extract based on one experiment

Experiment No.	MDT41
Feed	Patchouli SCFE Extract
SCFE total Yield %	7.00
Feed weight (kg)	1
Temperature (°C)	60 (Stage 1 231a)

	90 (Stage 2 231b) 110 (Stage 3 231c)	
	Patchouli Alcohol %	Yield %
Water %	-	0.63
Molecular Distilled Stage 2 Sample 311	32.74	24.39
Molecular Distilled Stage 3 Sample (in one part) 313	52.70	31.97
Molecular Distilled Stage 3 Sample (in other part) 315	54.37	40.04
Recovery of Clear Oil (Sample 311+Sample 313)	-	56.36
Clear Oil % = (crude extract%-water%) * recovery rate	(7.0-0.63) *0.564= 3.59	

[0073] The same plant material gave a yield of 2% with steam distillation extracted for 8 hours using a lab scale steam distillation system. The yield of SCFE extract from same plant material was 7% and after post processing with molecular distillation system a total of 3.5% essential oil was recovered. The other sample 315 of viscous liquid was recovered from Stage 3 (in other part) 231c with oil containing mainly pigments, iron, wax/gum/fatty acids that may be further post processed to obtain absolute with a significantly high patchouli alcohol. The further post processing (not described in detail here) may enable conversion to other high-quality products such as oleoresin, absolute, concrete, patchouli alcohol crystal powder with a purity of 99% or above.

[0074] Currently, using existing methods, absolute is prepared by solvent extraction of plant material without going through SCFE or MD processes. Here, the by-product (e.g. the other sample 315) of the combined SCFE and MD process (e.g. as in the method 100, 200 of FIGS. 1A and 2) may be used to convert to absolute. For example, an ethanol post processing of the by-product (e.g. 315) may be carried out. The ethanol post processing may include steps of dissolving the by-product (e.g. 315), or interchangeably referred to as oil components, in ethanol in 1:10 ratio, precipitating waxes by winterization at reduced temperatures, separating waxes by filtration and subsequently removing ethanol by vacuum evaporation to recover absolute. In brief, related products of patchouli oil may include terpenes (low PA of less than 30%), normal PA oil (of about 30%), high PA (50-70%), patchouli water (hydrosol) and other related products which may be derived from

further processing such as oleoresin, absolute, concrete, patchouli crystals (more than or equal to 99% PA), patchouli wax and so on.

[0075] The proposed combined technology (that is, continual post processing of SCFE extract using molecular distillation as in the method 100, 200 of FIGS. 1A and 2) clearly demonstrated several advantages over steam distillation in terms of yield, quality of oil, patchouli alcohol content. The essential oil may also be fractionated into different quality of oils and with simple blending being converted to various market ready products to fulfil specific requirements of customers. PA may still be customized during molecular distillation processing (e.g. in Step 106). In other words, during molecular distillation, the PA% in each stage may be changed by changing the parameter in that particular stage. For example, a higher temperature may evaporate more PA in that stage leading to lesser PA going to next stage and vice versa. In case the PA of essential oils coming out of molecular distillation stages do not exactly meet the requirements of a customer, a manual blending of low and high PA oil may be done to obtain a desired PA%.

[0076] More specifically, as SCFE utilizes high pressure, sticky extracts are often produced, and researchers typically reduce to minimum pressure near CO₂ critical point (> 72 bar) to extract clearer wax free oils. Meanwhile, MD is normally used to increase the PA% of low PA (<30%) steam distilled oils to more than 30% as this is a main criteria to determine the price of the essential oil in the market. In the proposed technology, e.g. as described in the method 100, 200 of FIGS. 1A and 2, the MD module (e.g. 126) may be customized to handle sticky SCFE extracts with more steps being included so that MD process (as described in Step 126 of FIG. 1A) is capable to handle the sticky SCFE extract (e.g. 207 of FIG. 2). This may include a setting tank for the separation of water from the SCFE extract, a heater for increasing the temperature (40-60 °C) of the SCFE extract to keep it in molten and free flowing form, and an evaporator to remove or at least minimize any traces of ethanol present inside the SCFE extract due to washing of the equipments.

NUTMEG

[0077] A specific example of preparing essential oils and related products from nutmeg, along with experimental data will be presented below.

[0078] Nutmeg fruits (10 kg) were powdered, filtered using 1-mm sieve and extracted with SCFE at 300 bars, 60 °C and 15 kg/hr flow rate for about 2 hours. A total of 2.5 kg powder was loaded for each run and every run produced about 25% of crude extract. Total 2.5 kg crude extract was prepared homogenized by heating over a water bath at 60 °C and re-packed into 1L bottles. (Table 3)

[0079] For MD post processing, a simulated nutmeg extract was prepared by mixing commercial steam distilled nutmeg oil with nutmeg butter separated from crude extract in a ratio of 60% oil and 40% butter. The combined extract was heated to 60 °C in a hot water bath and processed through molecular distillation. Parameters chosen for MD processing of the extract were as follow: the temperature of Stage 1 231a was 60 °C, Stage 2 231b was at 60 °C and Stage 3 231c was at 110 °C. Flow rate and other parameters were same as selected for treatment of Patchouli crude extract, as described earlier. Reference may be made to Table 6 below.

[0080] FIG. 4 shows a photograph 403 depicting a Stage 2 sample 411, and a Stage 3 sample 413 - both being clear oil and another part of the Stage 3 sample 415 being nutmeg butter, according to one example. The contents 411, 413, 415 of the samples may be described in similar context to 211, 213, 215 of FIG. 2.

[0081] Table 3 shows the characteristics of nutmeg SCFE extract, and samples processed with different stages.

Experiment No.	Nutmeg Extract (MDT59)			
Parameters	Crude Extract	Molecular Distilled Stage 2 Sample 411	Molecular Distilled Stage 3 Sample (in one part) 413	Molecular Distilled Stage 3 Sample (in other part) 415
Color	Dark Yellow	Colorless	Light Yellow	Yellow brown
Wax	Present	Not Present	Not Present	Present
Myristicin (>10%)	23.58	22.10	43.25	31.17
Refractive Index (1.470-1.497)	1.427	1.486	1.508	1.470
Density (0.880- 0.910)	0.973	0.844	0.890	0.937

Miscibility in 90% Ethanol	Fail	Pass	Pass	Fail
SNI Standard	Fail	Pass	Pass	Fail

CLOVE BUDS

[0082] A specific example of preparing essential oils and related products from clove buds, along with experimental data will be presented below.

- 5 **[0083]** Clove buds (30 kg) were powdered, filtered with 2-mm sieve and extracted with SCFE at 300 bar, 50 °C and 15 kg/hr flow rate for 2 hours. Every run produced around 19-20% of total extract by loading 2.7 kg powder. Total 5 kg extract was prepared and homogenized by heating over a water bath at 60 °C and re-packed into 1 L bottles for post processing with MD.
- 10 **[0084]** The combined extract was heated to 60 °C in a hot water bath and processed through molecular distillation. Parameters chosen for MD processing of the extract were as follow: the temperature of Stage 1 231a was 60 °C, Stage 2 231b was at 100 °C and Stage 3 231c was at 110 °C. Flow rate and other parameters were same as selected for treatment of Patchouli crude extract, as described earlier. Reference may be made to
- 15 Table 6 below.
- [0085]** FIG. 5 shows a photograph 503 depicting the extract 507 (described in similar context to the SCFE 207 of FIG. 2) obtained from SCFE, a Stage 2 sample 511, and a Stage 3 sample 513 and another part of the Stage 3 sample 515, according to one example. The contents 511, 513, 515 of the samples may be described in similar context
- 20 to 211, 213, 215 of FIG. 2.
- [0086]** Table 4 shows the characteristics of clove buds SCFE extract, and samples processed with different stages.

Experiment No.	Clove Buds (MDT50)			
Parameters	Crude Extract 507	Molecular Distilled Stage 2 Sample 511	Molecular Distilled Stage 3 Sample (in one part) 513	Molecular Distilled Stage 3 Sample (in other part) 515
Colour	Dark	Colorless	Light yellow	Reddish

	Yellow			Brown
Wax	Present	Not present	Not Present	Present
Eugenol (80-95%)	77.07 *	73.97	76.51	66.33
Refractive Index (1.529-1.537)	1.523	1.529	1.539	1.528
Density (1.04-1.07)	1.03	1.05	1.05	1.04
Miscibility in 90% Ethanol	Fail	Pass	Pass	Fail
SNI Standard	Fail	Pass	Pass	Fail

*The low Eugenol in the final processed oil may be due to a poor-quality feedstock where the original Eugenol content was already on a lower side (of about 77%).

GINGER

- 5 [0087] A specific example of preparing essential oils and related products from ginger, along with experimental data will be presented below.
- [0088] Elephant Ginger (*Zingiber officinale*) (100 kg) was extracted with SCFE at 300 bar, 50 °C and 15 kg/hr flow rate for 2 hours. Total 5.6 kg extract was obtained from 100 kg ginger (yield 5.6%). The extract was homogenized by heating over a water bath at 60 °C and re-packed into 1L bottles for MD post processing.
- 10 [0089] The Ginger extract (1kg) was heated to 60 °C in a hot water bath and processed through molecular distillation. Parameters chosen for MD processing of the extract were as follow: the temperature of Stage 1 231a was 60 °C, Stage 2 231b was at 90 °C and Stage 3 231c was at 120 °C. Flow rate and other parameters were same as selected for treatment of Patchouli crude extract, as described earlier. Reference may be made to Table 6 below.
- 15 [0090] FIG. 6A shows a photograph 601 depicting the extract 607 (described in similar context to the SCFE 207 of FIG. 2) obtained from SCFE, according to one example. FIG. 6B shows a photograph 603 depicting a Stage 2 sample 611, and a Stage 3 sample 613 and another part of the Stage 3 sample 615. The contents 611, 613, 615 of the samples may be described in similar context to 211, 213, 215 of FIG. 2.
- 20

[0091] Table 5 shows the characteristics of ginger SCFE extract, and samples processed with different stages.

Parameters	Experiment No. Ginger MDT65			
	Crude Extract	Molecular Distilled Stage 2 Sample 611	Molecular Distilled Stage 3 Sample (in one part) 613	Molecular Distilled Stage 3 Sample (in other part) 615
Feed	SCFE Extract 607	SCFE Extract	SCFE Extract	SCFE Extract
Color	Reddish Brown	Light Yellow	Light Yellow	Reddish Brown
Wax	Present	Not present	Not present	Present
Zingiberene (>20%)	25.67	19.8	20.7	17.54
Refractive Index (1.485-1.492)	1.507	1.488	1.494	1.513
Density (0.8720-0.8890)	0.978	0.881	0.886	0.977
Miscibility in Ethanol	Fail	Pass	Pass	Fail
SNI Standard	Fail	Pass	Pass	Fail

[0092] As seen from Tables 1, 3, 4 and 5, the SCFE crude extract of various exemplary spices failed the SNI testing, and the samples obtained after MD processing at Stages 2 and 3 (in one part), i.e. molecular distilled Stage 2 sample and molecular distilled Stage 3 sample (in one part), respectively, passed the SNI testing.

[0093] Table 6 is a comparison table for all four products' parameters for material processing/feedstock preparation, SCFE extraction, and post processing with molecular distillation.

No.	Parameters	Patchouli	Ginger	Nutmeg	Clove Buds
Feedstock Parameters					
1	Powder Mesh Size (mm)	2	2	1	2
2	Pellets Compression Ratio	1/3.3	1/3.3	1/2.7	1/2.7

3	Pellets Water (%)	1	3	None	None
4	Pellets Moisture (%)	~8-10	~12	~7	~17
5	Pellets Density (g/cm ³)	~0.6	~0.5	~0.6	~0.6
SCFE Extraction Parameters					
6	Pressure (Bar)	300	300	300	300
7	Temperature (°C)	50	50	60	50
8	Flow Rate (kg/hr)	15	15	15	15
9	Duration (Hours)	2	2	2	2
MD Processing Parameters					
10	Feed Flow rate (L/hr)	3	3	3	3
11	Feed Temperature (°C)	60	60	60	50
12	Pipes Temperature (°C)	60	60	60	50
13	Stage 1 Temperature (°C)	60	60	60	60
14	Stage 2 Temperature (°C)	90	90	60	100
15	Stage 3 Temperature (°C)	110	120	110	110
16	Stage 1 Rotation Speed (RPM)	100	100	100	100
17	Stage 2 Rotation Speed (RPM)	140	140	140	140
18	Stage 3 Rotation Speed (RPM)	140	140	140	140
19	Chiller Temperature (°C)	-15	-15	-15	-15
20	Air cooler Temperature (°C)	7	7	7	7

- [0094] For the ease of comparison, Table 7 summarizes the results for samples (nutmeg, clove buds and ginger) obtained from molecular distillation processing of the respective SCFE extracts, based on one experiment for each, and reflecting the yields of the
- 5 respective SCFE extracts after MD processing.

Experiment No.	MDT65	MDT50	MDT59
Feed	Ginger SCFE Extract	Clove Bud SCFE Extract	Nutmeg SCFE Extract
SCFE total Yield %	5.60	20	25
Feed weight	1 kg	1 kg	1 kg

Temperature (°C)	60 (Stage 1 231a) 90 (Stage 2 231b) 120 (Stage 3 231c)		60 (Stage 1 231a) 100 (Stage 2 231b) 110 (Stage 3 231c)		60 (Stage 1 231a) 60 (Stage 2 231b) 110 (Stage 3 231c)	
	Zingiberene %	Yield %	Eugenol %	Yield%	Myristicin %	Yield%
Molecular Distilled Stage 2 Sample	19.90	18.8	73.97	57.88	22.1	10.85
Molecular Distilled Stage 3 Sample (in one part)	20.65	31.5	76.51	26.77	0.55	27.17
Molecular Distilled Stage 3 Sample (in other part)	-	27.4	-	7.28	43.25	36.57
Recovery of Clear Oil (Stage 2 Sample + Stage 3 Sample (in one part))	-	50.3	-	84.64	-	38.03
Clear Oil %= crude extract%* recovery rate	5.6 *0.503= 2.81%		20*0.85 = 17.00%		25*0.38= 9.50%	

[0095] It is noted that for Experiment No. MDT59, the nutmeg SCFE extract was blended with a commercial oil to merely increase the fluidity of the extract for the equipment during laboratory trials to be used. It should be appreciated that this blending do not affect the comparative data as presented in Table 7.

[0096] While the invention has been particularly shown and described with reference to specific embodiments, it should be understood by those skilled in the art that various changes in form and detail may be made therein without departing from the spirit and scope of the invention as defined by the appended claims. The scope of the invention is thus indicated by the appended claims and all changes which come within the meaning and range of equivalency of the claims are therefore intended to be embraced.

CLAIMS

1. A method for preparing a botanical extract, the method comprising:
subjecting a botanical material to an extract pressure under influence of liquid
5 carbon dioxide to perform a supercritical carbon dioxide extraction process to obtain an
extraction mixture;
separating an intermediate extract from the extraction mixture; and
substantially immediately following the step of separating, feeding the
intermediate extract to a series of stages to perform molecular distillation to obtain the
10 botanical extract with an improved level of one or more parameters as compared to the
intermediate extract,
wherein the one or more parameters comprise at least one of density, acid
value, refractive index, appearance, or miscibility, and
each stage is set to a predetermined distillation temperature, a
15 predetermined vacuum pressure and a predetermined rotation speed.
2. The method as claimed in claim 1, further comprising prior to the step of
subjecting the botanical material to the extract pressure under influence of liquid carbon
dioxide, grounding at least part of a plant into a powder and pelleting the powder to
20 obtain the botanical material in granular form.
3. The method as claimed in claim 2, wherein the powder has a size of about 140
mesh or less, and the botanical material in granular form has an average diameter of
about 2 mm to about 10 mm, and an average thickness of about 2 mm to about 4.5 mm.
25
4. The method as claimed in claim 2 or 3, further comprising prior to grounding the
at least part of the plant, drying the at least part of the plant to reduce moisture content to
15% or less.
- 30 5. The method as claimed in any one of claims 1 to 4, wherein the extract pressure
is about 100 bar to about 500 bar.

6. The method as claimed in any one of claims 1 to 5, wherein the supercritical carbon dioxide extraction process is performed under an extract temperature ranging from 40 °C to 60 °C.
- 5 7. The method as claimed in any one of claims 1 to 6, wherein the liquid carbon dioxide is provided at a flow rate ranging from 5 kg/h to 30 kg/h for about 10 minutes to about 3 hours.
8. The method as claimed in any one of claims 1 to 7, wherein the step of separating
10 is performed under a depressurized condition of about 40 bar.
9. The method as claimed in any one of claims 1 to 8, wherein the step of feeding the intermediate extract further comprises heating the intermediate extract at about a temperature ranging from 40 °C to 60 °C into molten form.
15
10. The method as claimed in any one of claims 1 to 9, wherein the predetermined distillation temperature is ranging from 30 °C to 110 °C.
11. The method as claimed in any one of claims 1 to 10, wherein the predetermined
20 vacuum pressure varies from 0.2 mbar to 1 mbar.
12. The method as claimed in any one of claims 1 to 11, wherein the predetermined rotational speed is ranging from 100 rpm to 140 rpm.
- 25 13. The method as claimed in any one of claims 1 to 12, wherein a first stage of the series of stages is set to the predetermined distillation temperature of about 60 °C and the predetermined rotational speed of about 100 rpm; a second stage of the series of stages is set to the predetermined distillation temperature of about 90 °C and the predetermined rotational speed of about 140 rpm; and a third stage of the series of stages is set to the
30 predetermined distillation temperature of about 110 °C and the predetermined rotational speed of about 140 rpm.

14. The method as claimed in claim 13, wherein the first stage precedes the second stage, and the second stage precedes the third stage.

5 15. The method as claimed in any one of claims 1 to 14, wherein the botanical extract has an alcohol content of about 25 % to about 50 %.

16. The method as claimed in any one of claims 1 to 15, wherein the at least part of the plant comprises patchouli leaves, clove buds, nutmeg, ginger, cinnamon, cassia, black
10 pepper or a plant material containing essential oils.

17. An apparatus for preparing a botanical extract, the apparatus comprising:
a supercritical carbon dioxide extraction module configured to apply an extract pressure under influence of liquid carbon dioxide to a botanical material to perform a
15 supercritical carbon dioxide extraction process to obtain an extraction mixture;
a separation module in fluidic communication with the supercritical carbon dioxide extraction module, the separation module configured to separate an intermediate extract from the extraction mixture; and
a molecular distillation module in fluidic communication with the separation
20 module, the molecular distillation module comprising:
a series of stages configured to perform molecular distillation to obtain the botanical extract with an improved level of one or more parameters as compared to the intermediate extract; and
one or more pipes, each coupled between one stage of the series of stages
25 and a subsequent stage of the series of stages,
wherein the one or more parameters comprise at least one of density, acid value, refractive index, appearance, or miscibility, and
each stage is operable with a predetermined distillation temperature, a predetermined vacuum pressure and a predetermined
30 rotation speed.

18. The apparatus as claimed in claim 17, wherein the separation module is further configured to perform separation under a depressurized condition of about 40 bar.

19. The apparatus as claimed in claim 17 or 18, further comprising a heating module
5 configured to heat the intermediate extract at about a temperature ranging from 40 °C to 60 °C into molten form.

20. The apparatus as claimed in any one of claims 17 to 19, wherein the one or more pipes is arranged to be heated to about 50 °C.

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21. The apparatus as claimed in any one of claims 17 to 20, wherein the series of stages comprises a first stage configured to operate at the predetermined distillation temperature of about 60 °C and the predetermined rotational speed of about 100 rpm; a second stage configured to operate at the predetermined distillation temperature of about
15 90 °C and the predetermined rotational speed of about 140 rpm; and a third stage configured to operate at the predetermined distillation temperature of about 110 °C and the predetermined rotational speed of about 140 rpm.

22. The apparatus as claimed in claim 21, wherein the first stage precedes the second
20 stage, and the second stage precedes the third stage.

23. A botanical extract prepared by performing a method as claimed in any one of claims 1 to 16.

24. The botanical extract as claimed in claim 23, having an alcohol content of about
25 25 % to about 50 %, or preferably about 29% to about 31%, or more preferably about 40% to about 50%.