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(54) **Essential oils treated to remove harsh notes therefrom.**

(57) Essential oils extracted from botanical material are treated with a peroxide-acid reagent to remove harsh flavor off-notes therefrom.

1 ESSENTIAL OILS TREATED TO
 REMOVE HARSH NOTES THEREFROM

5 The present invention relates to essential oils
 extracted from botanical matter which are treated with a
 peroxide-acid reagent to remove harsh flavor notes therefrom.

10 Essential oils which are freshly distilled from
 botanical matter usually have, when freshly made, what are
 known as harsh tasting flavor notes. These harsh flavor
 off-notes are also known as green, burnt, still off-notes.
15 This is particularly true in the case of peppermint oil or
 spearmint oil which is freshly made by means of steam
 distillation of the parent botanical matter from which the
 oil is extracted. The customary procedure employed for
 removing those harsh flavor notes which are present in the
20 freshly obtained oil is to allow the oil to age or mellow for
 periods of about 6 to 24 months, in full containers in the
 presence of oxygen and in the absence of actinic radiation.
 This aging-mellowing process is economically unattractive
 since it requires the use of carefully monitored storage
25 facilities, for long periods of time and supervised by
 technically trained personnel. All of these storage
 requirements are economically burdensome.

 It has now been found according to the present
25 invention that essential oils freshly extracted from
 botanical matter which then contain harsh flavor off-notes
 can be readily freed of such harsh flavor off-notes by
 treating the oil with a peroxide-acid reagent.

 The peroxide-acid reagent which is used to treat
30 the essential oils in accordance with the present invention
 is a combination of one or more water soluble inorganic
 peroxides such as hydrogen peroxide and/or ozone and one or

1 more water soluble inorganic acids such as hydrochloric acid,
sulfuric acid and phosphoric acid. The peroxide is the
active agent in the reagent system of the present invention.
The acid is merely used to acidify the peroxide.

5 The peroxides and the acids are preferably employed
as aqueous solutions having a peroxide or acid concentration
of about 30 to 40 weight percent. Aqueous solutions of the
peroxides and/or acids having higher or lower concentrations
may also be used.

10 The peroxide and acid components of the reagent are
added together to the oil to be treated therewith, as
disclosed below.

15 The essential oils which are to be treated with the
peroxide-acid reagent in accordance with the present
invention are the freshly extracted essential oils of
botanical matter. These oils are used as flavorants in the
food, confectionary, perfume and cosmetic industries. These
oils would include those obtained from the following
botanical materials: anise, basil, dill weed, chamomile,
20 eucalyptus, fennel, geranium, hop, laurel leaf, lemongrass,
bois de rose, caraway, amber, camphor, amyris, galbanum,
davana, mentha (spearmint and peppermint).

25 The essential oils which are to be treated with the
peroxide-acid reagent in accordance with the present
invention may be extracted from their parent botanical
matter, i.e., leaves, fruit, bark, root, grass, wood,
heartwood, gum, balsam, berries, seed, flowers, twigs and
buds, by the commonly employed technique for doing so, i.e.,
steam distillation.

30 U.S. 4,478,864 discloses the treatment of freshly
prepared peppermint oil with maleic anhydride for the
purposes of preventing the formation of certain off flavor

1 notes during the aging process. This process removes
most of the menthofuran from the fresh peppermint oil, in the
form of a menthofuran-maleic anhydride complex. Thus, very
little menthofuran is present, during the aging process, to
5 oxidize to produce undesirable flavor notes. This process,
however, does not cure all the off-flavor note problems
inherent in fresh peppermint oil. An essential oil treated
by the process of U.S. 4,478,864 may still have to undergo an
aging process to remove off-flavor notes that are present in
10 the freshly prepared oil.

Prior to all of the present inventors work,
therefor, it has not been possible to treat freshly made
essential oils in a facile manner so as to readily remove
therefrom harsh flavor off-notes then present therein with a
15 reagent that can be readily removed from the oil.

The present invention relates to an essential oil
of botanical material treated with an acidified peroxide
and/or ozone to remove harsh flavor off-notes therefrom. The
present invention also relates to a process for removing
20 harsh flavor off-notes from the essential oil of a botanical
material which comprises treating said oil with an acidified
peroxide and/or ozone.

The fresh oil may be rectified (redistilled) prior
to or after treatment with the peroxide-acid reagent to
25 improve a particular property characteristic. For example,
peppermint oil may be rectified to remove dimethyl sulfide
therefrom which provides a green weedy note.

The harsh flavor off-notes in the fresh essential
oils, which are to be removed by treatment with the
peroxide-acid reagent, may be characterized, as such,
30 organoleptically. Organoleptically these harsh off-flavor
notes may be characterized as: harsh, green, raw, weedy,
skunky and burnt.

1 The chemical components of the fresh essential oils
which are believed responsible for the harsh (tasting) flavor
off-notes have not been determined analytically. They are
present, at most, at trace concentrations in the oil. When
the essential oils are analyzed by gas chromatography prior
5 to and after the treatment of the present invention, no
apparent changes in the composition of the volatile
components of the oil can be detected (analytically).

By volatile components it is meant those components
of the oil which are volatile enough as to be capable of
10 being detected by gas chromatography analysis using a
Carbowax-20M column operated at a maximum temperature of
230°C and with an injector temperature of a maximum of 250°C.

In treating the essential oil with the peroxide-
acid reagent according to the present invention the oil may
15 be extracted in a liquid/liquid extraction with the peroxide-
acid reagent, or it may be contacted with the peroxide-acid
reagent immobilized on a solid support.

About 0.005 to 2.0, and preferably about 0.01 to
0.03, percent by volume of each of the peroxide and the acid
20 components of the reagent of the present invention is used in
treating the oil therewith, when using the peroxides and
acids as 30 to 40% by weight aqueous solutions.

The peroxide and acid components are added to the
oil, for the purposes of conducting the treatment therewith
25 of the present invention, by adding such components together.
The acid is used to form an acidified peroxide.

The treatment with the peroxide-acid reagent may be
conducted at room temperature, of about 20-25°C, although at
higher temperatures a more rapid/efficient extraction may be
30 effected. The liquid/liquid extraction may be done by
vigorously shaking a mixture of the oil and the peroxide-acid

1 reagent in commonly employed shaking devices designed to
effect efficient liquid/liquid extraction systems. Depending
on the amount of peroxide-acid reagent used, the size of the
oil sample being extracted, and the amount of harsh flavor
5 off-notes initially present in such sample, and the shaking
device employed, the extraction time required may be about
one to ten minutes.

Only one treatment of the oil with the
peroxide-acid reagent is needed in order to adequately
10 accomplish the removal of the harsh flavor off-notes. With
such a treatment all of the harsh flavor notes are readily
removed.

After the aqueous peroxide-acid reagent is used to
treat the essential oil in the liquid/liquid extraction
15 process, it is readily moved therefrom by the use of
oil/water separating devices such as a separatory funnel,
with or without prior centrifuging. Residual traces of the
peroxide-acid reagent can be further readily extracted from
the oil by treatment with a solution of Na_2CO_3 (to
20 neutralize the acid) followed by washing with distilled
water. The oil is then dried by high speed centrifugation.

Contrary to the teachings of U.S. 4,478,864,
whereby the agent which would cause the creation of the off
flavor note (upon aging in the oil) is actually removed from
the fresh oil as a menthofuran-maleic anhydride complex, the
25 agent(s) which are causing the off-flavor notes in the fresh
oil, and which are treated with the peroxide-acid reagent
according to the present invention, are not removed from the
fresh oil by such treatment. The peroxide-acid reagent
presumably oxidizes the agents causing the off-flavor notes
30 in such a way as to then render them innocuous from an

1 off-flavor point of view. Thus, it is not necessary to
further age the fresh oil, as is otherwise commonly done, to
accomplish the same result.

5 The treatment of the fresh essential oil with the
peroxide-acid reagent can be accomplished before or after any
treatment of the oil according to the process of U.S.
4,478,864.

10 The various types of products into which the
essential oils of the present invention may be added as
flavors or fragrances would include food, confectionary,
including chewing gum and pressed mints, perfumes, cosmetic
and body hygiene products and medicinals.

15 The following examples are merely illustrative of
the scope of the present invention and are not intended as a
limitation upon the scope thereof.

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1 Example 1

5 A 100 ml sample of freshly distilled peppermint oil
is shaken, in a 250 ml separatory funnel, with 0.02 grams
each of 30% by weight aqueous H_2O_2 and 12 N HCl for 1 to
2 minutes. The aqueous (bottom) layer is discarded after
complete separation of the layers. The peppermint oil layer
is washed by extraction with 2 x 50 ml 5% sodium carbonate
solution and 2 x 50 ml distilled water, and the completely
10 separated aqueous layer is then discarded. The oil is dried
with 4 g anhydrous sodium sulfate and by centrifugation at
2000 to 3000 rpm.

The resulting oil is crystal clear and possesses a
clean characteristic mellow aroma of a good quality aged
15 peppermint oil. The harsh objectionable aroma of the
starting oil is eliminated. The taste of the treated oil,
when evaluated in a sugar fondant at 0.2 weight percent level
and in a chewing gum at about a 0.5 to 1.5 weight percent
level, is similarly improved from that of the starting oil.

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1 Example 2

5 A sample of freshly distilled spearmint oil is also
treated according to the procedure of Example 1. A similar
improvement of the treated oil is noted.

10 The magnitude of improvement in a treated oil
depends on the quality of the starting oil; the more
objectionable and the harsher the starting oil is, the more
dramatic is the improvement obtained by the treatment thereof
according to the present invention.

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CLAIMS

1. An essential oil of botanical material treated with an acidified peroxide and/or ozone to remove harsh flavor off-notes therefrom.

2. An essential oil as in claim 1 in which the acidified peroxide and/or ozone is in the presence of at least one water soluble inorganic acid, and the peroxide is an inorganic peroxide.

3. An essential oil as in claim 1 or 2 in which the peroxide is hydrogen peroxide.

4. An essential oil as in any of claims 1 to 3 in which the acid is hydrochloric acid, sulfuric acid or phosphoric acid.

5. An essential oil as in any of claims 2 to 4 in which the peroxide is hydrogen peroxide and the acid is hydrochloric acid.

6. An essential oil as in any of claims 1 to 5 which is a mint oil, a peppermint oil, spearmint oil or a distilled oil.

7. A flavored or fragranced product made with the essential oil of any of Claims 1 to 6.

8. A confectionary, food, cosmetic, perfume, body hygiene product or medicinal made with the essential oil of any of Claims 1 to 6.

9. A confectionary as in Claim 8 which is chewing gum, a candy or a pressed mint.

10. A process for removing harsh flavor off-notes from the essential oil of a botanical material which comprises treating said oil with an acidified peroxide and/or ozone.

11. A process as in Claim 10 in which the acidified peroxide and/or ozone is in the presence of at least one water soluble acid, and peroxide is an inorganic peroxide.

1 12. A process as in Claim 10 or 11 in which the
peroxide is hydrogen peroxide.

 13. A process as in any of Claims 10 to 12 in which
the acid is hydrochloric acid, sulfuric acid or phosphoric
acid.

5 14. A process as in any of Claims 10 to 13 in which
the acidified peroxide is hydrogen peroxide and the acid is
hydrochloric acid.

 15. A process as in any of Claims 10 to 14 in which
10 said essential oil is a mint oil, a peppermint oil, a
spearmint oil or a distilled oil.

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