

PATENT SPECIFICATION (11)

1 389 260

1 389 260

- (21) Application No. 30976/72 (22) Filed 3 July 1972 (19)
 (31) Convention Application Nos. 160 095 (32) Filed 6 July 1971
 222 376 31 Jan. 1972 in
 (33) United States of America (US)
 (44) Complete Specification published 3 April 1975
 (51) INT. CL.² C12D 1/00 C12C 11/04 C12D 3/00 C12G 1/00
 (52) Index at acceptance
 C6E 3



(54) RECOVERING FLAVOURING SUBSTANCES

(71) We, TOBACCO RESEARCH AND DEVELOPMENT INSTITUTE LIMITED, of Baarerstrasse 10, Zug, Switzerland, a company registered according to the laws of Switzerland, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the recovery of flavouring substances.

The use of flavouring substances derived from vegetable materials has long been known. Thus some materials, e.g. spices, have been used as such to provide flavouring. In other cases fermented vegetable materials were distilled for the flavouring substances to collect in the distillate. In still other cases infusions of vegetable substances were made using liquid or vaporous ethyl alcohol. Sometimes combinations of these methods were used. A method of upgrading a fermented beverage is described in our co-pending Application No. 30975/72 (Serial No. 1,389,259).

The present invention is based on the discovery that when many vegetable materials are subjected to a microbial fermentation, the flavours contained in the materials are liberated into the off-gases of the fermentation process.

According to the invention a method of recovering flavouring substances from a vegetable material comprises the steps of pulping the vegetable material; adding water to the pulp, determining the sugar content of the volume and increasing the sugar content if it is below a suitable value, for fermentation to proceed, fermenting the resultant mixture with an alcohol-producing yeast culture, passing the off-gases through activated carbon to adsorb and absorb the flavouring substances contained in the off-gases, and desorbing the activated carbon to recover the flavouring substances.

The activated carbon can be described in a variety of ways known to the art.

Vegetable materials used by the present invention have wide origins, and, since one

is dealing with substances that exist in minute amounts and of which the chemical compositions are presently unknown, the only way of testing is organoleptic i.e. by taste and smell.

The invention has been applied to a large number of materials of vegetable origin. One way of proceeding is described generally as follows:

An amount of the material is collected and pulped. The volume of the pulp is brought up to a suitable volume by the addition of water and the sugar content is raised to 20°B on the Balling scale of densities, if there is not enough sugar present. The mash is then inoculated with a pure strain of yeast. In the majority of cases a pure strain of grape yeast was used. The fermentation temperature was kept at 25°C. The off-gases from the fermentation were passed through a bed of 5g of activated carbon.

The activated carbon was described in the main series of experiments with ethanol at for example 90°C i.e. with ethanol vapour. Later experiments show that desorption with liquid ethanol at room temperature at a rate of 0.1 to 0.2 of the bed volume per hour produced much better results.

It has also been shown that desorption by nitrogen gas and under vacuum can be used in certain cases. Desorption with steam also produces good results, even though this is done at higher than room temperature. Desorption by gases inert to the system such as nitrogen and carbon dioxide in gas or liquid form can also be used.

Fermentation was continued for 120 hours in each case.

The types of material which were examined can be classified as follows: the experiments being carried out generally under the conditions specified hereinbefore.

1. Spices

(a) In one experiment finely ground coriander was made into a pulp with water. 1.25 kg of coriander was used and the pulp was made up to 10 litres while sugar was added to bring it up to 20°B. The pulp

[Price 33p]

was fermented with grape yeast. On desorption with ethanol at 90°C the final liquid had a faint coriander flavour.

- (b) Ground ginger in an amount of 1.47 kg was pulped, the volume increased to 10 l and the sugar content to 20°B. On desorption a liquid with a marked ginger flavour was produced.

- (c) 380 g of cinnamon was made up to 10 l and 20°B also produced only a minute flavour.

2. Edible fruits

- (a) 9 litres of orange juice was diluted to 10 litres and brought up to 20°B. A grape yeast was used. The desorption liquid had a faint smell of orange.

- (b) In the case of lemon juice 4.5 kg was diluted to 10 litres at 20°B. Once more a faint lemon smell was noted.

- (c) Mandarin juice in an amount of 3 litres was mixed with 12.5 kg of mandarin rinds and the whole pulped. The pulp was brought up to 10 litres at 20°B. A faint mandarin smell was found in the final product.

- (d) An unrecorded amount of papaya fruit was pulped and mixed with water to bring the volume up to 10 litres. The sugar content was raised to 20°B. Desorption with ethanol at 90°C produced a liquid with a marked papaya flavour. Under this heading this was the most successful experiment.

3. Fruit rinds

- Under this heading in one experiment, 4 kg each of orange peels and mandarin peels were finely pulped in 10 litres of water brought up to 20°B. The final liquid had a detectable orange and a mandarin smell.

4. Flowers

- 2.2 kg of grape flowers were pulped and the pulp adjusted to 10 litres at 20°B. The final liquid had a very strong wine flavour and could be used to improve the organoleptic qualities of large volumes of inferior wine.

5. Berries

- 1,270 g of juniper berries were pulped in 10 litres of water and the sugar content brought up to 20°B. A liquid with a very strong juniper aroma was produced.

6. Beans and seeds

- (a) An experiment with vanilla beans was not successful.

- (b) Hops seeds produced a product with a very marked aroma. In the latter case 1,150 g of hops seeds were ground and pulped with 10 litres of water. The sugar content was adjusted as before.

- (c) Aniseed also yielded a faint aroma only.

7. Herbaceous parts of plants

(In this specification, the phrase "Herbaceous parts of plants" is to be taken to include all parts of plants, except fruits, fruit rinds, flowers, berries, beans and seeds).

In unquantified experiments wormwood and tobacco were tried. In each case the characteristic aroma was obtained.

8. Substances extracted from plants

2.2 kg of molasses was diluted to 10 litres and 20°B. A rum yeast was used. A very marked rum flavour was obtained in the final product.

WHAT WE CLAIM IS:—

1. A method of recovering flavouring substances from a vegetable material, comprising the steps of pulping the vegetable material, adding water to the pulp, determining the sugar content of the volume and increasing the sugar content if it is below a suitable value for fermentation to proceed, fermenting the resultant mixture with an alcohol-producing yeast culture, passing the off-gases through activated carbon to absorb and adsorb the flavouring substances contained in the off-gases, and desorbing the activated carbon to recover the flavouring substances.

2. The method as claimed in claim 1, in which the activated carbon is desorbed by a method selected from desorption by steam, desorption with ethanol vapour, desorption with liquid ethanol, desorption with gas inert to the system, and vacuum desorption.

3. The method claimed in either of the above claims in which the material is selected from spices, fruits, fruit rinds, flowers, berries, beans and seeds, herbaceous parts of plants and substances extracted from plants.

4. The method claimed in claim 3 in which the material consists of juniper berries, hops seeds, pulped papaya, grape flowers or molasses.

5. The method of extracting flavouring substances according to claim 1 and substantially as herein described.

6. Flavouring substances when produced by the method of any of the preceding claims.

ARTHUR R. DAVIES,
Chartered Patent Agents,
27, Imperial Square, Cheltenham,
and
115, High Holborn,
London, W.C.1.
Agents for the Applicants.



was fermented with grape yeast. On desorption with ethanol at 90°C the final liquid had a faint coriander flavour.

- 5 (b) Ground ginger in an amount of 1.47 kg was pulped, the volume increased to 10 l and the sugar content to 20°B. On desorption a liquid with a marked ginger flavour was produced.

- 10 (c) 380 g of cinnamon was made up to 10 l and 20°B also produced only a minute flavour.

2. Edible fruits

- 15 (a) 9 litres of orange juice was diluted to 10 litres and brought up to 20°B. A grape yeast was used. The desorption liquid had a faint smell of orange.

- 20 (b) In the case of lemon juice 4.5 kg was diluted to 10 litres at 20°B. Once more a faint lemon smell was noted.

- 25 (c) Mandarin juice in an amount of 3 litres was mixed with 12.5 kg of mandarin rinds and the whole pulped. The pulp was brought up to 10 litres at 20°B. A faint mandarin smell was found in the final product.

- 30 (d) An unrecorded amount of papaya fruit was pulped and mixed with water to bring the volume up to 10 litres. The sugar content was raised to 20°B. Desorption with ethanol at 90°C produced a liquid with a marked papaya flavour. Under this heading this was the most successful experiment.

35 3. Fruit rinds

- 40 Under this heading in one experiment, 4 kg each of orange peels and mandarin peels were finely pulped in 10 litres of water brought up to 20°B. The final liquid had a detectable orangy and a mandarin smell.

4. Flowers

- 45 2.2 kg of grape flowers were pulped and the pulp adjusted to 10 litres at 20°B. The final liquid had a very strong wine flavour and could be used to improve the organoleptic qualities of large volumes of inferior wine.

50 5. Berries

- 55 1,270 g of juniper berries were pulped in 10 litres of water and the sugar content brought up to 20°B. A liquid with a very strong juniper aroma was produced.

6. Beans and seeds

- (a) An experiment with vanilla beans was not successful.

- 60 (b) Hops seeds produced a product with a very marked aroma. In the latter case 1,150 g of hops seeds were ground and pulped with 10 litres of water. The sugar content was adjusted as before.

- (c) Aniseed also yielded a faint aroma only.

7. Herbaceous parts of plants

(In this specification, the phrase "Herbaceous parts of plants" is to be taken to include all parts of plants, except fruits, fruit rinds, flowers, berries, beans and seeds).

In unquantified experiments wormwood and tobacco were tried. In each case the characteristic aroma was obtained.

8. Substances extracted from plants

2.2 kg of molasses was diluted to 10 litres and 20°B. A rum yeast was used. A very marked rum flavour was obtained in the final product.

WHAT WE CLAIM IS:—

1. A method of recovering flavouring substances from a vegetable material, comprising the steps of pulping the vegetable material, adding water to the pulp, determining the sugar content of the volume and increasing the sugar content if it is below a suitable value for fermentation to proceed, fermenting the resultant mixture with an alcohol-producing yeast culture, passing the off-gases through activated carbon to absorb and adsorb the flavouring substances contained in the off-gases, and desorbing the activated carbon to recover the flavouring substances.

2. The method as claimed in claim 1, in which the activated carbon is desorbed by a method selected from desorption by steam, desorption with ethanol vapour, desorption with liquid ethanol, desorption with gas inert to the system, and vacuum desorption.

3. The method claimed in either of the above claims in which the material is selected from spices, fruits, fruit rinds, flowers, berries, beans and seeds, herbaceous parts of plants and substances extracted from plants.

4. The method claimed in claim 3 in which the material consists of juniper berries, hops seeds, pulped papaya, grape flowers or molasses.

5. The method of extracting flavouring substances according to claim 1 and substantially as herein described.

6. Flavouring substances when produced by the method of any of the preceding claims.

ARTHUR R. DAVIES,
Chartered Patent Agents,
27, Imperial Square, Cheltenham,
and
115, High Holborn,
London, W.C.1.
Agents for the Applicants.