

# Flavour extract

The present invention relates to a process for the extraction of flavours, and to the use of these flavours  
5 for making food compositions.

The extraction of flavours in the presence of polar solvents is known.

10 The extraction of meat or fish flavours using water as a polar solvent is particularly known. The flavour extract isolated by means of this type of extraction contains flavours but also non-volatile compounds which are important to the taste.

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Thus, US 3957745 describes a process for the preparation of a mixture of flavours in which a source of sulphur, i.e. inorganic sulphur and/or an amino acid, reacts with an alpha-hydroxycarbonyl, a beta-epoxycarbonyl or an alpha-  
20 epoxycarbonyl in the presence of at least one polar solvent, at a temperature of 65-200°C and a pressure of 1 to 100 atm, so as to produce flavours and the flavours are then separated from the residue, particularly by filtration. These flavours are particularly used to modify  
25 the flavour note or the taste of a food product.

It is further known to use CO<sub>2</sub> to extract spices and herbs or to isolate flavours of oil, fats, fruits or coffee, for example.

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Thus, US 3477856 describes a process for the isolation of volatile flavours from fruit juices or essences in which a liquid CO<sub>2</sub> extraction is performed on a fruit juice or essence, the phase containing the flavours is separated  
35 from the residual fruit juice or essence phase and the CO<sub>2</sub> contained in this phase is then evaporated so as to isolate a liquid extract containing volatile fruit flavours.



Further, US 4328255 describes a process for the extraction of a coffee oil containing flavours in a stable form, in which a supercritical CO<sub>2</sub> extraction is performed on roasted coffee at no less than 80 atm and at a temperature greater than 31.3°C.

The object of the present invention is to propose a simple and rapid process enabling volatile flavours to be extracted. In addition, the process according to the present invention has the advantage of enabling very concentrated flavours to be obtained.

To this end, in the process for the extraction of flavours according to the present invention, a mixture is prepared containing, at least, a source of sulphur, a reducing sugar, a phosphate compound and water, this mixture is heated at 50-180°C for 30 min to 5 h, this mixture is subjected to a CO<sub>2</sub> counter-current liquid extraction at 70-350 bar and a separation is then performed on the CO<sub>2</sub> loaded with flavours, so as to obtain a liquid flavour extract.

Hereafter in the description, the expression "phosphate compound" will be used to refer to phosphate, biphosphate or a polyphosphate.

In addition, hereafter in the description, the expression "extraction yield" will be used to refer to the ratio of the quantity of flavour extract obtained according to the present invention to the quantity of starting mixture.

Surprisingly, it has been found that the process according to the present invention allows volatile flavours to be extracted simply and rapidly.

For this purpose, then, a mixture is prepared containing, at least, a source of sulphur, a reducing sugar, a phosphate compound and water. A mixture may be prepared containing, at least, 1-30% of a source of sulphur, 1-30% of a reducing sugar, 1-15% of a phosphate compound and 25-55% of water, for example.

A mixture may be prepared containing glutathione, cysteine, thiamine and/or inorganic sulphur, for example, as the source of sulphur.

A mixture may be prepared containing glucose and/or xylose, for example, as the reducing sugar.

A mixture may be prepared containing a phosphate compound, particularly in such a way as to maintain the pH of the mixture at a value of between 4.5-7.5 inclusive. Phosphate, biphosphate or a polyphosphate is therefore used as the phosphate compound. An ammonium phosphate is preferably used.

This mixture is thus heated at 50-180°C for 30 min to 5 h so as to carry out a Maillard reaction, i.e. so as to make the reducing function of the sugar react with the amine function of the source of sulphur or of another compound contained in the mixture. If the source of sulphur does not have an amine function, ammonium phosphate may be used as the phosphate compound, for example, so as to carry out the Maillard reaction. The phosphate compound contained in the mixture acts as a catalyst during this reaction, in addition to its buffer capacity. In this way the flavours of the mixture are developed. This step may be performed with agitation, particularly in a double casing reactor such as that marketed by Schmizo A.G., Luzerner Strasse 27, CH-4665 Zofingen, so as to obtain a homogeneous reaction, for example.

A CO<sub>2</sub> counter-current liquid extraction is then performed on this mixture at 70-350 bar. This counter-current extraction may be performed in an extractor, particularly in a column extractor in which the mixture is fed in through the top of the column and the CO<sub>2</sub> is fed in through the bottom of the column, for example.

The flavour note of the flavour extract may particularly be varied according to the temperature at which the CO<sub>2</sub> counter-current liquid extraction at 70-350 bar is performed on the mixture.

Thus, in a first preferred embodiment of the present process, the CO<sub>2</sub> counter-current liquid extraction may be performed on the mixture at 20-31°C so as to reinforce the sulphurous flavour note of the flavour extract, for example. The counter-current liquid extraction is thus carried out in the presence of liquid CO<sub>2</sub>.

In a second preferred embodiment of the process, the CO<sub>2</sub> counter-current liquid extraction may be performed on the mixture at a temperature greater than 31°C and less than 100°C, for example. The counter-current liquid extraction is thus carried out in the presence of super-critical CO<sub>2</sub>.

5-20 kg of CO<sub>2</sub>/kg of mixture may be used to perform the counter-current liquid extraction, for example. The extraction yield may be increased by increasing the quantity of CO<sub>2</sub>/kg of mixture, for example.

An extraction residue on the one hand and, on the other hand, the CO<sub>2</sub> loaded with flavours, which leaves at the top of the extractor column and which is then passed into the separator, are then recovered.

A separation is then performed on the CO<sub>2</sub> loaded with flavours so as to obtain a liquid flavour extract. This separation may be performed in a separator in which the temperature of the medium is lowered to ambient temperature  
5 and the pressure of the medium is lowered to a value of less than 40 bar so as to reduce the solubility of the flavours in the CO<sub>2</sub> and to enable a liquid flavour extract to be isolated, for example.

10 The liquid flavour extract thus obtained may be dried on a support, such as maltodextrin or a starch, so as to obtain the flavour extract in the form of a powder, for example.

The SFF 5860 extraction unit, marketed by Separex Chimie  
15 Fine, 5, rue Jean Monod-BP 9, F-54520 Champigneulle, may particularly be used for the extraction step and the separation step.

The CO<sub>2</sub> may be recompressed and reintroduced into the  
20 extractor for another extraction, for example.

In addition, the extraction residue, which still contains about 20% original flavours, as well as reaction products such as the phosphate compound and the colouring compounds  
25 which are not volatile, may be recovered, for example. This extraction residue may particularly be mixed with salt, soy sauce and/or yeast extract. This mixture may then be dried with a drying support, such as maltodextrin, so as to obtain a flavouring powder, for example.

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The present invention also provides the flavour extract obtained according to the process and its use for the preparation of a food composition. Indeed, the flavour extract made in this way, in liquid or dried form, may  
35 advantageously be used for the preparation of a food composition, for example. It may particularly be used for

the preparation of cooked dishes, sauces or stocks so as to modify their flavour note, for example.

5 The process for the extraction of flavours and the flavour extracts according to the present invention are described in more detail by means of the analytical tests below. The percentages are given by weight, unless otherwise specified.

10 Test 1: Extraction yield as a function of the quantity of CO<sub>2</sub>, the temperature and the pressure during the counter-current liquid extraction step

5 samples of flavour extract are prepared, obtained by  
15 implementing the process according to the present invention. The conditions of the counter-current liquid extraction step are varied (the quantity of CO<sub>2</sub>, the temperature and the pressure) for the preparation of these 5 samples, and their extraction yield is then compared.

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For this purpose, a mixture is prepared containing 8.37% cysteine, 8.37% thiamine, 1.90% xylose, 20.29% glucose, 10.38% diammonium phosphate, 2.15% onion extract, 1.32% sodium hydroxide and 47.36% water.

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This mixture is heated at 100°C for 4 h so as to carry out the Maillard reaction between the reducing function of the xylose and the glucose and the amine function of the thiamine, the cysteine and the diammonium phosphate and  
30 thus to develop the flavours of the mixture.

A CO<sub>2</sub> counter-current liquid extraction is then performed in a column extractor in which the mixture is fed in through the top of the column and the CO<sub>2</sub> is fed in through  
35 the bottom of the column.

For the different tests performed, the temperature and pressure conditions and the quantity of CO<sub>2</sub> used during

this CO<sub>2</sub> counter-current liquid extraction are indicated in Table I below.

The CO<sub>2</sub> loaded with flavours, which leaves at the top of the extraction column, is then recovered and subjected to a separation in a separator so as to remove the CO<sub>2</sub> and to isolate the liquid flavour extract. For this purpose, the solubility of the flavours in the CO<sub>2</sub> is reduced by lowering the temperature in the extractor to a temperature less than or equal to 20°C and lowering the pressure to a pressure of less than 40 bar.

The removed CO<sub>2</sub> is recompressed and reintroduced into the extractor for another extraction.

The flavour extract is dried using maltodextrin as the drying support.

For each test performed, the extraction yield is calculated by calculating the % of flavour extract isolated in relation to the quantity of starting mixture used.

The results of the different tests performed are shown in Table I below.

Table I

|       |                |                  | extraction yield (%) |      |       |
|-------|----------------|------------------|----------------------|------|-------|
| tests | pressure (bar) | temperature (°C) | a                    | b    | c     |
| 1     | 100            | 75               | 2.75                 | 4.90 | 7.25  |
| 2     | 70             | 20               | 1.15                 | 2.05 | 2.70  |
| 3     | 140            | 40               | 1.90                 | 3.90 | 5.40  |
| 4     | 280            | 77               | 5.15                 | 8.65 | 11.90 |
| 5     | 280            | 20               | 1.55                 | 2.65 | 3.75  |

key: (a) 5 kg CO<sub>2</sub>/kg of mixture  
(b) 10 kg CO<sub>2</sub>/kg of mixture  
(c) 15 kg CO<sub>2</sub>/kg of mixture

5 The results of these tests highlight the fact that the extraction yield of the flavours is better when a greater quantity of CO<sub>2</sub> is used. Indeed, for the 5 tests carried out, an increase in the extraction yield is observed depending on whether 5, 10 or 15 kg CO<sub>2</sub>/kg of mixture was  
10 used during the counter-current liquid extraction on the mixture.

In addition, the results of these tests highlight the fact that the extraction yield of the flavours is also better when the counter-current liquid extraction is performed on  
15 the mixture at elevated temperature. Indeed, tests 1, 3 and 4 give better yield results. For these tests, a counter-current liquid extraction was performed on the mixture with super-critical CO<sub>2</sub>.

The results of these tests also show that the flavour  
20 extraction yield is better when a CO<sub>2</sub> counter-current liquid extraction is performed on the mixture under high pressure.

Finally, the fact that the density of the CO<sub>2</sub> has no influence on the extraction yield of the flavours is  
25 highlighted.

Thus the extraction yield of the flavours is better when the CO<sub>2</sub> counter-current liquid extraction is performed on the mixture at elevated temperature, under high pressure  
30 and in the presence of a significant quantity of CO<sub>2</sub>.

#### Test 2: Sensory evaluation of the flavour extract

A sensory evaluation is carried out on samples containing a  
35 flavour extract obtained by implementing the process according to the present invention.

For this purpose, 5 flavour extracts are prepared in accordance with the process of the present invention. During the preparation of these 5 extracts, a counter-current liquid extraction is performed on the mixture, as  
5 described in test 1, in the presence of 15 kg of CO<sub>2</sub> and under different temperature and pressure conditions. The temperature and pressure conditions for the preparation of these 5 extracts are shown in Table II below.

- 10 5 samples are then made by mixing 0.2 g/l of each extract made in this way per litre of hot water, also containing 2 g/l salt and 1 g/l MSG.

At the same time, a reference sample is made by adding  
15 4 g/l of the mixture, as described in test 1, per litre of hot water, also containing 2 g/l salt and 1 g/l monosodium glutamate.

These 5 samples, together with the reference sample, are  
20 then tasted by 5 taste testers so as to describe and compare their flavour note.

The sensory evaluation of these 5 samples and that of the reference sample are shown in Table II below.

Table II

| sample    | extraction conditions |                  | flavour note                |
|-----------|-----------------------|------------------|-----------------------------|
|           | pressure (bar)        | temperature (°C) |                             |
| reference |                       |                  | meat, bitter, sulphur taste |
| 1         | 100                   | 75               | meat                        |
| 2         | 70                    | 20               | meat, sulphur taste         |
| 3         | 140                   | 40               | meat                        |
| 4         | 280                   | 77               | meat                        |
| 5         | 280                   | 20               | meat, sulphur taste         |

The sensory evaluation enables the fact that the 5 samples made in this way have a good meat flavour to be highlighted.

In addition, this evaluation highlights the fact that, when the counter-current liquid extraction is carried out at low temperature, i.e. with the aid of liquid CO<sub>2</sub>, a flavour extract having a sulphurous flavour note is isolated.

### Test 3: Evaluation of the extraction yield with the aid of flavour indicators

A sample of flavour extract, obtained by implementing the process according to the present invention, is prepared. To make this sample, a CO<sub>2</sub> counter-current liquid extraction is performed on the starting mixture at a pressure of 280 bar, a temperature of 77°C and in the presence of 15 kg of CO<sub>2</sub>.

The extraction yield is evaluated with the aid of flavour indicators. For this purpose, the concentration of 3 flavour indicators is measured in the starting mixture and in the flavour extract: 2,5-dimethyl-4-hydroxy-3(2H)-furanone, acetic acid and sulphurol, having similar

chemical properties and a similar polarity to those of the other relevant flavour compounds contained in the flavour extract.

- 5 The concentration of acetic acid is determined enzymatically using the 148261 enzymatic kit marketed by Boehringer Mannheim, CP 6343, Industrie Strasse 7, CH-Rotkreuz.
- 10 The concentration of sulphurol, which is a degradation product of thiamine, is determined by gas phase chromatography.

Finally the concentration of 2,5-dimethyl-4-hydroxy-3(2H)-furanone is determined by marking with an isotope, C<sub>13</sub>, by the method described by Sen et al. (Sen et al., Lebensm Wiss Technol 24: 364-369, 1991).

The concentration values of the 3 flavour indicators in the starting mixture and in the flavour extract, as well as the extraction yields of these 3 flavour indicators, are shown in Table III below.

Table III

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| flavour indicator | concentration in starting mixture (mg/kg) | concentration in flavour extract (mg/kg) | extraction yield (%) |
|-------------------|-------------------------------------------|------------------------------------------|----------------------|
| a                 | 76                                        | 308                                      | 48                   |
| b                 | 470                                       | 2800                                     | 71                   |
| c                 | 12400                                     | 7300                                     | 70                   |

key: (a) 2,5-dimethyl-4-hydroxy-3(2H)-furanone  
 (b) acetic acid  
 (c) sulphurol

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The results shown in Table III highlight the fact that the flavour indicators were, in fact, extracted when the extraction process according to the present invention was implemented. In addition, the fact is highlighted that even  
5 the highly polar flavours, which are very difficult to extract by distillation, are extracted during the extraction according to the present invention. Indeed, 2,5-dimethyl-4-hydroxy-3(2H)-furanone, which is a highly polar flavour, has also been extracted. An extraction yield value  
10 of 48% is obtained for this flavour indicator. It is therefore assumed that this flavour was probably partially decomposed during the extraction process according to the present invention.

15 The following examples are given as an illustration of an industrial use, in the foodstuffs sector, of the flavour extracts according to the present invention. In the examples below, the percentages are given by weight unless otherwise indicated.

20

#### Example 1

A beef stock is prepared using a flavour extract obtained by implementing the process according to the present  
25 invention.

For this purpose, a mixture is prepared containing 8.37% cysteine, 8.37% thiamine, 1.90% xylose, 20.29% glucose, 10.38% diammonium phosphate, 2.15% onion extract, 1.32%  
30 sodium hydroxide and 47.36% water.

This mixture is heated to 100°C for 4h so as to carry out the Maillard reaction between the reducing function of the xylose and the glucose, and the amine function of the  
35 thiamine, the cysteine and the diammonium phosphate and to develop the flavours of the mixture in this way.

A CO<sub>2</sub> counter-current liquid extraction is then performed in an extractor, in which the mixture is fed in through the top of the extraction column and the CO<sub>2</sub> is fed in through the bottom of the extraction column. This extraction is  
5 performed in the presence of 15 kg of CO<sub>2</sub> at a temperature of 77°C and a pressure of 280 bar.

The CO<sub>2</sub> loaded with flavours, which leaves at the top of the extraction column, is then recovered and subjected to  
10 separation in a separator so as to remove the CO<sub>2</sub> and isolate the liquid flavour extract. For this purpose the solubility of the flavours in the CO<sub>2</sub> is reduced by lowering the temperature to a temperature less than or equal to 20°C and the pressure to a pressure of less than 40  
15 bar in the extractor.

The extraction and the separation are performed in an SFF 5860 extraction unit, marketed by Separex Chimie Fine, F-54250 Champigneulle.

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The CO<sub>2</sub> which has been removed is recompressed and reintroduced into the extractor for another extraction.

The flavour extract is dried, using maltodextrin as the  
25 drying support.

At the same time a composition is prepared containing 373 g maltodextrin, 190 g salt, 100 g yeast extract, 90 g dextrose, 80 g starch, 65 g beef fat, 36 g sugar, 5 g  
30 caramel colour, 2.5 g onions, 2 g citric acid, 1 g pepper, 0.5 g garlic, 0.5 g thyme and 0.5 g marjoram.

0.8 g of the flavour extract, 1.2 g salt and 19 g of this composition are then added to 1 l of hot water.

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A reference is also made by mixing 19 g of the composition and 1.2 g salt into 1 l of hot water.

5 The flavour note of the beef stock prepared with the flavour extract obtained by implementing the process according to the present invention is then compared with that of the reference stock.

10 The stock made with the flavour extract has a very pronounced beef flavour whereas the reference stock has a very weak meat flavour.

#### Example 2

15 A chicken stock is prepared using a flavour extract obtained by implementing the process according to the present invention.

20 For this purpose, a flavour extract is prepared as described in Example 1.

At the same time, a composition is prepared containing 378 g maltodextrin, 190 g salt, 100 g yeast extract, 91 g dextrose, 80 g starch, 65 g chicken fat, 36 g sugar, 0.5 g  
25 caramel colour, 2.5 g turmeric, 2 g citric acid, 1 g pepper, 0.5 g garlic, 0.5 g rosemary and 0.5 g ginger.

0.8 g of the flavour extract, 19 g of this composition and 1.2 g salt are then added to 1 l of hot water.  
30

A reference stock is also prepared from 1 l of hot water into which 19 g of the composition and 1.2 g salt are mixed.

35 The flavour note of the chicken stock prepared with the flavour extract obtained by implementing the process

according to the present invention is then compared with that of the reference stock.

The stock made with the flavour extract has a very  
5 pronounced chicken flavour whereas the reference stock has a flavour bearing little resemblance to that of chicken.



## Claims

1. Process for the extraction of flavours in which:
  - a mixture is prepared containing, at least, a source of
  - 5 sulphur, a reducing sugar, a phosphate compound and water,
  - this mixture is heated at 50-180°C for 30 min to 5 h,
  - this mixture is subjected to a CO<sub>2</sub> counter-current liquid extraction at 70-350 bar,
  - a separation is then performed on the CO<sub>2</sub> loaded with
  - 10 flavours, so as to obtain a liquid flavour extract.
2. Process according to claim 1, in which a mixture is prepared containing, at least, 1-30% of a source of sulphur, 1-30% of a reducing sugar, 1-15% of a phosphate
- 15 compound and 25-55% water.
3. Process according to claim 1, in which the mixture contains glutathione, cysteine, thiamine and/or an inorganic sulphur as the source of sulphur.
- 20 4. Process according to claim 1, in which 5-20 kg of CO<sub>2</sub>/kg of mixture are used to perform the counter-current extraction.
- 25 5. Process according to claim 1, in which the CO<sub>2</sub> counter-current liquid extraction is performed on the mixture at 20-31°C.
6. Process according to claim 1, in which a CO<sub>2</sub> counter-
- 30 current liquid extraction is performed on the mixture at a temperature greater than 31°C and less than 100°C.
7. Process according to claim 1, in which the liquid flavour extract is dried so as to obtain a powdered flavour
- 35 extract.



8. Flavour extract obtained by implementing the process according to one of claims 1 to 7.

9. Use of a flavour extract according to claim 8 for the  
5 preparation of a food composition.