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Description

Field of the invention

[0001] The present invention relates to a method for manufacturing of a flavouring concentrate, a flavouring concentrate which can be manufactured by the method in accordance to the invention, which is free from disturbing aroma components with an OAV (odor activity value) ≥ 1 and which contains no solvent additives, and the use of the flavouring concentrates.

Prior Art

[0002] Flavourings play an important role in numerous industrial methods. On the one hand, these are methods that are aimed solely at the recovery of flavourings in the form of essences from fruits, vegetables, spices, flowers and the like, and on the other hand, methods for manufacturing or refinement of foodstuff and luxury food in which the preservation of natural flavourings, such as fragrances and aromas, determines the quality of the products or makes these products edible in the first place.

[0003] The flavour originating from a specific source, e.g. a fruit, is not a chemically uniform substance, but is composed of a plurality of different chemical components which only in their entirety produce the sensory result of the natural flavour of a foodstuff. Experience has shown that, when handling media containing flavourings or when processing foodstuff, parts of the natural mixture may be lost or degraded, thereby reducing or even destroying the natural content, resulting in an "off-flavour". This is particularly the case when solutions containing flavourings are concentrated.

[0004] During the manufacture of vegetable and fruit juice as well as during the extraction of foodstuff, industrially significant quantities of odor- and taste-intensive aqueous solutions are formed which, when concentrated and added as a concentrate, re-aromatize the same foodstuff or aromatize other foods. For example, an aromatic vapour condensate is recovered during the recovery of fruit or vegetable juice concentrate. Freshly squeezed fruit juices from drupaceous, pomaceous and berry fruits as well as juices from citrus and tropical fruits are concentrated by evaporation and thus preserved. During the evaporation method, readily volatile flavourings are removed from the thin

juice in the form of a vapour condensate, which is then concentrated. Before bottling, the fruit or vegetable juice concentrate is diluted and the previously separated aroma concentrate is added to the fruit or vegetable juice to reconstitute the aroma. Fruit or vegetable juice concentrate that has only one sixth to one eighth of its original volume saves storage and transportation costs. Another important application for evaporation technology in the juice industry is the concentration of extracts from a wide variety of starting materials. For example, the juice residues and oils are extracted from the pulp and peel of citrus fruits, concentrated by evaporation, then separated and further processed. Evaporation plants are also used in other areas of the beverage industry, for example in the brewing industry to concentrate malt extract, brewer's yeast, yeast extract and hop extract.

[0005] Reconstituted fruit or vegetable juices must be compared to the taste of freshly squeezed juices. Due to the increased consumer interest in authentic taste experiences and health-conscious nutrition, existing methods for concentrating aqueous solutions are only of limited use.

[0006] For decades, aqueous solutions of foodstuff have been concentrated with odor- and taste-active substances by means of distillation methods. Despite the application of gentle process parameters such as the use of vacuum to reduce the vapour pressure of the volatile components, disturbing aroma components can be formed and desired aroma components can be degraded even at temperatures as low as 30 °C. For example, distillation can result in cooking notes due to the thermal stress on the aqueous product that are not considered to be an authentic taste experience. When strawberry vapour condensates are concentrated, cooking notes are formed that remind the consumer of jam rather than of eating fresh strawberries.

[0007] A more recent method relates to the recovery of an aroma concentrate by adsorption of aromas on solid phase material, as described, for example, in EP 2 075 320 A1. In the adsorptive concentration of aqueous solutions, as described, for example, in EP 0 082 284 A1, the aqueous solution is passed through a bed of a porous sorbent surface-modified with organic residues and the bed is then eluted with a small amount of an organic solvent compared to the aqueous solution. However, this method requires the use of solvents for desorption, such as methanol, which is considered toxicologically questionable. A further disadvantage of these prior art methods is that the

aroma profile can shift due to the selectivity of the adsorbent and the solvent for elution/desorption.

[0008] The solvent-free and non-thermal concentration of odor- and taste-intensive aqueous solutions has therefore been a concern of the food industry for some time. In particular, work is being carried out on membrane-based methods to separate water and enrich value-adding components without using solvents. For example, a combination of ultrafiltration and reverse osmosis was used to remove water from aqueous solutions used in citrus processing (Braddock, RJ "Ultrafiltration and Reverse Osmosis Recovery of Limonene from Citrus Processing Waste Streams", Journal of Food Science, 1982, 47(3), 946 - 948). Hollow fiber flat membranes made of classic materials such as cellulose acetate, polysulfone and polytetrafluoroethylene were used. However, reverse osmosis typically uses pressures of 10 to over 100 bar, which are known to accelerate degradation reactions, which negatively impacts the sensory profile of the resulting concentrates.

[0009] In WO 2017 080852 A1 a membrane method is described for concentrating aqueous coffee extracts which are more reminiscent of freshly brewed coffee in sensory terms than instant coffees conventionally recovered via evaporation, spray drying or freeze drying. In addition to water, other substances with a low molecular weight, i.e. less than 1 kDa, such as small sensory active substances, penetrate through the membrane and are thereby lost. A high loss of flavourings, which usually have a molecular weight of 100 - 300 Da, is accepted in order to achieve a high permeation flux of 8.3 $l/m^2/h$ at 10 - 20 bar and thus rapid dewatering. The examples show that membranes made of conventional materials such as cellulose acetate, polysulfone, polyamide, polytetrafluoroethylene, etc. are unsuitable for concentrating aqueous solutions in a stable and cost-effective process without changing the aroma profile. Either the low permeability means that the flux is too low to operate the process economically, or the transmembrane pressure is so high that the aroma is degraded, or the good permeability results in poor selectivity, whereby the flavourings are lost with the water.

[0010] Recently, biomimetic membranes containing aquaporins have come onto the market. Aquaporins are proteins that are found in cell membranes and are widespread in living organisms and which, under certain conditions, form a water channel to allow water molecules to pass through and retain dissolved inorganic and organic substances.

[0011] As described byTang et al. 'Desalination by biomimetic aquaporin membranes: Review of status and prospects", Desalination 308, 2013, in nature aquaporins exhibit a selective permeability, for example, to water, glycerol or salts, although the mechanism of selectivity in many aspects is still unclear. To make matters worse, industrial processing into membranes, such as recombinant manufacture of AQPs and embedding them in the membrane polymer, as well as process instability, degrades the performance of the membrane. It does not contain any teaching on the filterability of aqueous solutions containing organic flavourings and odors.

[0012] In WO 2014 108827 A1 it is described, for example, that aquaporin membranes can be used for the dialysis of blood, in particular for the separation of human metabolites such as urea, p-cresyl sulfate and peptides up to a molecular weight of 692 Da. However, there is no teaching on the targeted selective enrichment or depletion of organic flavourings and odors for the manufacture of a taste or aroma-imparting preparation.

[0013] US 2016 016127 A1 discloses the use of aquaporin membranes for concentrating liquid foodstuff in order to gently remove water from solutions of, for example, small organic compounds. The membranes used are thin-film composite membranes.

[0014] US 2017 259217 A1 describes the aquaporin membranes and their possible application in biotechnology as well as the food and beverage sector, e.g. for the separation of bacteria, concentration of whey, fruit or vegetable juice, etc. The aquaporins are stabilized by β-proteins.

[0015] US 2012 152841 A1 discloses a method for extracting water from aqueous solutions such as, for example, wine, fruit or vegetable juices, milk, etc. using aquaporin-containing membranes and forward osmosis. A liquid membrane matrix is used.

[0016] US 4 952 751 discloses a method for concentrating fruit juice distillates by means of pervaporation membranes.

[0017] DK 2017 70753 A1 describes the concentration of coffee extract by means of aquaporin membranes.

[0018] The aim of the present invention was to develop a method by which a flavouring concentrate can be manufactured from an odor- and taste-active aqueous solution from a foodstuff

without any loss of these value-adding flavourings and odors and the sensory profile of the original sample can be retained.

Description of the invention

[0019] The present problem is solved by the subject matter of the independent claims. Preferred configurations emerge from the wording of the dependent claims and the following description.

[0020] A first subject of the present invention relates to a method for manufacturing a flavouring concentrate, comprising the following steps:

- preparing an aqueous starting solution comprising a flavouring from a foodstuff, wherein the aqueous starting solution beinga vapour condensate from fruits or vegetables,
- concentrating the aqueous starting solution by osmosis usinga semipermeable biomimetic membrane, and
- formingof a flavouring concentrate as retentate,

wherein the semipermeable biomimetic membrane comprises:

vesicles of liposomes or polymersomes containing at least one aquaporin protein;

a thin film composite matrix containing embedded vesicles; and

a carrier layer;

wherein the method is performed at a temperature in arange of 10 to 40 °C; and

wherein the aqueous starting solution is concentrated by a factor of 20 to 1,000.

[0021] A further subject matter of the present invention relates to a flavouring concentrate obtainable by a method in accordance to the invention exhibiting no off-flavour and being free of disturbing aroma components, especially being free of disturbing aroma components with an OAV (odor activity value) ≥ 1.

[0022] Furthermore, subject matter of the present invention relates to a flavouring concentrate as described above, which contains no solvent additives.

[0023] A further aspect of the present invention relates to the use of the flavouring concentrate in accordance to the invention for flavouring or for reconstituting the flavour of foodstuff, beverages, semifinished products, oral hygiene products, cosmetic or pharmaceutical products.

Description of the figures

[0024]

Figure 1 is a schematic representation of the structure of an aquaporin membrane used in accordance to the invention

Figure 2 is a schematic representation of the principle of forward osmosis

Figure 3 is a schematic representation of the method using the example of a coffee extract (not part of the present invention).

Figures 4 a and 4 b are a chromatogram comparison of a tea extract (water phase) and the diluted concentrate of the same tea extract (concentrating tea extract is not part of the present invention).

Figure 5 is a representation of a sensory profiling of water phase and diluted concentrate from the aquaporin membrane filtration of yellow peach (concentrating aqueous peach extracts/juices without obtaining a vapour condensate in the method is not part of the present invention).

Figure 6 is a representation of a sensory profiling of water phase and diluted concentrate from aquaporin membrane filtration of strawberry

Detailed description of the invention

[0025] In the method in accordance to the invention for manufacturing a flavouring concentrate, an aqueous starting solution from a foodstuff is prepared in a first step, wherein the aqueous starting solution is a vapour condensate from fruit or vegetables.

[0026] Foodstuff is any substance or product intended to be ingested by humans in a processed, partially processed or unprocessed state. Foodstuff also includes beverages and all substances, including water, that are intentionally added to the foodstuff during its manufacture or treatment or processing. Luxury foods are also classified as foodstuff. Strictly speaking, luxury foods are foods that are not consumed primarily for their nutritional value or to satiety, but rather for their stimulating effect or taste. Examples of luxury items include, for example, coffee, tea, cocoa and spices. In the description, vegetable or fungal raw materials that are not suitable for direct consumption but are suitable and typically needed for the manufacture of flavouring preparations are also included in foodstuff.

[0027] The term aqueous starting solution of a foodstuff is understood to mean either a foodstuff which is naturally present in aqueous form, such as, for example, milk, an aqueous solution of a foodstuff obtained by adding water to a foodstuff, an aqueous extract of a foodstuff obtained by adding water to a foodstuff, such as, for example, edible plants such as, for example, tea or coffee, or a direct juice from fruit or vegetables. An aqueous starting solution of a food also includes typical aqueous solutions that arise during the treatment or processing of a foodstuff, for example an aromatic vapour condensate that arises during the manufacture of juice concentrates from fruit or vegetables by means of juice evaporation.

[0028] To produce a direct juice, ripe fruit or vegetables are first washed, sorted and crushed in a mill or a milling machine to form the so-called mash. Optionally, the mash is depectinized with special enzymes such as, for example, pectinases, cellulases and hemicellulases before further processing in order to obtain a better juice yield. The juice is then extracted in a press at a pressure of 5 to 20 bar or by centrifugation. The raw juice thus obtained is depectinized, clarified and filtered before further processing into concentrate.

[0029] Alternatively, the raw juice can be further processed into concentrate. Before further processing into concentrate, the juice must be depectinized, clarified and filtered. In addition to freeze concentration, the evaporation method is used to manufacture juice concentrate. Concentration by evaporation produces a vapour condensate which contains the flavourings and aromas of the starting product and is used as an aqueous starting solution in the method in accordance to the invention.

[0030] In the method in accordance to the invention, a vapour condensate from fruit or vegetables is used as an aqueous starting solution.

[0031] In the second step of the method in accordance to the invention, the above-described aqueous starting solution from a foodstuff is concentrated by osmosis with a semipermeable biomimetic membrane.

[0032] In the natural sciences, osmosis is the directed flow of molecular particles through a selective or semi-permeable separation layer or membrane. In particular, osmosis is described as the spontaneous passage of water or another solvent through a semipermeable membrane that is

permeable to the solvent but not to the substances dissolved in it.

[0033] Osmosis in the method in accordance to the invention can be carried out as forward osmosis or as reverse osmosis at low pressure. Forward osmosis is preferred in the method in accordance to the invention.

[0034] Reverse osmosis is a physical method for concentrating substances dissolved in liquids by using pressure to reverse the natural osmosis process. The operating principle is that the medium in which the concentration of a certain substance is to be reduced is separated from the medium in which the concentration is to be increased by a semipermeable membrane. This medium is subjected to a pressure that must be higher than the pressure created by the osmotic desire to equalize the concentration. This allows the molecules of the solvent to migrate against their "natural" osmotic propagation direction. The method pushes them into the compartment in which dissolved substances are less concentrated.

[0035] Drinking water has an osmotic pressure of less than 2 bar, the pressure applied for reverse osmosis of drinking water is 3 to 30 bar, depending on the membrane used and the system configuration. The osmotic membrane, which only allows the carrier liquid (solvent) to pass through and retains the dissolved substances (solutes), must be able to withstand these high pressures. If the pressure difference more than compensates for the osmotic gradient, the solvent molecules fit through the membrane just like through a filter, while the "impurity molecules" are retained. In contrast to a classic membrane filter, osmosis membranes do not have continuous pores. Rather, the ions and molecules migrate through the membrane by diffusing through the membrane material.

[0036] The osmotic pressure increases with increasing concentration difference. If the osmotic pressure becomes equal to the applied pressure, the process stops. There is then an osmotic equilibrium. A steady drainage of the concentrate can prevent this.

[0037] Forward osmosis is an osmotic method that uses a semipermeable membrane to effect the separation of water from a solution containing solutes. The driving force for this separation is an osmotic pressure gradient between a solution of high concentration also known as the "draw solution", and a solution of lower concentration, also known as the "feed solution". An osmotic

pressure gradient is used to induce a flow of water through the membrane into the high concentration solution, effectively concentrating the low concentration solution.

[0038] In both reverse osmosis and forward osmosis, water is extracted from the vapour condensate from fruit or vegetables by means of a biomimetic membrane, which is described in more detail below, in the method in accordance to the invention, so that the aqueous starting solution is concentrated.

[0039] Preferably, in the method in accordance to the invention, the concentration of the vapour condensate from fruit or vegetables is carried out using a biomimetic membrane as forward osmosis.

[0040] Even more preferably, the concentration of the vapour condensate from fruit or vegetables is carried out with a biomimetic membrane as forward osmosis against an osmosis solution. For this purpose, the osmotically active solution (draw solution) is run in countercurrent with the vapour condensate from fruit or vegetables, as a result of which water is removed from said vapour condensate.

[0041] Typically, forward osmosis in the method in accordance to the invention utilizes osmosis solutions which comprise one or more chemical components from the following group: salts consisting of a cation selected from the group consisting of ammonium, sodium, potassium, calcium and magnesium; and an anion selected from the group consisting of acetate, chloride, citrate, biarbonate, formate, lactate, malate, propionate, sulfate, succinate and tartrate. In addition, salts of amino acids and simple sugars such as glucose, fructose and sucrose are utilized.

[0042] Preferably, osmotically active solutions utilized are those comprising NaCl, $MgCl_2$, KCl, K-lactate, NH_4HCO_3 or sucrose. In the method in accordance to the invention, osmosis solutions comprising NaCl, K-lactate or sucrose are most preferred. Particularly preferred is an osmosis solution comprising NaCl.

[0043] The osmosis solution utilized in the method in accordance to the invention has an starting concentration of the chemical components in the range of 0.05 to 4 M, preferably in the range of 0.5 to 3 M.

[0044] The method in accordance to the invention is characterised in that in the step for

concentrating the vapour condensate from fruit or vegetables by means of one of the previously described osmosis methods, a semipermeable biomimetic membrane according to the claims is used. These biomimetic membranes are preferably membranes of the type described by Joachim Habel et al. in "Membranes" Aquaporin-based biomimetic polymeric mambrances: Approaches and challenges"; 2015, 5, 307 - 351; ISSM 2077-0375", in particular pages 312 - 319. The disclosure of the aquaporin-based biomimetic polymer membranes described therein is fully incorporated into the present application by specifically referring to the membranes disclosed therein.

[0045] Such biomimetic membranes, which are permeated with aquaporins, are already commercially available. They are manufactured by Aquaporin A/S, Denmark, and sold under the trade name Aquaporin Inside R. Such aquaporin membranes are utilized, for example, in blood dialysis or seawater desalination.

[0046] The semipermeable biomimetic aquaporin membranes used in the method in accordance to the invention are characterised in that they comprise

- vesicles of liposomes or polymersomes containing at least one aquaporin protein;
- a thin-film composite matrix conatining embedded vesicles; and
- a carrier layer

.

[0047] The structure of the semipermeable biomimetic aquaporin membrane described above is shown in Figure 1.

[0048] As can be seen from Figure 1, the aquaporin membrane in the active layer comprises vesicles into which aquaporins are incorporated. Aquaporins are proteins that are found in cell membranes and are widespread in living organisms and which, under certain conditions, form a water channel to allow water molecules to pass through and retain dissolved inorganic and organic substances in the cell. They are therefore also called water channels. In the aquaporin membrane, the aquaporins form water channels that preferentially allow water molecules to pass through, while ideally blocking the passage of other molecules, regardless of their size, molecular weight and chemical structure.

[0049] The vesicle matrix consists of at least one liposome or at least one polymersome. The liposome comprises: lipids such as DPhPC, DOPC, mixed soybean lipids, asolectin or mixed lipids from E. coli. The polymersome comprises: triblock copolymers of the hydrophilic-hydrophobic-hydrophilic type A-B-A, A-B-C, C-B-A, wherein A stands for PMOXA, B stands for PDMS and C stands for PEO, diblock copolymers of the hydrophilic-hydrophobic type A-B, wherein A stands for PB and B stands for PEO, or combinations thereof.

[0050] In accordance to the invention the aquaporins preferably comprise at least one protein selected from the group consisting of AQPO, AqpZ, SoPIP2, AQP10 and their isoforms. AQPO is particularly preferred.

[0051] As can be further seen from Figure 1, the vesicles containing the aquaporins are incorporated on top of a thin-film composite matrix. The thin-film composite matrix is manufactured by polymerizing an aqueous solution of an amine with a solution of an acid chloride in an organic solvent. The aquaporin water channel vesicles are incorporated into this aqueous solution.

[0052] The thin film composite matrix is in turn applied to a porous carrier layer. The carrier layer is preferably a polyethersulfone.

[0053] The manufacture of these semipermeable biomimetic membranes, further compositions and physical and chemical properties are described, for example, in WO 2014108827 A1, in particular pages 6 to 33.

[0054] The following Table 1 lists exemplary compositions of such aquaporin membranes:

Table 1: Composition of aquaporin membranes

Polymer	Membrane protein	Permeability
$PMOXA_{20}$-$PDMS_{41}$-$PMOXA_{20}$	AQP0	H_2O
$PMOXA_{12}$-$PDMS_{54}$-$PMOXA_{12}$	AqpZ	H_2O
$PMOXA_{8}$-$PDMS_{55}$-$PMOXA_{8}$	AqpZ	H_2O
$PMOXA_{12}$-$PDMS_{55}$-$PMOXA_{12}$	AQP0	H_2O

Polymer	Membrane protein	Permeability
$PMOXA_{12}$-$PDMS_{55}$-$PMOXA_{12}$	AqpZ	H_2O
$PMOXA_{8}$-$PDMS_{60}$-$PMOXA_{8}$	AqpZ	H_2O
$PMOXA_{15}$-$PDMS_{68}$-$PMOXA_{15}$	AqpZ	H_2O
$PMOXA_{20}$-$PDMS_{75}$-$PMOXA_{20}$	AqpZ	H_2O
$PMOXA_{110}$-$PDMS_{40}$-PEO_{25}	AQP0	H_2O
$PMOXA_{45}$-$PDMS_{40}$-$PMOXA_{67}$	AQP0	H_2O
PB_{12}-PEO_{10}	AQP0	H_2O
PB_{12}-PEO_{10}	AqpZ	H_2O
PB_{12}-PEO_{10}	SoPIP2; 1	H_2O
PB_{22}-PEO_{14}	AQP0	H_2O
PB_{22}-PEO_{23}	AqpZ	H_2O
PB_{22}-PEO_{23}	SoPIP2; 1	H_2O
PB_{29}-PEO_{16}	AQP10	H_2O
PB_{35}-PEO_{14}	AqpZ	H_2O
PB_{35}-PEO_{14}	SoPIP2;1	H_2O
PB_{43}-PEO_{32}	AQP10	H_2O
PB_{46}-PEO_{30}	AqpZ	H_2O
PB_{46}-PEO_{32}	AQP10	H_2O
PB_{92}-PEO_{78}	AQP10	H_2O

[0055] The mechanism of concentrating a vapour condensate from fruit or vegetables with a semipermeable biomimetic aquaporin membrane by forward osmosis in accordance to the invention will now be described in more detail with reference to Figure 2. In forward osmosis, the aqueous

starting solution, which contains, for example, value-adding flavourings and aromas, is spatially separated from the osmosis solution by the semi-permeable biomimetic aquaporin membrane. The concentration of the osmosis solution is greater than the concentration of the starting solution. Due to the concentration gradient and the semipermeable biomimetic aquaporin membrane, water molecules preferentially migrate from the starting solution through the aquaporin membrane to the osmosis solution without the application of pressure. The value-adding flavourings and aromas cannot pass through the semipermeable biomimetic aquaporin membrane and thus remain in the retentate, regardless of their size, molecular weight and chemical structure. Due to the migration of the water molecules, water is removed from the starting solution, i.e. a flavouring concentrate is created as a retentate in which the value-adding flavourings and aromas are concentrated. The osmosis solution, on the other hand, is diluted by the influx of water; a permeate is created. To keep the osmosis system in balance, on the one hand the foodstuff concentrate is continuously removed from the system and on the other hand fresh osmosis solution is continuously fed into the system.

[0056] Figure 3 shows an example of the concentration of a coffee extract (not part of the present invention). The starting product is an aqueous coffee extract. Water is removed from this coffee extract by forward osmosis against a sugar solution with high osmotic potential. During the osmosis method, the water content of the coffee extract decreases and a concentrated coffee extract is created as a retentate, which is withdrawn from the system and further processed in a subsequent step, for example by freeze-drying. Due to the selectivity of the aquaporin membrane for water, only water molecules diffuse through the aquaporin membrane towards the sugar solution. The value-adding flavourings and aromas remain in the coffee extract and are enriched. The sugar solution is diluted by the addition of water.

[0057] The biomimetic membrane used is characterised by a high selectivity, which ensures a targeted separation of water while simultaneously retaining the value-adding flavourings and aromas. As a result, the value-adding flavourings and aromas remain completely in the water phase (retentate) and do not migrate across the biomimetic membrane to the osmosis solution. In addition, the biomimetic membrane used enables a high flux and thus a short residence time of the starting solution

with the sensitive flavourings and aromas in the system, which prevents degradation of the flavourings and aromas, for example through oxidation.

[0058] The semipermeable biomimetic aquaporin membranes used in the method in accordance to the invention are utilized either as flat hollow fiber modules or as spiral modules. In a spiral module, the aquaporin membrane is wound in a spiral shape, which increases the surface area of the membrane and improves the efficiency of the osmosis procedure. Preferably, hollow fiber modules are used in the method in accordance to the invention.

[0059] The osmosis method in accordance to the invention is carried out at a pH of 2 to 10. Furthermore, the osmosis method in accordance to the invention is carried out at a temperature which has no negative effect on the ingredients, for example flavourings and aromas, of the aqueous starting solution. The method in accordance to the invention is carried out at a temperature in the range of 10 to 40 °C, preferably at a temperature of about 25 °C.

[0060] Preferably, the forward osmosis is carried out at a flux, i.e. the amount of water flowing through the membrane, of 4 to 30 l/m^2h, preferably of 10 to 20 l/m^2h. Forward osmosis is particularly preferably carried out at a flux of at least 12 l/m^2h. It is preferable to work without pressure.

[0061] If the method in accordance to the invention is carried out as reverse osmosis, a working pressure of 2 to 15 bar is applied to the aqueous starting solution.

[0062] In a further preferred embodiment, the osmosis method in accordance to the invention is carried out continuously. For this purpose, the retentate recovered by membrane filtration, i.e. the foodstuff concentrate, is removed from the system and in return the permeate is also removed and replaced with fresh osmosis solution.

[0063] The method in accordance to the invention achieves a concentration of the aqueous starting solution by a factor of 20 to 1,000, preferably at least by a factor of 100.

[0064] The aqueous starting solution prepared in the first step of the method can be concentrated in a preceding step by conventional method steps such as distillation, adsorption, freeze-drying, membrane filtration or by reverse osmosis or forward osmosis by means of a membrane.

[0065] Likewise, the retentate or foodstuff concentrate obtained by osmosis methods can be

further concentrated in a subsequent step by conventional method steps such as distillation, adsorption, freeze-drying, membrane filtration or by reverse osmosis or forward osmosis using a membrane.

[0066] Surprisingly, it has been found that the method in accordance to the invention for concentrating a vapour condensate from fruit or vegetables by osmosis using the biomimetic membrane described above leads to flavouring concentrates whose sensory profile is identical to the sensory profile of the starting product, i.e. a flavouring concentrate is obtained which has an authentic sensory profile.

[0067] The analysis of the sensorily value-adding components showed that both volatile odorous and non-volatile aromas can be found in the concentrate in the same proportions as they were present in the starting solution.

[0068] Figure 4 shows the comparison by liquid chromatography of non-volatile aromas of an aqueous tea extract and the concentrate recovered by osmosis by means of a semipermeable aquaporin membrane of the aqueous tea extract (not in accordance to the invention). For this purpose, 600 g black tea was extracted with water 20 times its weight for two hours. The resulting extract was filtered to obtain the starting solution. The starting solution was run on a 0.6 m^2 aquaporin membrane module by means of forward osmosis countercurrent to an osmosis solution (draw solution). The extract was concentrated from 1.3°Brix to 33 °Brix at an initial flux of 13.5 l/m^2h. The comparison in Figure 3 shows that small aroma molecules such as, for example, simple sugars, quinic acid and gallic acid remain in the retentate and are not lost as in adsorptive methods or degraded in distillative methods. The area ratios of the individual value-adding components are almost identical. Caffeine was fully recovered in the concentrate at > 99%.

[0069] Water phases from fruit processing such as, for example, from citrus fruits, berry fruits, pomaceous fruits, drupaceous fruits undergo a change in the sensory profile during the distillation process, which is often described by, for example, "cooked note", "less fresh", "potato-like", "fatty" and "metallic". Surprisingly, it was found that, for example, water phases of yellow peach and strawberry are not subject to such a change in profile after concentration with a biomimetic

membrane.

[0070] The sensory comparison based on a sensory profile according to DIN10967-1-1999, as shown in Figure 5, shows that the peach-water phase and the concentrate, which was diluted to the initial concentration, smell almost identical. Only a slightly less strong fruity, estery and ripe note was perceived. The peach-like and fleshy flavours, which are mainly caused by sulfur-containing components and are changed by thermal and adsorptive methods, are just as pronounced in the concentrate as in the water phase.

[0071] To create a sensory profile, the descriptive terms (descriptors) are first collected in the panel, wherein the term lists are structured, similar terms are summarized and hedonic attributes are eliminated. The intensity of the descriptors is assessed on a scale of 1 - 10 by at least ten trained assessors. The samples are coded and tasted in a sensory room in a randomized sequence, excluding disturbing influences such as color, noise and foreign odors. The final result is determined by summing the individual results and then calculating the arithmetic mean and is presented graphically in the form of a radar chart.

Table 2: Flavourings in the water phase and the concentrate of yellow peach

	Water phase [mg/kg]	Concentrate [mg/kg]	Recovery [%]	Molecular weight [Da]
Ethanol	379.9	10295.5	48.7	46
Ethyl acetate	41.9	621.2	90.6	88
Hexanol	26.4	572.1	99.1	102
cis-2-Hexenol	19.6	408.4	98.1	100
trans-2-Hexenal	9.9	128.0	98.8	98
cis-3-Hexenol	4.7	101.1	>99.0	100
gamma-Decalactone	2.3	33.8	>99.0	170
Hexanal	1.7	31.0	>99.0	100

	Water phase [mg/kg]	Concentrate [mg/kg]	Recovery [%]	Molecular weight [Da]
3-Methyl-1-butanol	1.0	18.2	>99.0	88
1,3-Pentenol	0.3	7.7	>99.0	86

[0072] Table 2 shows the contents of value-adding flavourings in the water phase and the corresponding concentrate of the yellow peach. The aqueous starting solution/water phase was recovered by distillation from a vapour condensate, so that flavourings were already enriched. Further concentration was carried out using the biomimetic aquaporin membrane and shows that at a high water flux of 14 l/m^2/h, very small molecules are recovered with high yield. The recovery rate of the flavourings ranged from 90 to > 99%. Thermal degradation products of value-adding components could not be detected. Instead, it turns out that ethanol is removed, which benefits the resulting flavouring concentrate. The recovery rate for ethanol was about 50%.

[0073] Strawberry is a particularly suitable system for investigating negative effects of the processing process on the sensory profile, since thermal and pressure-driven impact creates a cooked note that reminds the consumer of "cooked jam". Concentration by means of a biomimetic aquaporin membrane has surprisingly shown that concentration of the aroma components is possible without negative impact on the sensory profile with almost complete recovery of the aroma components and a high flow rate of the membrane.

Table 3: Flavourings in the water phase and the concentrate of strawberry

	Water phase [mg/kg]	Concentrate [mg/kg]	Recovery [%]	Molecular weight [Da]
Ethyl acetate	43.1	566.8	95.0	88
trans-2-Hexenal	37.8	540.8	99.4	98
Hexanol	28.3	543.6	>99.0	102
Methyl butanoate	18.7	245.5	99.2	102

	Water phase [mg/kg]	Concentrate [mg/kg]	Recovery [%]	Molecular weight [Da]
trans-2-Hexenol	17.3	332.6	>99.0	100
gamma-Decalactone	12.0	127.3	>99.0	170
Ethyl butanoate	8.4	94.3	>99.0	116
Hexanoic acid	6.3	84.3	>99.0	116
Hexanal	4.7	73.8	>99.0	100
Methyl hexanoate	4.1	31.9	>99.0	130
2-Methylbutyric acid	4.0	25.0	>99.0	102
Ethyl hexanoate	1.7	7.1	>99.0	144
Methyl propionate	1.3	14.2	>99.0	88
Butyl acetate	1.1	13.2	>99.0	116
Propyl acetate	0.7	7.2	>99.0	102
Methyl ethyl acetate	0.6	11.0	>99.0	102

[0074] Table 3 shows the contents of the aroma components in the water phase and the corresponding concentrate of the strawberry. An enrichment factor of 19 was achieved in a short time at high water flux. The recovery rate of the flavourings ranged from 95 to > 99%.

[0075] The sensory comparison in Figure 6 shows that the aqueous starting solution/water phase and the concentrate, which was diluted to the initial concentration, smell almost identical. Only a slightly less strong fruity-estery note with a slightly increased impression of a buttery note was perceived.

[0076] The concentration of vegetable juices such as tomato, cucumber, carrot, celery, beetroot and onion using conventional processes known from the prior art also leads to an undesirable change in the sensory profile. Surprisingly, it was found that the method in accordance to the invention leads to a concentration without the aforementioned negative impacts. For example, 20 kg cucumber juice

was recovered from 75 peeled and pureed cucumbers after decanting, centrifugation and bag filtration at a mesh size of 100 µm and concentrated by reverse osmosis or the osmosis method described herein. For this purpose, 10 kg the cucumber water were first dewatered at room temperature and 10 bar using a conventional 0.37 m^2 reverse osmosis membrane module from General Electric for 2.5 hours at a pressure of 30 bar and for a further 3.5 hours at an initial flow of 4 $l/m^2/h$ until a residual amount of 1640 g was reached. The other half (10 kg) of cucumber water was run with a 0.6 m^2aquaporin membrane module against 2 kg potassium lactate solution as draw solution with a concentration of 60 °Brix at 300 ml/min for 5 hours. The product weight was 1,091 g. The membrane flux was initially 12 $l/m^2/h$. The osmosis method described herein using the biomimetic aquaporin membrane showed a 3-fold higher initial membrane flux and concentrated the cucumber water significantly more in the same time than the conventional reverse osmosis used. (The concentration of juices and extracts from fruits or vegetables using aquaporin membranes without obtaining a vapour condensate in the method is not part of the present invention.)

Table 4: Flavourings in cucumber water and the concentrate from reverse osmosis and aquaporin membrane osmosis

	Cucumber water [mg/kg]	Reverse osmosis [mg/kg]	AQP membrane osmosis [mg/kg]
6-(Z)-Nonenol	0.600	2.823	0.148
2,6-(E,Z)-Nonadienol	0.521	0.800	0.104
Hexanol	0.210	1.085	0.138
2,6-(E,Z)-Nonadienal	0.078	0.886	2.610
2-(E)-Nonenal	0.039	0.428	0.715
8,11,14-(Z,Z,Z)-Heptadecatrienal	0.025	0.247	0.769
2-(E)-Hexenal	0.007	0.078	0.296
Hexenal	0.003	0.074	0.773

	Cucumber water [mg/kg]	Reverse osmosis [mg/kg]	AQP membrane osmosis [mg/kg]
2,6-(E,E)-Nonadienal	<0.002	0.035	0.042
2-(E)-Octenal	<0.002	<0.002	0.039
2,4-(E,E)-Decadienal	<0.002	<0.002	0.061
8,11-(Z,Z)-Heptadecadienal	<0.002	<0.002	0.604

[0077] Reverse osmosis is considered industrially to be the standard method for non-thermal and thus gentle concentration by dewatering the juice/aqueous starting solution used.

[0078] However, the comparison listed in Table 4 shows that the value-adding aldehydes in cucumber such as 2,6-(E,Z)-nonadienal and 2-(E)-hexenal are degraded by reverse osmosis and alcohols are formed, whereas in osmosis by means of the biomimetic aquaporin membrane the aldehydes dominate in terms of quantity and are thus retained. The experiment surprisingly showed that the concentrate from the method described herein had an intense odor of freshly cut cucumber and the already brownish colored concentrate from the reverse osmosis smelled greenish, balsamic, like cooked cucumber. In addition, the draw solution from osmosis with the biomimetic aquaporin membrane was odorless, while the permeate from reverse osmosis had slight green notes. Further osmosis experiments with classical membranes known from the prior art showed, as with reverse osmosis, that losses of aroma components occur with classical membranes. The biomimetic membrane used according to the method described herein therefore shows a high selectivity, which enables a targeted separation of water while retaining the value-adding aroma components and at the same time a high flux and thus a short residence time of the sensitive aroma components in the system.

[0079] Surprisingly, no degradation products or disturbing degradation products of the value-adding components were detected in the described experiments and sensorily disturbing components were also not detected. The disturbing components are in particular those compounds which are formed in prior-art distillative concentration methods, for example by thermal degradation, by pH shift, oxidation or by chemical rearrangement reactions, and which impact the sensory profile of the

flavouring concentrate.

[0080] Flavourings which, depending on the foodstuff and concentration, negatively impact the flavour profile and are the result of process-related physical and chemical degradation, are listed in Table 5 below:

Table 5: Impact of the membrane process on odor and aroma components

Foodstuff	**Process-related loss**	**Formation of disturbing component(s)**
Pineapple	4-hydroxy-2,5-dimethyl-3(2H)-furanone, ethyl 2-methylbutanoate	methional, 2,4-(E,E)-decadienal
Apple	2-(E)-hexenal, cis-3-hexenol, hexanal, ethyl butanoate, 2-methylbutyl acetate	2-3-methylbutyraldehyde, furfural, 1,3-pentanal, 3,5-octadienal, linalool oxide, 8-p-cymenol
Beer	isoamyl acetate, 3-methylbutanol, linalool, myrcene, phenylethanol, ethyl butanoate	trans-2-nonenal, dimethyl sulfide
Citrus fruits	acetaldehyde, cis-3-hexenal, hexanal, ethyl butanoate	α-terpineol, p-dimethylstyrene, limonene epoxide, carveol, carvone, 4-vinylguaiacol
Strawberry	acetaldehyde, methyl butanoate, ethyl 2-methylbutanoate, cis-3-hexenal, hexanal	β-damascenone, 2,4-(E,E)-decadienal, furfural, guaiacol
Coffee	methanethiol, dimethyl sulfide, methylpropanal, 2,3-butanedione, 2-furfurylthiol	bis-(2-furfuryl)disulfide, 2-(2-furyl)methylthio-hydroxy-1,4-dihydro-pyrazine
Milk	diacetyl, 2-methylbutanol, cis-4-heptenal, trans-2-nonenal, 1-octen-3-one, 1-octen-3-ol	dimethyl sulfide, dimethyl disulfide, 2-hexanone, 2-heptanone, 2,3-methylbutanal

Foodstuff	Process-related loss	Formation of disturbing component(s)
Passion fruit	acetaldehyde, hexanal, ethylbutyrate, linalool	benzaldehyde, α-terpineol, furfural, acetic acid, acetophenone, β-ionone, linalool oxide
Tea	cis-3-hexenal, cis-3-heptenal, linalool, 3-methyl-2,4-nonanedione, 2,3-methylbutyric acid, 1,5-octadien-3-one	β-damascenone, 2,4-(E,E)-decadienal, 3-hydroxy-4,5-dimethyl-2(5H)-furanone, 4-hydroxy-2,5-dimethyl-3(2H)-furanone
Tomato	cis-3-hexenol, cis-3-hexenal, trans-2-hexenal, hexanal, β-ionone, 2-isobutylthiazole	dimethyl disulfide, methional, 4-hydroxy-2,5-dimethyl-3(2H)-furanone
Onion juice	propanal, hexanal, trans-2-heptenal, dipropyl disulfide, dipropyl trisulfide, methylpropyl disulfide	dimethyl trisulfide, dipropyl trisulfide, dimethyl tetrasulfide, 3,5-diethyl-1,2,4-trithiolane, thiophene

[0081] Concentration of the foodstuff metnioned in Table 5 using the method described in accordance to the invention leads to a significantly more authentic sensory profile than with conventional distillation, adsorption and membrane methods known from the prior art. In particular, there are no process-related losses of flavourings and no distubing components are formed that change the sensory profile.

[0082] A further advantage of the method in accordance to the invention is that, in comparison to conventional thermal methods, for example distillative methods, it is particularly suitable for concentrating thermolabile foodstuff or foodstuff with thermolabile ingredients, since it can be carried out at low temperatures, preferably at 25 °C, and thus flavouring concentrates can be obtained whose taste and aroma are not impacted by heat.

[0083] The flavouring concentrates recovered by the method in accordance to the invention are also characterised by the fact that they do not contain any solvent additives. In contrast to the conventional adsorption methods known from the prior art for concentrating aqueous starting

solutions from a foodstuff, no solvents are used in the method in accordance to the invention; thus, no solvent residues are found in the resulting flavouring concentrate. At the same time, this also prevents solvent-related precipitation reactions and discoloration, so that clear, unclouded flavouring concentrates are obtained. Solvents that are typically used in such adsorption methods for recovering the aroma concentrate from foodstuff are acetone, butane, butan-1-ol, butan-2-ol, cyclohexane, nitrous oxide, diethyl ether, ethyl acetate, ethanol, ethyl methyl ketone, hexane, methanol, methyl acetate, propane, propan-1-ol and propan-2-ol and which do not originate from the foodstuff.

[0084] The present invention thus also relates to a flavouring concentrate which is obtainable by the method described above.

[0085] In accordance to the invention, the foodstuff concentrate is a flavouring concentrate.

[0086] As described in detail above, the flavouring concentrate in accordance to the invention has no off-flavour. Off-flavour is the recognized and used technical term for flavouring defects. Flavouring defects arise when an aroma component that is not normally present is present in the affected food, one or more characteristic compounds (impact compounds) have been lost, or a concentration shift has occurred between the individual aroma components.

[0087] As is apparent from the detailed description above, the present invention also relates to a flavouring concentrate according to the claims, in which the sensory profile of a diluted concentrate from the osmosis in accordance to the invention with the semipermeable biomimetic membrane deviates from the sensory profile of an aqueous starting solution on a scale of 0 to 10 points in the 6 taste axes of a conventional panel profile by a total of max. 1 point, wherein the sensory profile is measured via a test panel according to DIN 10967-1-1999, or according to the protocol in accordance to the invention described below.

[0088] To create a sensory profile, the descriptive terms (descriptors) are first collected in the panel, wherein the term lists are structured, similar terms are summarized and hedonic attributes are eliminated. The intensity of the descriptors is assessed on a scale of 1 - 10 by at least ten trained assessors. The samples are coded and tasted in a sensory room in a randomized sequence, excluding disturbing influences such as color, noise and foreign odors. The final result is determined by

summing the individual results and then calculating the arithmetic mean and is presented graphically in the form of a radar chart.

[0089] As can be seen from Figures 5 and 6, the sensory profile of the concentrate after redilution, which was recovered according to the described method, deviates from the sensory profile of the aqueous starting solution (water phase) of yellow peach and strawberry in the corresponding 6 taste axes by a maximum of 1 point each.

[0090] Further, as can be seen from the above analyses, the recovery rate of the flavourings with an OAV (odor activity value) ≥ 1 in the aqueous starting solution of ≥ 1 in the case of the flavouring concentrate in accordance to the invention is > 90 % in the retentate, preferably > 98 %, in particular > 99 %, based on the aqueous starting solution. As is clearly evident from Tables 3 and 5, the concentration of the respective vapour condensates from fruit or vegetables in accordance to the invention leads to a concentration of the aroma components without any negative impact on the sensory profile with almost complete recovery of the aroma components, whereby an authentic sensory profile is obtained.

[0091] The OAV value is defined as a measure of the importance of a specific component in the odor of a sample, for example a foodstuff. The OAV value is calculated by dividing the concentration of a particular substance in the foodstuff (mg/kg) by the concentration threshold of that substance in the foodstuff (mg/kg). In case of an OAV > 1, the concentration of the substance exceeds the odor threshold concentration and the substance then makes a sensory contribution to the overall profile. Substances with an OAV < 1 therefore do not contribute to the sensory profile.

[0092] The method described herein can also be used to manufacture concentrates which are particularly outstanding. These are characterised by higher authenticity in taste and odor, since odor-active substances are retained during the method. It was found that the flavouring concentrates can be represented by the following relationship: accordingly, the flavouring concentrate in accordance to the invention is characterised by the fact that the flavourings, i.e. the odor-active substances, in the flavouring concentrate with a parameter W (log of the sensory recovery coefficient), which is preferably > - 3, even more preferred > - 5, based on the aqueous starting solution, have a recovery

rate of ≥ 50%, preferably ≥ 70%. Particularly preferred is a recovery rate of ≥ 90% or ≥ 95%, and most preferred is a recovery rate of ≥ 98% or ≥ 99%.

[0093] The sensory recovery coefficient (SWF) of a flavouring is composed of the logP of the flavouring and the odor threshold concentration of the flavouring (mg/kg in water) and is defined as follows:

$$\text{Sensor. Wiederfindungskoeffizient SWF} = \frac{10\ \text{^logP}}{\text{Geruchsschwellen–Konzentration}\ (\frac{\text{mg}}{\text{kg}})}$$

The logP value is defined as follows:

$$\text{logP} = \log K(\text{n-Octanol/Wasser}) = \frac{\text{Cn–Octanol}}{\text{CWasser}},$$

wherein:

C (n-octanol) is the concentration of the flavouring in the n-octanol phase; and

C (water) is the concentration of the flavouring in the water phase.

The log K(n-octanol/water) is a dimensionless partition coefficient that indicates the ratio of the concentration of the flavouring in a two-phase system of n-octanol and water. It is therefore a model measure for the polarity or water/fat solubility of the flavouring.

LogP is a common physical parameter and is positive for lipophilic flavourings and negative for hydrophilic flavourings.

The parameter W is therefore defined as follows:

$$W = log_{10}(SWF)$$

[0094] The parameter W is thus an absolute value of the respective substance, which allows discrimination in accordance to the invention, taking into account the sensory activity of the substances.

[0095] LogP, odor threshold values and sensory recovery coefficients of flavourings are listed in Table 6 below:

Tabelle 6: Geruchsschwellen-Werte und Sensorische Wiederfindungskoeffizienten

Verbindung	logP	Schwelle [mg/kg in Wasser]	Sensorischer Wiederfindungs-koeffizient (SWF)	W = log (SWF)
Methanol	-0,358	369828	1,18577E-06	-5,93
Ethanol	-0,19	52000	1,24164E-05	-4,91
Propanol	0,5145	5700	0,000573621	-3,24
Hexanol	1,8831	50	1,528023367	0,18
Z-3-Hexenol	1,4385	13	2,111332572	0,32
Acetaldehyd	-0,1825	63	0,010427002	-1,98
Propanal	0,4844	170	0,017945315	-1,75
2-Methylbutyraldehyd	1,4031	0,9	28,10978284	1,45
Hexanal	1,853	16	4,455331438	0,65
Essigsäure	-0,2299	180000	3,27211E-06	-5,48
Buttersäure	0,8932	1000	0,007819878	-2,11
Hexansäure	1,8056	3944	0,016205525	-1,79
Ethylacetat	0,73	3125	0,001718502	-2,76
Ethylbutanoat	1,493	1	31,11716337	1,49
Ethylhexanoat	2,4054	5	50,86628212	1,71
2,3-Butandion	-0,5078	54	0,005751833	-2,24
2,3-Pentandion	0,1591	5	0,28848949	-0,54
gamma-Octalacton	2,0901	24	5,127300347	0,71
Linalool	2,7354	103	5,279135088	0,72
Geranial	3,1904	320	4,84451184	0,68

[0096] Odor-active substances with a log of the sensory recovery coefficient W of the flavourings > - 3, irrespective of their size, molecular weight and chemical structure, are well retained in the retentate by the biomimetic membrane, so that the concentrates obtained have a very authentic and almost identical sensory profile based on the aqueous starting solution. Due to their polar structure, methanol and ethanol partially migrate through the aquaporin water channels; however, due to their high threshold value, these substances are of little relevance for the sensory profile.

[0097] In accordance to the invention, a flavouring concentrate in which the ethanol content is reduced by at least 50%, based on the aqueous starting solution, is particularly preferred. The advantage of the method in accordance to the invention is therefore a high reduction of ethanol, while at the same time strongly retaining the characteristic flavourings. This means that concentrates can

also be sold in countries that have cultural reservations about ethanol. Even more preferably, the recovery rate of ethanol according to the present method is < 90%, even more preferably < 99%, so that the ethanol contents can be significantly reduced without losing the characteristic taste of the starting solution.

[0098] Preferably, the recovery rate of the a flavourings and aromas having a molecular weight of 40 to 300 Da, preferably 86 to 170 Da, in the retentate is > 90%, based on the aqueous starting solution, even more preferably the recovery rate of the flavourings having a molecular weight of 98 to 200 Da is > 98%.

[0099] A further particular advantage of the flavouring concentrate in accordance to the invention is that it is free from solvent additives, in particular free from solvent additives selected from the group consisting of: acetone, butane, butan-1-ol, butan-2-ol, cyclohexane, nitrous oxide, diethyl ether, ethyl acetate, ethanol, ethyl methyl ketone, hexane, methanol, methyl acetate, propane, propan-1-ol and propan-2-ol. The above-memtioned solvent additives are those solvents which are utilized in the conventional adsorptive methods for the elution/desorption of the value-adding flavourings and aromas and thus for recovering the aroma concentrate and which do not originate from the native foodstuff. These solvents remain as residues in the aroma concentrate and are toxicologically questionable in some cases.

[0100] An advantageous property of the flavouring concentrate in accordance to the invention is also that it is free from disturbing aroma components, preferably that the flavouring concentrate is free from disturbing aroma components with an OAV (odor activity value) ≥ 1, which change the sensory profile.

[0101] The disturbing components are in particular those compounds which are formed in conventional osmosis methods with known membranes or in distillative methods for recovering the aroma concentrate, for example by thermal degradation, by pH shift, by oxidation or by chemical rearrangement reactions, and thus impact the sensory profile of the foodstuff concentrate. Such disturbing components formed by thermal degradation of foodstuff ingredients, for example in conventional distillative methods for recovering the aroma concentrate, can negatively change the

sensory profile of the resulting flavouring concentrate.

Such disturubing components are preferably:

[0102]

For pineapple: methional, 2,4-(E,E)-decadienal

For apple: 2-3-methylbutyraldehyde, furfural, 1,3-pentanal, 3,5-octadienal, linalool oxide, 8-p-cymenol

For citrus fruits: α-terpineol, p-dimethylstyrene, limonene epoxide, carveol, carvone, 4-vinylguaiacol

For strawberry: β-damascenone, 2,4-(E,E)-decadienal, furfural, guaiacol

For passion fruit: benzaldehyde, α-terpineol, furfural, acetic acid, acetophenone, β-ionone, linalool oxide

For tomatoes: dimethyl disulfide, methional, 4-hydroxy-2,5-dimethyl-3(2H)-furanone

For onion juice: dimethyl trisulfide, dipropyl trisulfide, dimethyl tetrasulfide, 3,5-diethyl-1,2,4-trithiolane, thiophene

[0103] In a further preferred embodiment, the flavouring concentrate in accordance to the invention is free from disturbing aroma components with an OAV (odor activity value) ≥ 1 and/or contains no solvent additives. Even at a concentration of at least 100 times, in particular a concentration of 200 to 1,000 times, the flavouring concentrate in accordance to the invention is free of disturbing aroma components with an OAV (odor activity value) ≥ 1 and/or contains no solvent additives. Accordingly, the flavouring concentrate in accordance to the invention is at least 100-fold, in particular 200- to 1,000-fold.

[0104] In this flavouring concentrate, the disturbing components are preferably as defined above and, depending on the starting solution, none of the disturbing components defined above are contained in the respective foodstuff starting solutions in such quantities that are odor-active. Furthermore, no solvent additives are included, which are usually utilized as eluents in adsorptive concentration methods. This flavouring concentrate is therefore unique in taste and composition at

these concentration ratios, e.g. at a flavouring concentrations of 1000 to 10000 ppm. These foodstuff concentrates generally have less browning and fewer precipitation reactions.

[0105] The flavouring concentrate in accordance to the invention is preferably a concentrate which is manufactured from one or more of the following foodstuff: fruit juices such as pineapple juice, apple juice, aronia juice, citrus fruit juice, strawberry juice, passion fruit juice and pear juice; vegetable juices such as cucumber juice, carrot juice, asparagus juice, tomato juice, onion juice; squeezed juices of basil, kiwi, mango, parsley, celery or spinach.

[0106] The flavouring concentrate in accordance to the invention can be used for aromatisation or reconstititon of the aroma in foodstuff, beverages, semifinished products, oral hygiene products, cosmetic or pharmaceutical products.

Examples

[0107] The method of the present invention and the flavouring concentrates obtained thereby will now be described in more detail using individual examples.

[0108] In general, the following operating parameters are used unless explicitly stated otherwise in the examples:

Procedural details for the operation of the aquaporin membrane module:

- Module design: Polycarbonate tube, 23 cm long and 5 cm in diameter, interspersed with hollow fibers (so-called hollow fiber module)
- Flux: min. > 12 l/m^2/h
- pH: 2 to 11
- Temperature: max. 50 °C
- Pressureless at flow of max. 18 l/h of the water phase and max. 18 l/h of the osmosis solution with a 0.6 m^2 module
- 4 M NaCl as osmosis solution

Example 1 (not in accordance to the invention): Preparation of a cucumber juice concentrate

[0109]

Product: cucumber juice, freshly squeezed

Starting amount: 10,000 g cucumber juice, water phase

2,000 g osmosis solution 60 °Brix potassium lactate

Concentrating the flavourings by a factor of 20

[0110]

Apparatus: Forward osmosis module from Aquaporin LHF033 combined with 10 liter tank from Sartorius + 2 Ismatec pumps

Membrane: FO Module Aquaporin LHF033,

0.6 m^2 active membrane area

Experimental procedure: The cucumber juice and the draw solution were pre-filtered using a 100 µm bag filter to remove solids and suspended matter. The feed tank was filled with 10 kg cucumber juice, and 2.0 kg draw solution (osmosis solution) was provided in the canister. The feed pump and the draw solution pump were then started with a flow of 300 ml/min each. After removing 9 kg, the experiment is terminated. The feed tank is rinsed with 2 kg distilled water. The rinse water is also sent for analysis.

Weights:

[0111]

Starting amount	10,000 g
Permeate:	8,835 g
Retentate:	1,091 g
Rinse water (weight):	2,004 g

Draw Solution start: 60 °Brix

Draw Solution end: 12.4 °Brix

Temperature: 25 °C

Recording of data:

[0112]

Time (min)	0	15	30	60	90	120	150
Amount of product added (g)	10,000						
Amount of draw solution added (kg)	2.0						
Weight of the draw solution (kg)	0	2,013	3,352	5,150	6,352	7,227	7,912
Flux (l/m^2·h)			11.7	8.6	7.1	6.0	5.3

Time (min)	180	210	240	270	300
Weight of the draw solution (kg)	8,385	8,629	8,753	8,810	8,835
Flux (l/m^2·h)	4.7	4.1	3.6	3.3	2.9

Example 2 (not in accordance to the invention): Preparation of a yellow peach concentrate

[0113]

Product: Yellow peach, pure extract

Starting amount: 10,000 g yellow peach, water phase

2,000 g osmosis solution 60 °Brix potassium lactate

Concentrating the flavourings by a factor of 20

[0114]

Apparatus: Forward osmosis module from Aquaporin LHF033 combined with 10 liter tank from Sartorius + 2 Ismatec pumps

Membrane: FO Module Aquaporin LHF033,

0.6 m^2 active membrane area

Experimental procedure: The feed tank was filled with 10,000 g water phase. 2.0 kg draw solution (60 °Brix) was provided in the canister. The feed pump and the draw solution pump

were then started with a flow of 300 ml/min each.

Weights:

[0115]

Starting amount 10,000 g

Retentate: 413.5 g

Permeate: 9,500 g

Draw solution start: 60 °Brix

Draw solution end: 12,6 °Brix

Temperature: 25 °C

Recording of data:

[0116]

Time (min)	0	15	30	60	116 end
Amount of product added (g)	10,000				
Amount of draw solution added (kg)	2.0				
Weight of the draw solution (kg)	0	2,100	3,890	6,500	9,500
Flux ($l/m^2 \cdot h$)		14.0	11.9	8.7	5.4

The sensory profile is as follows:

Intensity: 1 - 10

[0117]

	Starting material yellow peach, water phase	Concentrate after dilution
Fruity-estery	5	4
Ripe	6	5.5
Peach-like	7	7
Fleshy	4	4

	Starting material yellow peach, water phase	Concentrate after dilution
Juicy	5	5
Cooked	0	0

[0118] The sensory profile is presented in Figure 5.

Example 3: Manufacture of a strawberry concentrate

[0119]

Product: Water phase from strawberry juice concentration

Starting amount 10,000 g strawberry, water phase

2,000 g osmosis solution 60 °Brix potassium lactate

Concentrating the flavourings by a factor of 20

[0120]

Apparatus: Forward osmosis module from Aquaporin LHF033 combined with 10 liter tank from Sartorius + 2 Ismatec pumps

Membrane: FO Module Aquaporin LHF033,

0.6 m^2 active membrane area

Experimental procedure: The feed tank was filled with 10,000 g water phase, and 2.0 kg draw solution (60 °Brix) was provided in the canister. Then the feed pump and the draw

solution pump were started with

a flow of 300 ml/min each.

Weights:

[0121]

Starting amount: 10,000 g

Retentate: 510.0 g

Permeate: 9,500 g

Draw solution start: 60 °Brix

Draw solution end: 12.4 °Brix

Temperature: 25 °C

Recording of data:

[0122]

Time (min)	0	15	30	60	90
Amount of added product (g)	10,000				
Amount of added draw solution (kg)	2.0				
Weight of the draw solution (kg)	0	1,890	3,638	6,054	7,990
Flux ($l/m^2 \cdot h$)		12.6	11.7	8.1	6.5

Time (min)	120	123 end			
Amount of product added (g)					
Amount of draw solution added (kg)					
Weight of the draw solution (kg)	9,440	9,500			
Flux ($l/m^2 \cdot h$)	4.8	4.0			

The sensory profile is as follows:

Intensity: 1 - 10

[0123]

	Starting material strawberry, water phase	Concentrate after dilution
Fruity-estery	6	5
Buttery	4	5
Green	5	5
Ripe	4	4

	Starting material strawberry, water phase	Concentrate after dilution
Juicy	6	6
Cooked	0	0

[0124] The sensory profile is presented in Figure 6.

Example 4 (not in accordance to the invention): Preparation of a concentrate of black tea, Ceylon

[0125]

Product: Black tea from Ceylon

Starting amount: 600 g tea

12,000 g water

2,000 g osmosis solution 60 °Brix potassium lactate

Concentrating the flavourings by a factor of 20

[0126]

Apparatus: Forward osmosis module from Aquaporin LHF033 combined with 10 liter tank from Sartorius + 2 Ismatec pumps

Membrane: FO Module Aquaporin LHF033,

0.6m^2 active membrane area

Experiment preparation: 600 g tea were extracted in 12 kg water for 2 hours at room temperature, then the suspended matter was removed using a sieve and pleated filter. 10 kg of the water phase was used for forward osmosis.

Experimental procedure: The feed tank was filled with 10,000 g water phase, and 2.0 kg draw solution (60 °Brix) was provided in the canister. The feed pump and the draw solution pump were then started with a flow of 300 ml/min each.

Weights:

[0127]

Starting amount: 10,000 g, 1.3 °Brix

Retentate: 278.1 g, 33.1 °Brix

Permeate: 9,602 g

Draw solution I start: 60 °Brix

Draw solution II start: 60 °Brix

Draw solution end: 18,4 °Brix

Draw Solution II end: 21.9 ° Brix

Recording of data:

[0128]

Time (min)	0	15	30	60	120
Amount of added	10,000				
product (g)					
Amount of added	2.0				
draw solution (kg)					
Weight	0	2,028	2,880	3,760	4,744
Draw solution (kg)					
Flux ($l/m^2 \cdot h$)		13.5	5.7	2.9	1.6

Time (min)	180	240	300	344	
Amount of added					
product (g)					
Amount of added		2.0			
draw solution (kg)					
Weight	5,569	2,068	3,510	4,033	

Time (min)	180	240	300	344	
Draw solution (kg)					
Flux ($l/m^2 \cdot h$)	1.3	7.6	2.4	1.2	

Patentkrav

1. Fremgangsmåde til fremstilling af et aromakoncentrat omfattende følgende trin:

- fremstilling af en vandig udgangsopløsning fra fødevare, hvor den vandige udgangsopløsning er et dampkondensat fra frugter eller grøntsager,
- opkoncentrering af den vandige udgangsopløsning ved osmose ved hjælp af en semipermeabel, biomimetisk membran, og
- dannelse af et aromakoncentrat som retentat,

hvor den semipermeable, biomimetiske membran omfatter:

vesikler af liposomer eller polymersomer indeholdende mindst ét aquaporinprotein;
en tyndfilmskompositmatrix indeholdende indlejrede vesikler; og
et bærelag;
hvor fremgangsmåden udføres ved en temperatur i området fra 10 til 40 °C; og
hvor den vandige udgangsopløsning er koncentreret med faktoren 20 til 1000.

2. Fremgangsmåde ifølge krav 1, hvor liposomet omfatter: lipider såsom DPhPC, DOPC, blandede sojabønnelipider, asolectin eller blandede lipider af *E. coli;* og hvor polymersomet omfatter: triblokcopolymerer af den hydrofile-hydrofob-hydrofile type A-B-A, A-B-C eller CBA, hvor A står for PMOXA, B står for PDMS og C står for PEO, diblokcopolymerer af den hydrofile-hydrofobe type A-B, hvor A står for PB og B står for PEO eller kombinationer deraf.

3. Fremgangsmåde ifølge et hvilket som helst af kravene 1 eller 2, hvor akvaporinproteinet vælges fra gruppen bestående af AQPO, AqpZ, SoPIP2 AQP10 og isoformer deraf, fortrinsvis hvor akvaporinproteinet er AQPO.

4. Fremgangsmåde ifølge et hvilket som helst af kravene 1 til 3, hvor tyndfilmskompositmatrixen fremstilles ved polymerisation af en vandig opløsning af en amin med en opløsning af et syrechlorid i et organisk opløsningsmiddel.

5. Fremgangsmåde ifølge et hvilket som helst af kravene 1 til 4, hvor den vandige udgangsopløsning opkoncentreres med mindst faktoren 100.

6. Smagskoncentrat opnået ved en fremgangsmåde ifølge et hvilket som helst af de foregående krav 1 til 5, der ikke udviser nogen bismag og er fri for forstyrrende aromabestanddele, navnlig fri for forstyrrende aromabestanddele med en OAV (lugtaktivitetsværdi) ≥ 1.

7. Smagskoncentrat ifølge krav 6, hvor den aromatiske profil af et fortyndet koncentrat fra osmosen med en semipermeabel, biomimetisk membran afviger med maksimalt 1 point fra den aromatiske profil af en vandig udgangsopløsning ved anvendelse af en skala fra 0 til 10 point til at evaluere de 6 smagsakser af en konventionel panelprofil, hvor den aromatiske profil måles af et testpanel ifølge DIN 10967-1-1999 eller en protokol ifølge opfindelsen.

8. Smagskoncentrat ifølge et hvilket som helst af kravene 6 til 7, hvor ethanolindholdet er reduceret med mindst 50 % i forhold til den vandige udgangsopløsning.

9. Aromakoncentrat ifølge krav 6, som er fri for forstyrrende stoffer afhængigt af udgangsopløsningerne, udvalgt fra grupperne indeholdende:

til ananas: methional, 2,4-(E,E)-decadienal

til æble: 2,3-methylbutyraldehyd, furfural, 1,3-pentanal, 3,5-octadienal, linalooloxid, 8-p-cymenol

til citrusfrugter: α-terpineol, p-dimethylstyren, limonenepoxid, carveol, carvon, 4-vinylguaiacol

til jordbær: β-damascenon, 2,4-(E,E)-decadienal, furfural, guaiacol

til passionsfrugt: benzaldehyd, α-terpineol, furfural, eddikesyre, acetophenon, β-ionon, linalooloxid

til tomat: dimethyldisulfid, methional, 4-hydroxy-2,5-dimethyl-3(2H)-furanon

til løgjuice: dimethyltrisulfid, dipropyltrisulfid, dimethyltetrasulfid, 3,5-diethyl-1,2,4-trithiolan, thiophen.

10. Smagskoncentrat ifølge et hvilket som helst af de foregående krav 6 til 9, som er

mindst 100 gange, fortrinsvis 200 gange til 1000 gange.

11. Anvendelse af aromakoncentratet ifølge et hvilket som helst af de foregående krav 6 til 10 til aromatisering eller rekonstituering af aromaen i fødevarer, drikkevarer, halvfabrikata, mundhygiejneprodukter, kosmetiske eller farmaceutiske produkter.

FIGURES

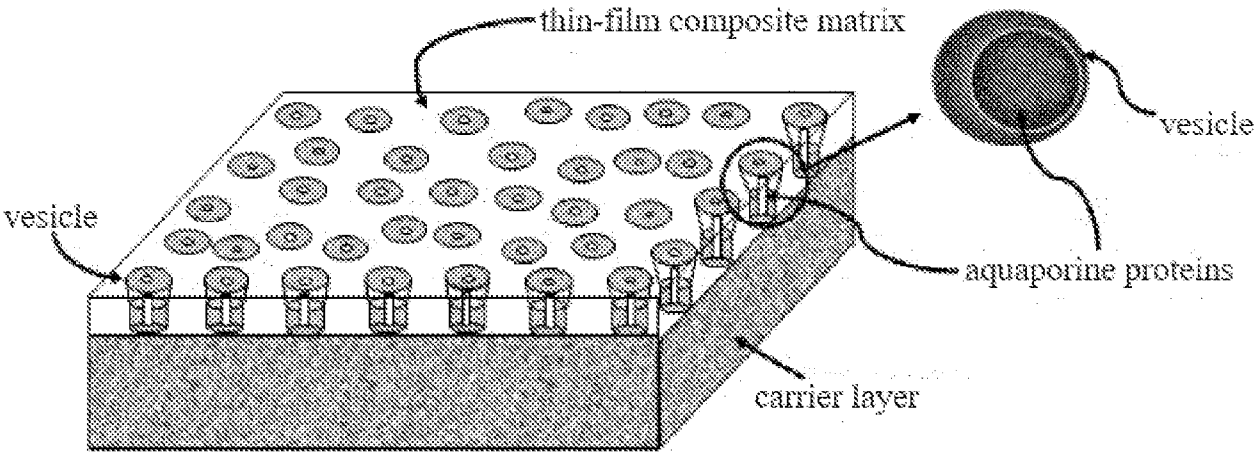


Figure 1: Structure of an aquaporine membrane

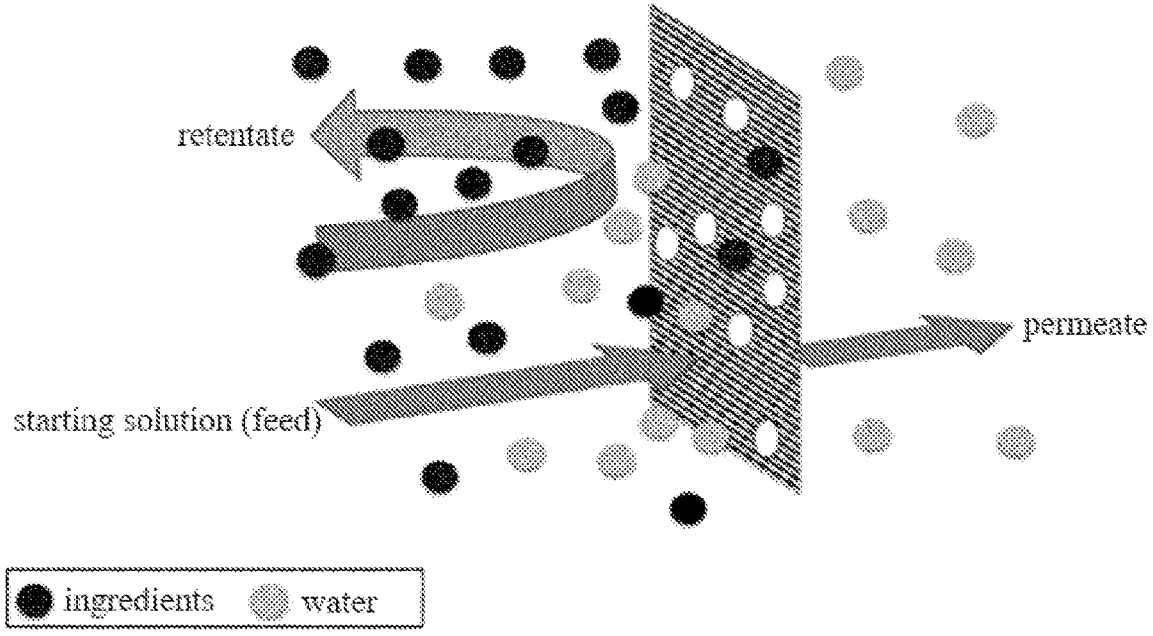


Figure 2: Principle of forward osmosis

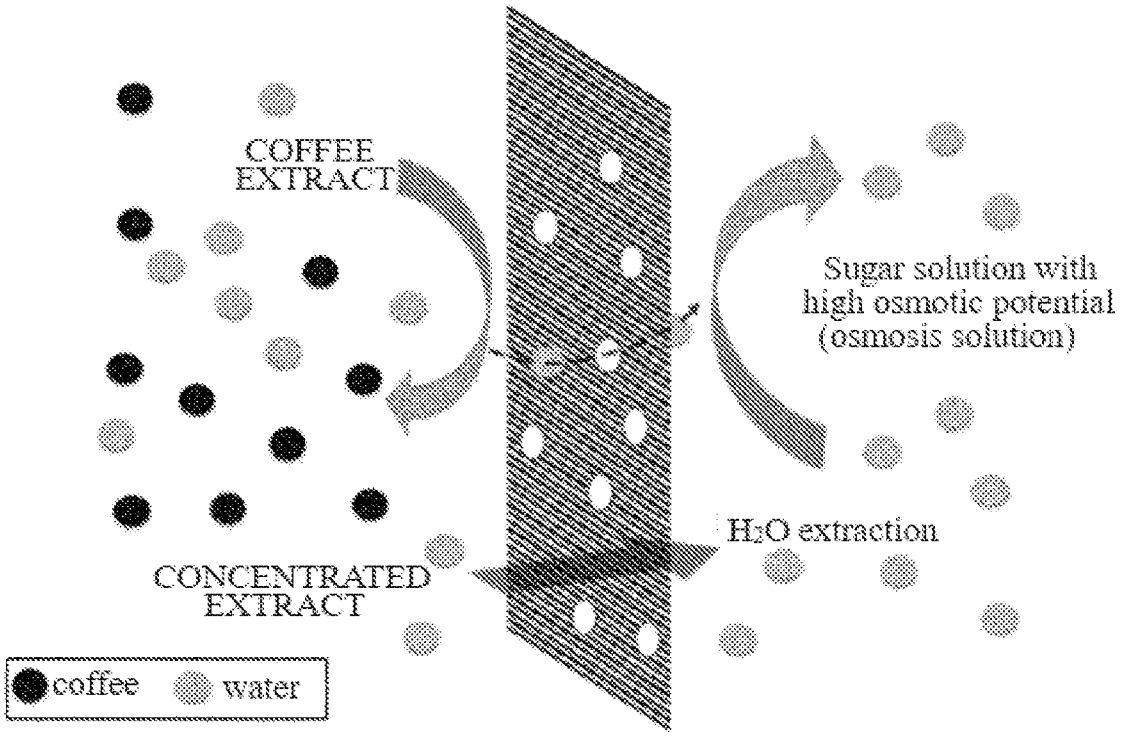
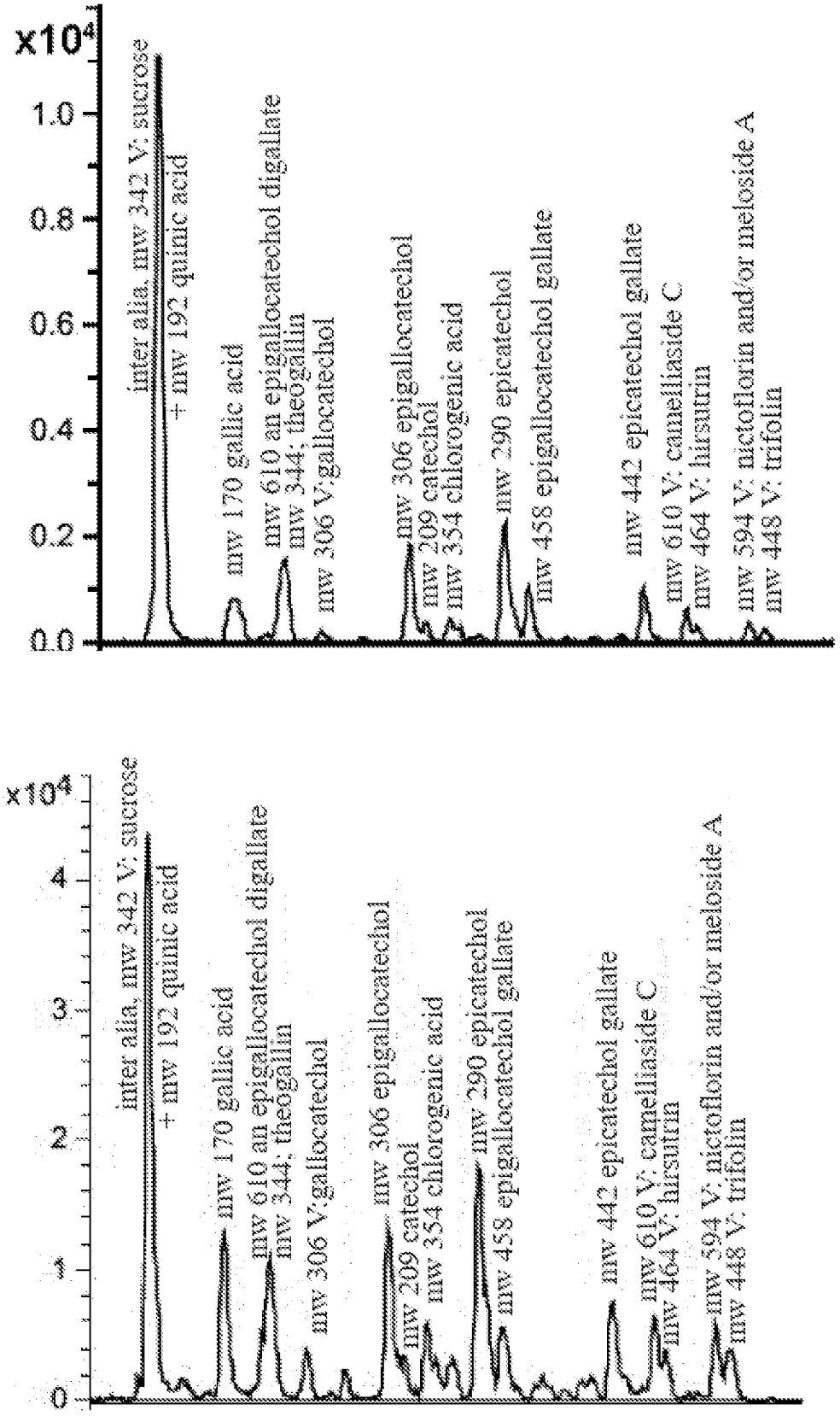


Figure 3: Forward osmosis with aquaporin membrane in the case of a coffee extract as an example



Figures 4a and 4b: Comparison of chromatograms of a tea extract, top, and the diluted concentrate of the same tea extract, bottom

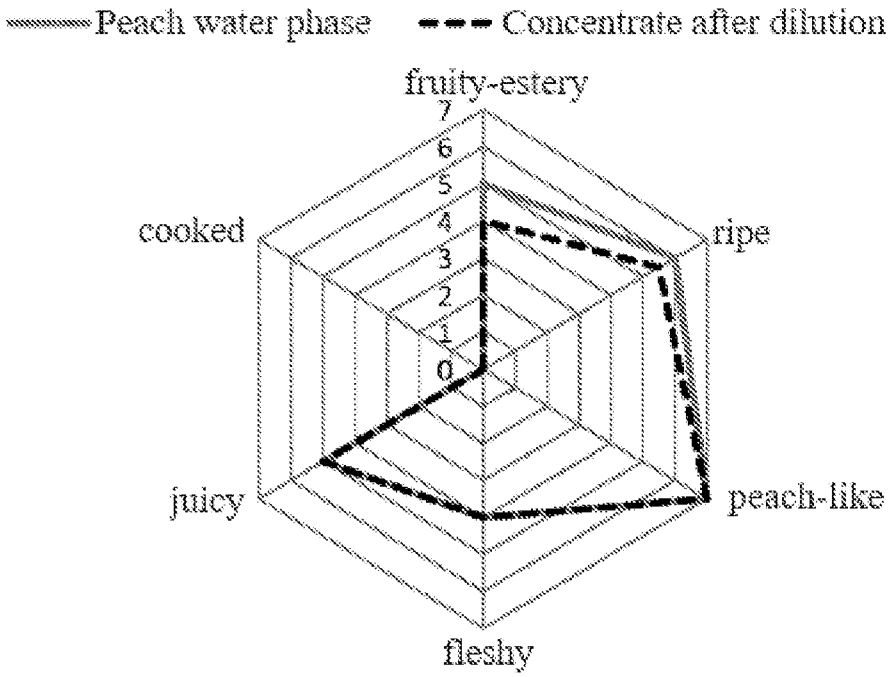


Figure 5: Yellow peach: Sensory profiling of water phase and diluted concentrate from aquaporine membrane filtration

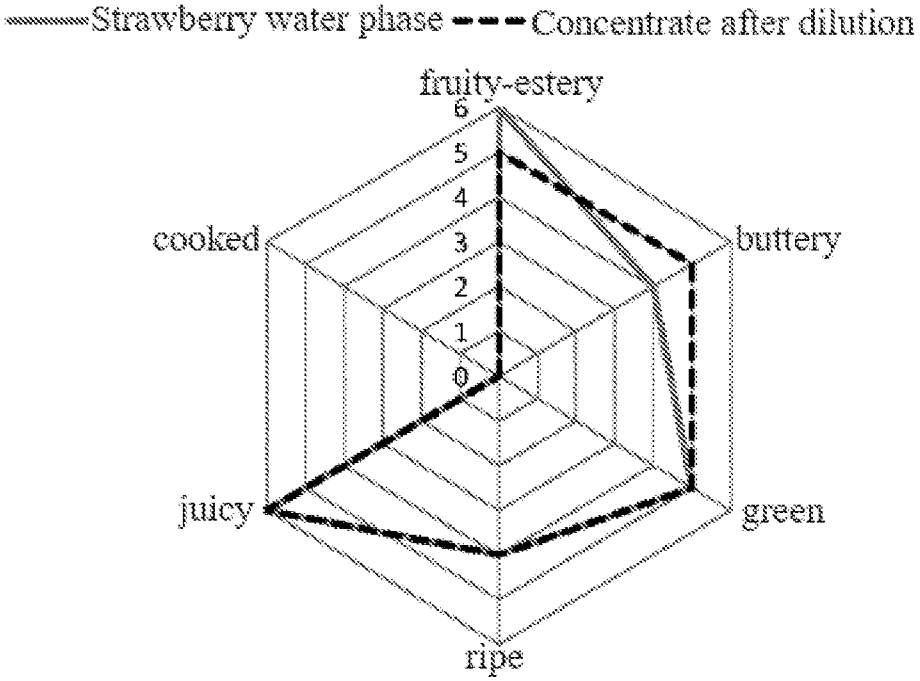


Figure 6: Strawberry: Sensory profiling of water phase and diluted concentrate from aquaporine membrane filtration