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(71) Ansøger: **AQUAPORIN A/S, Nymøllevej 78, 2800 Kongens Lyngby, Danmark**

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(54) Benævnelse: **Dewatering of aqueous coffee extract**

TITLE

Dewatering of aqueous coffee extract

TECHNICAL FIELD

The present invention relates to a method for concentrating an aqueous coffee extract in a forward osmosis flow cell comprising a feed solution compartment and a draw solution compartment separated by a membrane. The invention has a better ability to retrain coffee aroma components in the final product.

BACKGROUND

The green coffee beans are relatively stable and are transported from the coffee-producing countries to the consumers all over the world. At industrial sites for extraction the green coffee beans are processed to produce extract and/or instant soluble coffee. The green coffee beans are initially subjected to roasting. The roasting transforms the chemical and physical properties of the green coffee beans and produces the characteristic flavour of coffee. In the same process the coffee beans expand and change in colour and density.

A commonly used roasting plant in the industry is rotating cylinders containing the green beans and hot combustion gases. When the bean temperature reaches typically 165-200°C the roasting begins, accompanied by a popping sound similar to that produced by popcorn. These batch cylinders take about 8-15 min to complete the roasting depending on the initial moisture and desired final colour. Coffee roasting using a

fluidized bed is also commonly used. The roasted beans are then ground to enhance extraction with water. Grinding reduces the beans to 0.2-5.0 mm depending on the extraction process. Traditionally, roasted beans are ground by dry milling.

Traditional household coffee brewing is performed at a water temperature around the boiling point, i.e. 100°C. In industrial facilities, the extraction temperature is generally higher, e.g. 180°C, to obtain a higher yield.

To produce instant coffee powder, water must be removed from the extract in one or more steps. Due to process economy, it is desired to use a dewatering or up concentrating step before the final spray drying or freeze drying step to produce the instant coffee powder. In addition to removal of water from the aqueous coffee extract, many conventionally used dewatering methods also remove valuable aroma components. The reduction of the concentration of these aroma components results in the final product providing a lesser taste and smell experience for the coffee drinker. The present invention has the object of improving the amount and concentration of aroma components in coffee extracts.

SUMMARY

It is an object to provide a method for concentrating an aqueous coffee extract using a forward osmosis flow cell comprising a feed solution compartment and a draw solution compartment separated by a membrane, wherein

the feed solution compartment comprises an inlet and an outlet for passing the aqueous coffee extract,

the draw solution compartment comprises an inlet and an outlet for passing a draw solution containing one or more solutes, and

the membrane comprises an active layer and a support layer, wherein the active layer comprises immobilized aquaporin water channels.

The aquaporin-embedded membranes enable high retention of flavor and aroma components in the feed solution and low reverse diffusion of the solute using only osmotic pressure. Osmotic pressure is a natural process that seeks to equalize osmotic pressures in the two compartments with different solute concentrations. Osmotically facilitated water extraction is the gentlest process for concentration of delicate components and can therefore guarantee a better-quality product compared to traditional processes (e.g. thermal or pressure driven processes). Aquaporin proteins are 100% selective to water molecules, which drastically lowers back diffusion of draw solutes by the aquaporin-containing membrane.

The aqueous coffee extract and/or the draw solution may be allowed only a single or a few passes of the forward osmosis flow cell if no high degree of water removal is required or the flow path along the membrane is sufficiently long for the desired water recovery to occur. In general, however, it is desired to let the aqueous coffee extract be recirculated in the feed solution compartment and/or the draw solution containing one or more solutes to be recirculated in the draw solution compartment.

The aquaporin water channels may be incorporated in the active layer of the membrane using various methods. Presently, it is preferred that the aquaporin water channels are immobilized in a thin film composite (TFC) layer. The TFC layer ideally should highly reject any component in the feed as well as the draw solution to minimize transmembrane feed and draw solute flux. In reality, however, a minor level of solute flux is observed. To obtain a thin film composite layer which is able to reject solutes to a high degree, the active layer is preferably selected as a cross-linked aromatic amide layer formed by interfacial polymerization. In a certain embodiment, the TFC layer is formed through interfacial polymerization of an aqueous solution of a di- or triamine with a solution of di- or triacyl halide in an organic solvent. The TFC layer may be prepared using an amine reactant, preferably an aromatic amine, such as a diamine or triamine, e.g. 1,3-diaminobenzene (m-Phenylenediamine - abbreviated MPD) in an aqueous solution, and an acyl halide reactant also known as an amine-reactive molecule, such as a di- or triacid chloride, preferably an aromatic acyl halide, e.g. benzene-1,3,5-tricarbonyl chloride (CAS No. 84270-84-8, trimesoyl chloride (TMC)) dissolved in an organic solvent where said reactants combine in an interfacial polymerization reaction, cf. US Patent No: 4,277,344 which describes in detail the formation of a composite membrane comprising a polyamide laminated to a porous membrane support, at the surface of the support membrane.

The aquaporin water channels may need to be stabilized before incorporation in the TFC layer. In a certain aspect of the invention the aquaporin water channels are incorporated in

vesicles prior to the incorporation in the TFC layer. The vesicles may be prepared from lipids such as DPhPC, DOPC, mixed soy bean lipids, asolectin or E. coli mixed lipids. In a further embodiment, the vesicles comprise triblock copolymers of the hydrophile-hydrophobe- hydrophile (A-B-A or A-B-C) type or diblock copolymers of the hydrophile-hydrophobe type (A-B). In a certain embodiment of the present invention the vesicles are prepared from PDMS-PMOXA diblock copolymers to form self-assembled vesicles with aquaporin water channels. For the production of separation membranes comprising aquaporins, the vesicles may be added to an aqueous liquid composition comprising an aromatic amine, such as a diamine or triamine, e.g.,1,3— diaminobenzene (MPD) applied to the surface of a selectively permeable or semipermeable support, which when brought into contact with a solution of an acid chloride in an organic solvent will participate in an interfacial polymerization reaction to form a thin film composite active or selective layer on said support thus forming a separation membrane wherein said vesicles have become immobilized or incorporated. The vesicle incorporating the aquaporin water channel comprising an amphiphilic diblock copolymer of the $PMOXA_{a-b}$-$PDMS_{c-d}$ type (Poly(2-methyloxazoline)—block-poly(dimethylsiloxane) diblock copolymer) as vesicle membrane forming material, may further comprise as an additive of about 0.05% to about 1% v/v of reactive end group functionalised $PDMS_{e-f}$. Examples of said end-functionalised PDMS are, e.g. bis(aminoalkyl) or bis(hydroxyalkyl) terminated $PDMS_{e-f}$, where e—f represents the range of from 30 to 50, such as bis(aminopropyl) terminated poly(dimethylsiloxane) (CAS Number 106214-84—0, Aldrich product No. 481246).

In another embodiment, the aquaporin is stabilized using polyalkyleneimines (PAI), such as polyethyleneimine (PEI), to form self-assembled nanostructures with a detergent stabilized aquaporin. Without wishing to be bound by any particular theory, it is believed that the self-assembled nanostructures form through electrostatic interaction between positively charged nitrogen atoms present in the polyalkyleneimine molecules and amino acid residues in the transmembrane protein that are negatively charged under the conditions (pH, pKs etc.) used to form the nanostructures and/or the membranes comprising the nanostructures.

Generally, the PEI is a substantially linear polymer having an average molecular weight of between about 2,000 Da to about 10,000 Da, such as between about 3,000 Da to about 5,000 Da and the detergent is selected from the group consisting of LDAO, OG, DDM or a combination thereof.

The support layer preferably is a polysulfone or polyether sulfone support membrane.

Aquaporins are present in most living organisms including micro organisms, plants and animals. The present invention is not limited to any particular source for the aquaporins. Typically, the aquaporin is selected from a plant aquaporin, e.g. SoPIP2;1; a mammal aquaporin, e.g. Aqp1; or a bacterial aquaporin, e.g. aquaporin-Z.

The type of coffee beans is not confined to a particular species. Any species of the coffee plant, including the

species of Robusta (Robusta), Arabica (Arabica), Coffea liberia, C. excelsa, C. stenophylla, C. mauritiana. and C. racemose may be used for preparing the aqueous coffee extract concentrated in the present invention. The world production is dominated by the varieties Robusta and Arabica. Following a roasting process, the beans are milled to a certain particles size prior to extraction. The aqueous coffee extract is prepared by contacting roast and ground coffee beans with water and isolating the aqueous coffee extract from the spent roast and ground coffee beans.

A commonly used roasting plant in the industry is rotating cylinders containing the green beans and hot combustion gases. When the bean temperature reaches typically 165-200°C the roasting begins, accompanied by a popping sound similar to that produced by popcorn. These batch cylinders take about 8-15 min to complete the roasting depending on the initial moisture and the desired final color and degree of roasting. Coffee roasting using a fluidized bed is also commonly used.

The roasted beans are then ground to enhance extraction with water. Grinding reduces the particle size of the beans to 0.2-5.0 mm depending on the extraction process. Traditionally, roasted beans are ground by dry milling. The dry milling results in the escape of a characteristic odor, which reflects the escape of aroma components from the roasted coffee beans.

In a certain embodiment of the invention the coffee extract is prepared by cold brewing e.g. as suggested in WO2016004948. According to this method a coffee extract is prepared by

mixing roast coffee beans and water, milling the mixture of roast coffee beans and water in the pressurized chamber and separating the milled mixture into an aqueous coffee extract and spent coffee grounds.

The submersion of the roasted beans in water during the milling results in a substantial amount of volatile aroma components being dissolved in the water and appearing in the extract instead of being liberated to the ambient air. In addition, the closed pressurized chamber ensures that the solubility of the volatile, water soluble aroma components is increased and that the volatile components are maintained in the same compartment without escaping to the surroundings. Furthermore, oxidation is avoided. The use of a pressurized chamber for milling the mixture of roast coffee beans and water also reduces the tendency of foaming.

While water at any appropriate temperature may be used, it is generally preferred that the water is heated prior to the mixing with roast coffee beans to achieve a mixture of 80 °C or less. The use of relatively cold water prevents volatile aroma compounds from degrading. Many aroma components tend to degrade or react with oxygen, water, or compounds in the aqueous mixture. The reaction products produce a sensoric experience of an uncertain nature.

In another cold brewing method, roast beans are ground in dry condition to form a dry particulate product, which may be extracted with water having a temperature of 80°C or less. Subsequently, the aqueous coffee extract is separated from

the spent ground coffee and are concentrated in accordance with the present method.

The solute of the draw solution may be selected among a plurality of components, including organic and inorganic salts, and dissolvable compounds. As a minor amount of reverse membrane flux from the draw solution to the coffee extract may occur to a certain extent it is generally desired to select a solute with an acceptable taste. The type of the draw solution may be a carbohydrate. The carbohydrate may be selected as glucose, fructose, sucrose, maltose, isomaltose, glycerol, etc.

The forward osmosis flow cell may be a classical plate-and-frame module, spiral would module, tubular module, or a hollow fiber module. In a plate and frame module configuration a flat and planar membrane is used to separate the feed solution compartment and the draw solution compartment. Usually, the feed solution compartment comprises elements for distribution of the feed to obtain an efficient use of the membrane area. A tubular module is a crossflow filtration process using a tubular microporous membrane to separate the feed solution compartment and the draw solution compartment. Usually, the feed solution is passed through the bore of the membrane filtration tubes and the draw solution is present in the extra-tubular space. Along the tubes water permeate through the aquaporin-containing membrane through the inside towards the outside, which results in the feed solution being progressively concentrated.

Spiral-wound modules utilize flat-sheet membranes, feed spacers, and permeate spacers wrapped around a hollow tube. Spiral wound elements utilize cross flow technology, and because of its construction, can easily be created in different configurations with varying length, diameter, and membrane material. First, a membrane is laid out and folded in half with the membrane facing inward. Feed spacer is then put in between the folded membranes, forming a membrane sandwich. The purpose of the feed spacer is to provide space for water to flow between the membrane surfaces, and to allow for uniform flow between the membrane leaves.

Next, the draw spacer is attached to the hollow tube, and the membrane sandwich prepared earlier is attached to the draw spacer using glue. The next draw layer is laid down and sealed with glue, and the whole process is repeated until all of the required draw spacers have been attached to the membranes. The finished membrane layers then are wrapped around the tube creating the spiral shape. Feed travels through the flow channels tangentially across the length of the element. Filtrate will then pass across the membrane surface into the draw spacer, where it is carried down the draw spacer towards the drawtube. The coffee extract then becomes concentrated at the end of the element body.

Spiral-wound elements come in multiple configurations with different spacers, membrane types, lengths, and diameters that allow it to fit multiple applications. These elements have a high packing density, surpassing the packing density of plate and frame, tubular, and capillary configurations.

Spiral membranes allow for easy cleaning through cleaning in place.

In a certain aspect of the invention the forward osmosis flow cell is a hollow fiber module. Hollow fiber filtration utilizes numerous long porous filaments packed inside a body. Each filament is narrow in diameter and very flexible. Hollow fiber filtration works on the same principle as tubular and capillary filtration, but utilizes a small tube diameter which allows for flexibility, higher packing density and/or higher water permeability.

Hollow fiber features very high packing density because of the small strand diameter. Because of the flexibility of the strands, certain filter configurations are possible that cannot be achieved in other filtration configurations.

The hollow fibers may be coated with the active layer comprising immobilized aquaporin water channels on the inside or the outside. In a certain aspect, it is desired to use a hollow fiber module having an active layer on the inside. In another aspect, it is desired to use a hollow fiber module coated with an active layer on the outside of the fibers. Both inside and outside coating is disclosed in WO17137361 and WO2014108827, which are incorporated herein by reference.

When an inside coated hollow fiber module is used the feed, i.e. the aqueous coffee extract, is usually passed through the lumen of the fibers, thus allowing water to penetrate the fiber walls and dilute the draw solution, present on the outside of the fibers.

The draw solution may be regenerated by using any conventional technology, including evaporation and pressurized membrane filtration. Pressurized membrane filtration includes nanofiltration (NF), ultrafiltration (UF), and reverse osmosis (RO). The regenerated solution may be sent back to the forward osmosis flow cell. Optionally, the regenerated draw solution is supplemented with solutes to compensate for the reverse flux from the draw solution to the feed solution being concentrated.

The concentrated aqueous coffee extract obtained by the present method may be further dried to obtain an instant coffee product. Examples of such further drying methods include spray drying and freeze drying. Alternatively, the concentrated aqueous coffee extract is used as a concentrated liquid coffee product, which is intended to be diluted by a liquid such as water or milk by the consumer.

Example 1

A hollow fiber module from aquaporin A/S was used as the forward osmosis flow cell (Hollow Fiber Aquaporin Inside™ FO module). The hollow fiber module has a total membrane area of 2.3 m^2.

The hollow fiber module is disclosed in WO14108827. The hollow fibers of the module are covered on the inside with a thin film composite incorporating aquaporin water channels.

The aqueous coffee extract has a dry matter content of 1.5% by weight and is used as the feed solution. The feed solution is directed to the lumen of the hollow fibers, i.e. the feed solution passes through the individual hollow fibers from the inlet to the outlet in the other end of the fiber.

On the external side of the hollow fibers the draw solution is passed from the inlet of the housing surrounding the bundle of hollow fibers to the outlet. The draw solution contains 1 M sucrose.

During the experiment the aqueous coffee extract as well as the draw solution are recirculated by pumps for the respective fluid circuits. The duration of the experiment is 45 minutes. It is observed during the experiment that the amount of aqueous coffee extract in the storage container decreases due to the migration of water from the feed solution to the draw solution. At the end of the runtime the volume of the aqueous coffee extract was reduced more than 90% corresponding to a dry matter content of the aqueous coffee extracts of 15% by weight or more.

The experiment shows the usefulness of aquaporins in forward osmosis modules for dewatering of an aqueous coffee extract.

The various aspects and implementations has been described in conjunction with various embodiments herein. However, other variations to the disclosed embodiments can be understood and effected by those skilled in the art in practicing the claimed subject-matter, from a study of the drawings, the disclosure, and the appended claims. In the claims, the word “comprising”

does not exclude other elements or steps, and the indefinite article “a” or “an” does not exclude a plurality. A single processor or other unit may fulfill the functions of several items recited in the claims. The mere fact that certain measures are recited in mutually different dependent claims does not indicate that a combination of these measured cannot be used to advantage.

CLAIMS

1. A method for concentrating an aqueous coffee extract in a forward osmosis flow cell comprising a feed solution compartment and a draw solution compartment separated by a membrane, wherein

the feed solution compartment comprises an inlet and an outlet for passing the aqueous coffee extract,

the draw solution compartment comprises an inlet and an outlet for passing a draw solution containing one or more solutes, and

the membrane comprises an active layer and a support layer, wherein the active layer comprises immobilized aquaporin water channels.

2. The method according to claim 1, wherein the aqueous coffee extract is recirculated in the feed solution compartment and/or the draw solution containing one or more solutes is recirculated in the draw solution compartment.

3. The method according to claim 1 or claim 2, wherein the aquaporin water channels are immobilized in a thin film composite (TFC) layer.

4. The method according to any of the claims 1 to 3, wherein the aquaporin water channels are incorporated in vesicles or wherein a detergent stabilized aquaporin is comprised in a self-assembled nanostructure comprising polyethyleneimine.

5. The method according to any of the claims 1 to 4, wherein the active layer is a cross-linked aromatic amide layer formed

by interfacial polymerization and/or wherein the active layer faces to the feed solution compartment.

6. The method according to any of the claims 1 to 5, wherein the support layer is a polysulfone or polyether sulfone support membrane.

7. The method according to any of claims 1 to 6, wherein the aquaporin is selected from a plant aquaporin, e.g. SoPIP2;1; a mammal aquaporin, e.g. Aqp1; or a bacterial aquaporin, e.g. aquaporin-Z.

8. The method according to any of the claims 7, wherein the aqueous coffee extract is prepared by contacting roast and ground coffee beans with water and isolating the aqueous coffee extract from the spent roast and ground coffee beans.

9. The method according to claim 8, wherein the temperature of the water is 80°C or below.

10. The method according to any of the claims 1 to 9, wherein the solute of the draw solution is a carbohydrate.